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THE USE OF TABLETING INDICES TO STUDY THE COMPACTION PROPERTIES OF POWDERS

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ABSTRACT

Tableting indices are employed to investigate some compression properties of a binary mixture of a direct compression excipient, calcium sulfate, and a tablet lubricant, magnesium stearate. The technique involved modifications of previously described equipment used to acquire and quantitate the bonding index, brittle fracture index, and strain index. The present paper describes the significant modifications and contributions made on equipment, and presents some results obtained for some binary mixtures of powders.

INTRODUCTION

Knowledge of compression properties of powdered materials are important to ensure that the newly formed compacted mass will remain bonded after compression forces are released. Several events occur during the formation of a tablet. After a load of powder particles has been deposited in the die, an axial force is applied by means of a downward movement of the upper

punch for the case of an eccentric tablet press. At very low compression forces, densification of the powder bed occurs as the particles rearrange by sliding past one another within the die. As the force applied to the powder bed increases, the particles undergo either fragmentation or deformation (1). Subsequently, a reduction in porosity and an increase in the true contact areas between particles produce the inherent strength of the resultant tablet. The challenge in pharmaceutical systems is afforded by the variety of powdered materials which are candidates for formulation into tablets, and the diversity of their physical and chemical nature. This diversity insures that each system will act in a unique manner. Thus, a thorough understanding of the effects of the mechanical properties of powdered materials is imperative to the formulation scientist.

Recently, three tableting indices, the bonding index (BI), the brittle fracture index (BFI), and the strain index (SI), were proposed by Hiestand and Smith (2) to provide useful information about compaction properties of powders. Hiestand (3) stated that by using the BI and BFI, excipients may be rationally chosen to overcome an undesirable characteristic of an active medicament. He also indicated that the indices of the final formulation may indicate success or failure, and that knowing the indices for each component of a formulation may afford a quality control device to monitor their mechanical properties and screen out any problem lot of material intended for a formulation. The BI is defined as the ratio of the tensile strength to indentation hardness (i.e. σ_t / P). This ratio estimates the success of bond formation during compression and bond survival during decompression. The ratio of tensile strength, which is the strength of the material in a defined state of tension, to indentation hardness, which is proportional to the shear strength of the material under a standard state of compression, provides information concerning the success of bonding (3). Since

plastic flow is an important process involved in tableting, the determination of P is of obvious interest. In addition, if the shear strength and tensile strength result from equal numbers of bonded areas in a compact, then the ratio of tensile strength to indentation hardness may vary according to the influence of the mechanical structure on these terms. The presence of crack propagation will affect the magnitude of the tensile strength. Cracks are less likely to develop during indentation hardness testing than during tensile strength testing. The fracture plane that develops during the tensile strength test may be considered as a crack. The fracture plane is the only crack that should be allowed in this test since the presence of other cracks may negate the usefulness of the data derived from the tensile strength test. The development of cracks may negate the results of both the dynamic indentation hardness test and the tensile strength test. A large value of the bonding index corresponds to the survival of large true areas of contact during elastic recovery and significant plastic deformation during decompression. This contact area between particles occurs as the particles yield plastically. In contrast, a small value of the bonding index corresponds to the survival of only minimal areas of true contact established during compression, and minimal plastic deformation during decompression (4).

The BFI is described according to equation 1:

$$BFI = 0.5 [(\sigma_t / \sigma_{to}) - 1] \quad (1)$$

where σ_t is the tensile strength of a compact without a hole in the center, and σ_{to} is the tensile strength of a compact with a small axially oriented hole in the center. The brittle fracture index indicates the ability or inability of a compact to relieve stresses by plastic deformation or plastic flow that are caused by the

presence of a stress concentrating region in the compact. For a material that is unable to relieve stresses at the site of stress concentration, tensile fracture may occur at about one-third of the tensile stress required to produce tensile fracture when no hole is present (5). The ratio of the tensile strengths (i.e. σ_t / σ_{to}) is normalized so that the value of the brittle fracture index ranges between zero and one. The relevance of the ability of a compact to relieve locally concentrated stresses at the edge of the hole by plastic deformation is of obvious importance. Materials that demonstrate a high shear strength also have a high probability of undergoing brittle fracture. Brittle fracture occurs as a result of fracture by crack propagation from a stress concentrating flaw. Like the stress concentrating hole present in the tablet, the edge of a die is also an area of very high stress concentration. Knowledge of how a compact reacts to the presence of a hole may be applicable to studying the tendency of a compact to laminate or cap. Hiestand (6) commented that one of the primary causes of capping and lamination was the brittleness of the compact. A material demonstrating no brittle characteristics is able to relieve stress (i.e. the stress concentrating hole does not effect the magnitude of the tensile strength) by plastic deformation, and produces a ratio of $\sigma_t / \sigma_{to} = 1$ and a BFI = 0. Since all excess stresses at the edge of the hole are relieved by plastic deformation, there is no significant difference in magnitude between the tensile strength of a compact with and without a hole. Conversly, a brittle material that is unable to relieve stress by plastic deformation (i.e. undergoes brittle fracture by crack propagation from the stress concentrating hole) produces a ratio of $\sigma_t / \sigma_{to} = 3$ and a BFI = 1. The theoretical basis for the BFI is the Griffith crack theory. The Griffith crack theory says that in order for a crack to propagate spontaneously, the energy stored at the tip of a crack must exceed the energy required to form the two new

surfaces resulting in the growth of a crack (6). Therefore, when the energy stored as elastic energy at a region of stress concentration is of such magnitude to supply the surface energy necessary to create new surfaces, fracture and crack growth occur. The crack will grow when the loss of elastic energy is equal to the increase of surface energy due to the new surfaces. The brittle fracture index may be used to indicate whether a material has the ability to relieve shear stresses by plastic deformation, and whether capping or lamination may present a problem during the tableting process.

The strain index is defined as the ratio of P / E' where P is the dynamic indentation hardness of the compact including the particles and pores, and 1 / E' = $(1 - \nu_1^2) / E_1 + (1 - \nu_2^2) / E_2$, where ν is the Poisson's ratio and E is the Young's modulus of elasticity for particles 1 and 2, respectively. The strain index can also be calculated from equation 2:

$$SI = [5\ a / 6\ \pi\ r\ ] /\ [\ h_i/h_r - 3/8] \qquad (2)$$

where a is the chordal radius of the dent produced from the impaction of the indenter with the surface of the tablet, r is the radius of the spherical indenter, h_i is the initial height of the spherical indenter, and h_r is the height of rebound of the spherical indenter. Indirectly, the strain index is relevent to the theory of bond formation because the strain index values are used to extrapolate to the solid fraction of unity that is needed for bond formation. It permits the characterization of the radii of curvature resulting from elastic recovery following plastic deformation, and may correspond to the proximity of the surfaces that stay in contact following decompression. Since the ratio of P/E' is stress divided by stress/strain, it represents the actual strain in the x direction,

when the stress in the y direction and the strain in the z direction are both equal to zero (2). Therefore, the strain index may correspond to the relative strain energy that could develop at a defect or stress concentrator during elastic recovery following plastic deformation.

EXPERIMENTAL

The present paper describes the significant modifications and contributions made on equipment that was originally described by Hiestand and Smith (2) to obtain the parameters necessary for calculating the tableting indices. In addition, preliminary results obtained for some binary mixtures of powdered materials are presented.

Material

Direct compression calcium sulfate (Destab calcium sulfate 90A/A™, Desmo Chemical Corp., St. Louis, MO.) was used in combination with varying levels of magnesium stearate (Phibro Pharma, Inc., New York, NY).

Apparatus for Compact Formation

Compacts were made using a Carver™ 25 ton laboratory press (Fred Carver, Menomonee Falls, WI.). This basic apparatus was similar to the tablet forming equipment described by Hiestand and Smith (2) to include a variable speed motor capable of driving the lower platen. The variable speed motor allowed the critical parameter of platen velocity to be chosen such that the rate of load application could be precisely monitored and controlled. In these experiments, the hand pump of the press was pumped manually to obtain the desired compression force, and held constant for one minute. The one minute

time constant was maintained throughout the experiments in order to increase the probability of a series of compacts having nearly identical properties. A split square die measuring 2.54 centimeters on a side was used to form the compacts. The split die employed in the present study was of similar design to that used previously by Hiestand and Smith (2), but measured 1.27 centimeters (one-half inch) less on a side. This corresponded to a cross-section of 6.45 cm^2 in the present study compared with a cross-section of 14.5 cm^2 in the previous study of Hiestand and Smith (2). In addition, 10 to 15 gram tablets were compacted and evaluated by the previous investigators, whereas 5 gram tablets were employed in this study. The surfaces of the punches were flat because the curvature of the punch face may influence the distribution of the powder in the compressed tablet, and may influence the magnitude of the dynamic indentation hardness one observes. The square die was split along a line through the diagonal, and was capable of triaxial decompression as the compression pressure was reduced. Upon decompression, the die walls moved outwardly providing the triaxial decompression. A split die is preferred over a standard die (i.e. principally uniaxial decompression) because as tablets are ejected from the die, they are subjected to transverse shearing stresses at the point where they emerge from the die. At this point, structural failure of the tablet due to the transverse shearing stresses, coupled with inadequate tablet shear strength, may cause capping or lamination. In addition, tablets may fail or fracture within the die during decompression because of the shear stresses. Triaxial decompression may avoid fracture of compacts. The punches and die were made of aluminum, and coated with Tufram™ (General Magnaplate Corp., Arlington, TX.). Tufram™ was applied by a coating process, and provided a surface of extreme hardness, ultra low friction, nonstick mold release properties, and corrosion resistance. A

surface coat of a slurry of 50% magnesium stearate in a 50:50 ethanol / water mixtures was applied to the Tufram™ coating prior to each compact formation and allowed to dry. The Tufram™ coating and surface coating of the magnesium stearate slurry provided an adequately lubricated surface which prevented the solid particles from sticking to the surfaces of the punches and die.

The force on the punches was measured with a load cell (I.S.I., Inc., Round Rock, TX.) mounted directly on the press. The compression load cell system consisted of an electronic control and digital readout (with an analog output), and a compression load cell that used strain gauge sensors in a full wheatstone bridge configuration. The load cell capacity was 10,000 kg. The following procedure was used to establish calibration in kilograms: 1.) power was turned on and a five minute warm up period was observed (initial display on screen read "= 18888" for approximately two seconds); 2.) gain switch was set to "X1"; 3.) zero pot was unlocked with a 1/4 turn counter clockwise and adjusted for zero reading on the display screen, then locked to zero control; 4.) calibration switch was actuated at the same time that the span pot was unlocked and adjusted for calibration reading from the load cell calibration sheet, then the span pot was locked in position; and 5.) calibration switch was released, and display screen read zero. The load cell and display unit were then calibrated for 10,000 kg full scale and ready for use. The compression load cell system was also capable of being switched to a 2000 kg full scale by setting the gain switch to "X10" and readjusting the zero reading according to the procedure described above. The 2000 kg full scale was used for determining the tensile strength of compacts. The signal from the load cell was simutaneously monitored on a digital oscilloscope (Model 206-2 Explorer II, Nicolet Instrument Corp., Madison, WI.). A calibration was performed between the oscilloscope and load cell

compression system so that a force reading obtained on the oscilloscope could be converted from a millivolt to kilogram reading.

Apparatus for Measurement of Tensile Strength

The Carver™ hydraulic press was also modified (Havatec Inc., Austin, TX.) to include a precision drive for measuring the tensile strength of compacts. The precision drive consisted of a small permanent magnet motor with an integral gear reducer that drives a ball screw. The ball screw nut then served to drive a small hydraulic cylinder connected to the larger hydraulic cylinder that was originally supplied with the Carver™ press. The motor controller allowed movement in both forward and reverse directions, and 2% of base speed control over a 15 to 1 speed range. On the low speed setting, as the dial setting was varied from 20 to 90 (arbitrary scale reading), the speed of the platens ranged from 0.01 mm/sec to 0.25 mm/sec. The housing unit that held the square compacts for the transverse compression tensile test consisted of the upper and lower platens which measured 0.4 times the width of the square compacts, and the compression load cell system previously described. The compression load cell system could be altered and used on a 2,000 kg full scale instead of the 10,000 kg full scale used to form the tablet. A 0.4 ratio of platen width to compact width gave a maximum tensile strength value equal to 0.16 times the mean compressive stress acting on the edge of the tablet at the instant of fracture. The compact was centered on edge between the upper and lower platens, and the variable speed motor provided a constant rate of compression and an exponential rate of load application. A time constant of 10 seconds was chosen between the maximum force and 1/e times the maximum force. Pads were used to cover the face of the platens (i.e. positioned between the surfaces of the tablet and platen)

in order to get the proper fracture plane with more materials. Following fracture, the shape of the fragments was checked to determine the mode of failure. Only compacts that showed failure in tension as evidenced by splitting into two equal halves were used in acquiring tensile strength data. In order to acquire tensile strength data for determining the brittle fracture index, square compacts were also made to contain a small axially oriented hole in the center. This was accomplished by using a specially designed punch that contained a spring loaded pin in the center of the punch face as was previously described by Hiestand et al. (5). The pin measured one millimeter in diameter. After the punch was in place in the die, an axial compression pressure was applied causing the spring loaded pin to retract into the body of the punch except for that portion of the pin equal to the thickness of the compact. The pin was also coated with a slurry of magnesium stearate in water/ethanol and allowed to air dry as previously described in this paper. The results of the tensile test performed on tablets containing the small hole in their centers was used in determining the brittle fracture index.

Pendulum Impact Apparatus

The resistance of compacts to permanent deformation was measured dynamically using a pendulum arrangement. The basic design of the pendulum apparatus that was described earlier (2,7) was modified in the present study to include a ballistic sensor capable of accurately and reproducibly measuring the initial and rebound velocities of the steel sphere before and after impact with the compact. The pendulum impact apparatus and ballistic sensor are shown in Fig. 1. This method of dynamically measuring the indentation hardness was nondestructive. The pendulum impact apparatus employed in this investigation consisted of a steel sphere suspended on a one meter steel wire and positioned along an arc by a magnet (A), and a ballistic sensor for measuring the initial and

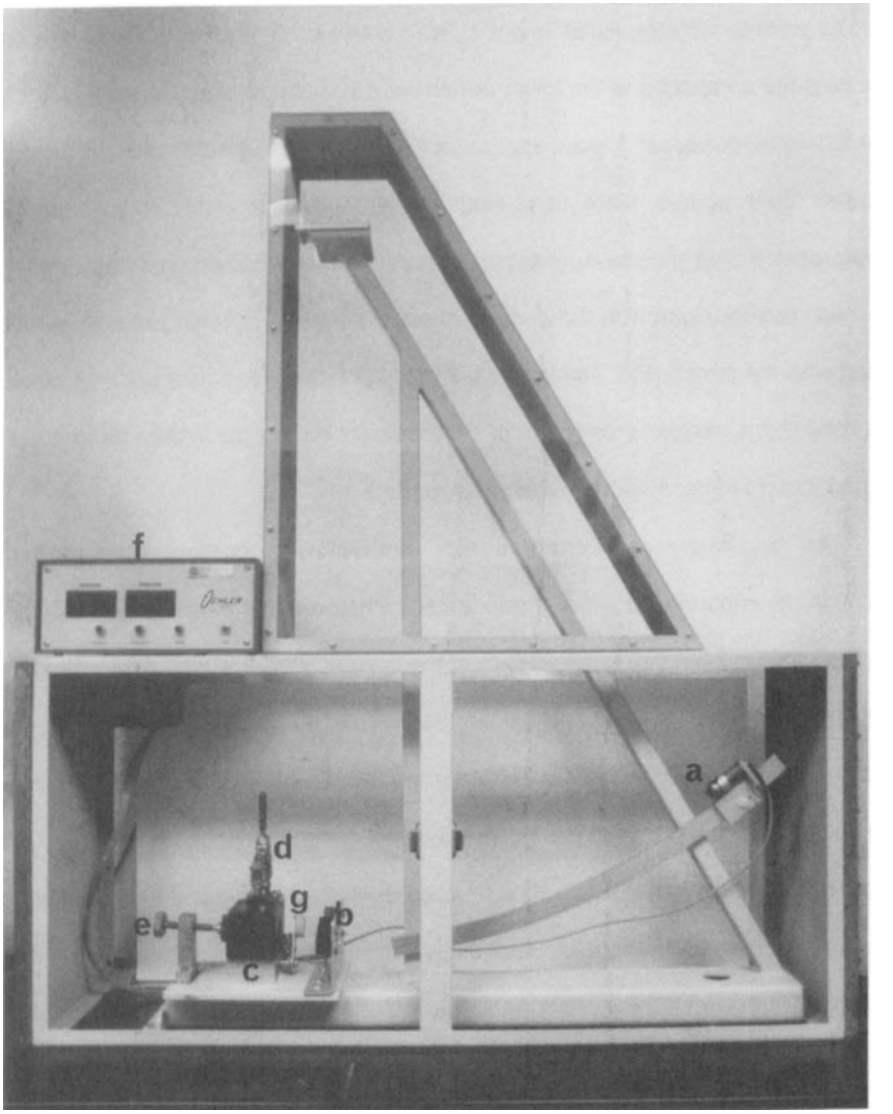

FIGURE 1

Pendulum Impact Apparatus - (a) steel sphere suspended on a one meter steel wire and held in position by a magnet; (b) ballistic sensor; (c) platform to align and secure die; (d) clamp to secure die; (e) screw lever to position punch and tablet in die; (f) chronograph; (g) bumper gate.

rebound velocities of the sphere (B). The distance of the sphere could be varied along an arc in order to vary the initial height of the indenter. After compact formation, the die containing the compact and the lower punch were positioned and clamped securely into place (D) in the pendulum impact apparatus (C). During the determination of indentation hardness, four screws were used with the split die assembly to prevent triaxial decompression after the pressure was decreased in the hydraulic press assembly. The tablet was pushed flush with the surface of the die by the tightening of a screw lever in contact with the punch (E). The screw lever and punch served as a back-up block, and prevented the backward movement of the compact during the hardness test. Hence, only one face of the compact was left unsupported.

As previously mentioned, earlier studies used photographic methods to determine the rebound height of the indenter. Photographic methods consisted of the following process. Following the release of the sphere, a time lapsed photo was taken of the rebound of the sphere, and subsequently the rebound height of the spherical indenter was obtained from the photograph and used to calculate the dynamic indentation hardness (2). In these studies, a chronograph (Havatec Inc., Austin, TX.) was used in combination with the pendulum impact apparatus for measuring the inbound and rebound velocities of the sphere. As observed in Fig. 1, the chronograph (F) that was developed for these studies was capable of: 1.) energizing the holding magnet as soon as the power was turned on; 2.) releasing the sphere when the "Start" button was pushed; 3.) recording the inbound velocity on the first pass through the timing gates; 4.) recording the rebound velocity on the second pass through the timing gates; and 5.) closing the bumper gate (G) after the second pass and re-energizing the holding magnet. The holding magnet held the indenter in position prior to release, and could be positioned anywhere along a 45^{o} arc by tightening a screw. The padded bumper gate prevented the sphere from hitting the surface of the compact a second time. The

chronograph contains a solid state infrared light source and detector with appropriate lenses to make a very narrow beam. After release of the sphere from the holding magnet, the beam was interrupted by the equator of the sphere as the sphere passed through the beam. The time of this interruption and the diameter of the sphere were used to compute the velocity of the sphere. The frequency of the crystal oscillator used for timing was chosen at 3.1496 MHz to provide a velocity readout in millimeters per second with a ball diameter of 1 inch and an internal distance switch setting of "20". It is possible to vary the size of the sphere used in the pendulum arrangement. If this is done, the internal distance switch setting must also be changed in accordance with the following relationships: 1.) 0.85 inch diameter sphere = 17 switch setting; 2.) 0.90 inch diameter sphere = 18 switch setting; 3.) 0.95 inch diameter sphere = 19 switch setting; 4.) 1.00 inch diameter sphere = 20 switch setting; and etc. Using the 1 inch sphere, the lowest velocity recorded was 305 mm/sec. This reading corresponded roughly to a half swing of the pendulum of approximately 5.5^{o} or a half travel of the ball of approximately 10 centimeters. When the velocity was below this level, the screen displayed "Error" instead of a numerical value for the velocity.

Verification that the infrared light beam was cut by the equator of the sphere was made by visually checking that the equator of the sphere passed through the center of the beam. It was also verified by clamping the holding magnet at a fixed position along the arc and performing a series of releases. As the length of the pendulum was varied, the average inbound velocity of the series was noted. The pendulum length which showed the lowest velocity corresponded to the length which caused the equator of the sphere to pass through the beam.

Apparatus for Measurement of Chordal Radius

Following the release of the sphere from the magnet, the sphere hit the surface of the compact and created a dent. The chordal radius of the dent was determined by

optical microscopy (Zeiss Corp., West Germany). A similar microscopic procedure for the determination of the chordal radius was used previously (2,7). At the same time, the inbound and rebound velocities were displayed on a screen and stored in a microcomputer. The microcomputer determined the mean, standard deviation, high and low values, and range of the stored data. The chronograph was reset at the start of each new data set. The dynamic indentation hardness and strain index were calculated from the chordal radius of the dent, inbound velocity of the sphere, and rebound velocity of the sphere using a specially written FORTRAN computer program.

Determination of Solid Fraction

The solid fraction of the compacts was determined by dividing the apparent density of the compact by the true density of the powdered solid. The apparent density was determined from the weight of the compact divided by the volume of the compact (i.e. the physical dimensions of the compact). The true density was determined using a helium pycnometer (Model 1302, Micromeritics, Norcross, GA.). Using the helium pycnometer, a sample of powdered material was placed in a sample cup within a sealed chamber. The air in the sealed chamber was evacuated and filled with helium. By comparison with a sample of known volume, the true volume of an unknown sample was determined. The true density of the material was subsequently measured by dividing the weight of the unknown sample by the true volume of the unknown sample previously determined by the helium pycnometer.

RESULTS AND DISCUSSION

The failure of some powders to compact favorably when compressed into tablets may be due to the amount of pressure which may be safely applied to the compacts, and to the distribution of the forces within the compact itself. In

addition, the presence of a lubricant in a tablet formulation will effect the integrity of compacts. In this preliminary investigation, the tensile strength, dynamic indentation hardness, bonding index, brittle fracture index, and strain index were determined for a binary mixture of direct compression calcium sulfate (Destab calcium sulfate 90A/A™ , Desmo Chemical Corp., St. Louis, MO.) in combination with varying levels of magnesium stearate (Phibro Pharma, Inc., New York, NY). This excipient will be referred to as calcium sulfate, and was used as received from the manufacturer. Magnesium stearate, which is present in tablet formulations as a lubricant, acts by contaminating the surfaces of the host particles of powder, and has been described as a boundary lubricant (8). Lubrication with magnesium stearate is considered to be a surface phenomenon, and the extent of lubrication may depend on how many of the lubricant particles are adsorbed on the solid substrate. Magnesium stearate was first passed through a number 100 mesh screen before being combined with calcium sulfate. All binary mixtures were blended in a twin-shell V blender for five minutes. The presence of magnesium stearate in a formulation assists particle movement and consolidation of the tablet by reducing die wall friction. The internal rearrangement within the particles probably leads to a much denser and closer packed system, although the attractive forces between host particles are reduced.

The effect of the presence of magnesium stearate on the tensile strength of calcium sulfate is illustrated in Fig. 2. It should be noted that all of the measurements are reported at a solid fraction of 0.57. Values of the tensile strength for the 3%, and 5% calcium sulfate / magnesium stearate admixtures were obtained from a least squares regression analysis of the log-linear relationship between tensile strength and solid fraction. The correlations for plots of the logarithm of tensile strength versus solid fraction yielded highly

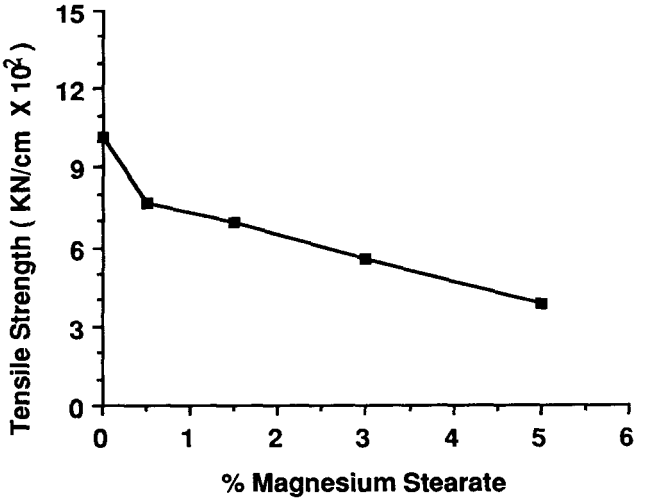

FIGURE 2
The tensile strength of tablets of calcium sulfate as a function of magnesium stearate concentration. (solid fraction = 0.57).

significant correlation coefficients. The correlation coefficients for the 3% and 5% admixtures were 0.9952 and 0.9964, respectively. As seen in Fig. 2, the tensile strength that was measured for compacts of pure calcium sulfate at a solid fraction of 0.57 was approximately 102 N/cm^2 . As the level of magnesium stearate in the compacts was progressively increased up to the 5% level, the magnitude of the tensile strength was reduced to approximately 50 N/cm^2 . This represented a decrease in the magnitude of the tensile strength of nearly 50%. The presence of particles of magnesium stearate in the binary mixture effectively contaminated the surfaces of the host particles and reduced the magnitude of the tensile strength. The magnitude of the lubricant action on the host particlesmay depend on the concnetration of the lubricant used in the binary mixture.

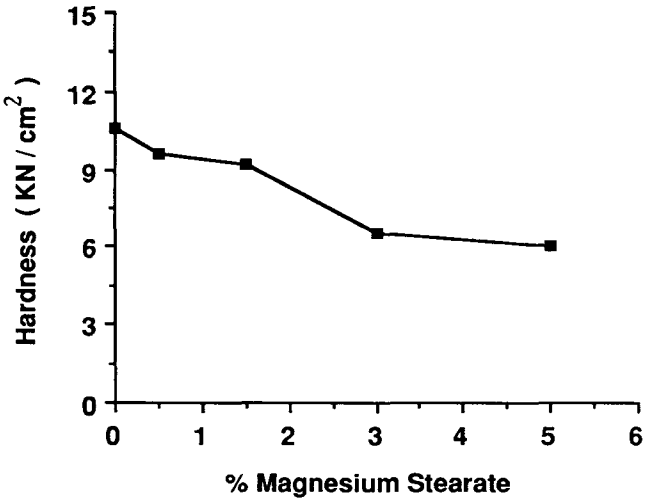

FIGURE 3
The indentation hardness of tablets of calcium sulfate as a function of magnesium stearate concentration. (solid fraction = 0.57).

Hardness was defined by Tabor as the resistance of a solid to permanent deformation (9). In this investigation, the dynamic indentation hardness would represent the resistance of a tablet to the indentation process of the spherical indenter employed in the pendulum arrangement. The results displayed in Fig. 3 demonstrate the variation of dynamic indentation hardness with increasing levels of magnesium stearate. It can be seen that the indentation hardness of compacts made from pure calcium sulfate was approximately 10 KN/cm^2 at a solid fraction of 0.57. As the levels of magnesium stearate were increased to 5%, the indentation hardness decreased to approximately 6 KN/cm^2. The hardness of the compacts decreased as the level of magnesium stearate was increased in the binary mixture. This probably occurred because of the coating of the larger particles of calcium sulfate by the smaller particles of magnesium stearate., and therefore the ability of the compact to resist permanent deformation was reduced.

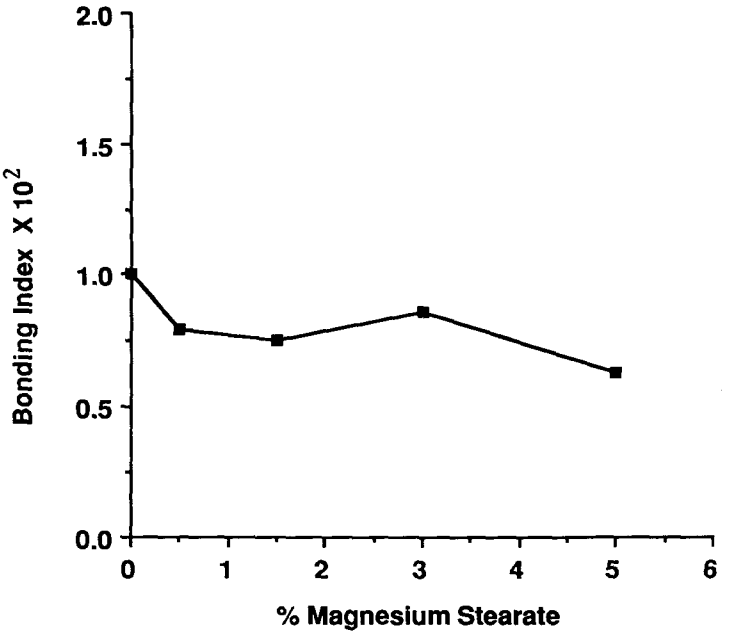

FIGURE 4
The bonding index of tablets of calcium sulfate as a function of magnesium stearate concentration. (solid fraction = 0.57).

The bonding index is defined as the ratio of the tensile strength to the dynamic indentation hardness. The results presented in Fig. 4 demonstrate the variation of the bonding index of calcium sulfate in combination with varying levels of magnesium stearate. As discussed previously, the tensile strength of compacts of pure calcium sulfate was approximately 102 N/cm^2 . As the level of magnesium stearate in the compacts was progressively increased up to 5%, the magnitude of the tensile strength decreased to approximately 38 N/cm^2 . Similarly, the dynamic indentation hardness decreased from a value of approximately 10 KN/cm^2 for compacts of pure calcium sulfate to a value of about 6 KN/cm^2 for compacts prepared from the 5% binary mixture. The tablet tensile strength decreased from 102 N/cm^2 for compacts of pure calcium sulfate

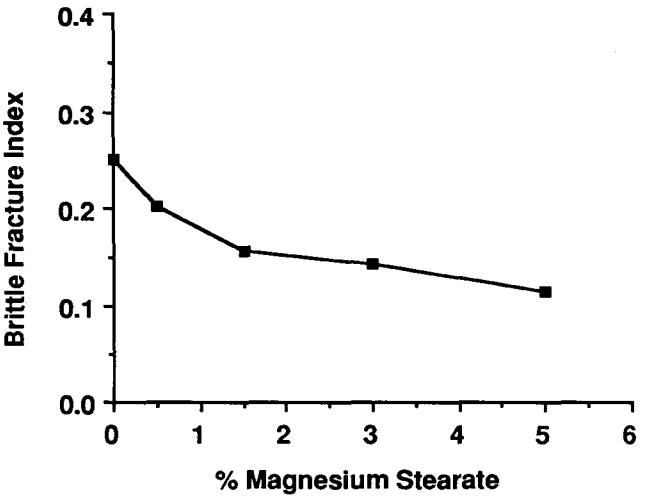

FIGURE 5
The brittle fracture index of tablets of calcium sulfate as a function of magnesium stearate concentration. (solid fraction = 0.57).

to 38 N/cm^2 for compacts made from the 5% admixture. It can be concluded that in the presence of increasing levels of magnesium stearate, compacts composed of calcium sulfate and magnesium stearate were unable to maintain the extensive areas of true contact that were established under maximum compressive stress during decompression. Studies are currently underway to investigate the effects of mixing times and blender shapes on the tableting indices.

The results in Fig. 5 demonstrate the variation of the brittle fracture index for compacts made from calcium sulfate and varying levels of magnesium stearate. It can be seen that initially, compacts prepared from pure calcium sulfate had a BFI of about 0.25. As the concentration of magnesium stearate in the compact was steadily increased, the BFI decreased monotonically up to the 5% level. The presence of increasing levels of magnesium stearate reduced the BFI to

a safer value, one where the propensity for brittle fracture was minimal. The accumulation of magnesium stearate at the surfaces of the host particles reduced the shear strength of the compact, hence it yielded at a lower stress and was less likely to propagate a crack. However, this must be balanced against the loss of bond strength as evidenced by the decrease in the bonding index as noted previously. As the level of magnesium stearate was increased in the formulation, compacts were able to relieve stresses created by the stress concentrating hole in the center of the compact more efficiently. Thus, the difference between the magnitudes of the tensile strengths with and without a hole was decreased.

The strain index allows for the characterization of the radii of curvature that result from the release of elastic stresses after plastic deformation, and is indirectly relevent to the theory of bond formation (6). The profile displayed in Fig. 6 shows the effect on the strain index as the level of magnesium stearate was steadily increased for binary mixtures prepared with calcium sulfate. It can be seen that in the presence of magnesium stearate, the magnitude of the strain index decreased monotonically from 0.68×10^{-2} at the 0% level to 0.53×10^{-2} at the 5% level. Since no other similar investigations have been done using the strain index, the significance of these decreases was not yet apparent. The results from the strain index should be used in conjunction with the results from the bonding index and the brittle fracture index.

The equipment necessary for obtaining the parameters for calculating the bonding index, brittle fracture index, and strain index has been successfully designed and constructed from modifications of previously described equipment (2). The pendulum impact apparatus fitted with a ballistic sensor provided accurate and reproducible determinations of the initial and rebound heights of the spherical indenter. The apparatus was easy and convenient to use as compared to

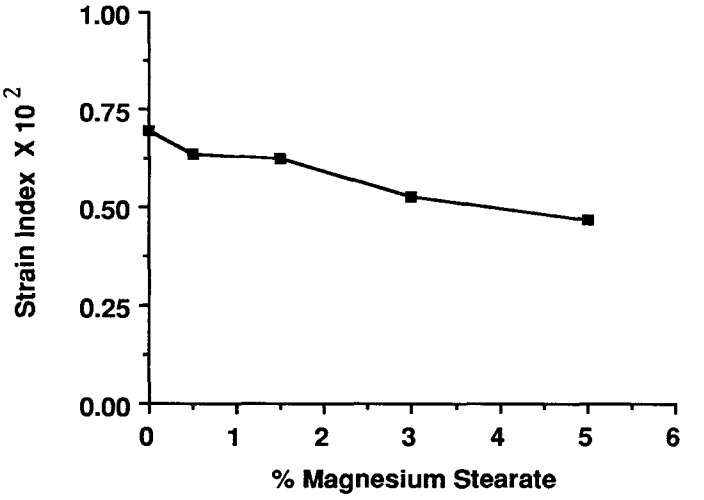

FIGURE 6
The strain index of tablets of calcium sulfate as a function of magnesium stearate concentration. (solid fraction = 0.57).

previously used photographic methods. Tablet compaction and tensile strength determination were suitably performed using a modified hydraulic press equipped with a large and small hydraulic cylinder instead of employing two apparatus, each dedicated for a single function, (i.e. either tablet compaction or tensile strength determination). Preliminary results indicated that the bonding index, brittle fracture index, and strain index may be useful in characterizing the compaction properties of single component and binary mixtures of tableting excipients. It was found that the tensile strength and dynamic indentation hardness decreased as the level of magnesium stearate in the binary mixture with Destab calcium sulfate was increased. For the same binary system of magnesium stearate and Destab calcium sulfate, it was also found that the bonding index decreased from the 0% level to the 0.5% level of lubricant, then showed a plateau up to the 5% level. In addition, it was found that the brittle fracture

index and the strain index decreased as the level of magnesium stearate was increased up to the 5% level. More investigations are warranted using binary systems before more complex systems are considered. The utility of the tableting indices may not be recognized if they are first used to characterize complex systems of materials before more basic systems.

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REFERENCES

1. P. York, Solid-state properties of powders in the formulation of solid dosage forms. Int. J. Pharm.,14(1983)1-28.

2. E. Hiestand and D. Smith, Indices of tableting performance. Powder Technol., 38(1984)145-159.

3. E. Hiestand, Use of Hiestand indices as a predictive tool. In Proceedings of the Fourth Wisconsin Update Conference, Madison, Wisconsin, November, 1984, pp 271-770.

4. E. Hiestand, Dispersion forces and plastic deformation in tablet bond. J. Pharm. Sci., 74(1985)768-770.

5. E. Hiestand, J. Wells, C. Peot, and J. Ochs, Physical processes of tableting. J. Pharm. Sci., 66(1977)510-519.

6. E. Hiestand, Tablet bond and related measurements. Pharm. Technol., Feb(1986)52-58.

7. E. Hiestand, J. Bane, and E. Strzelinski, Impact test for hardness of compressed powder compacts. J. Pharm. Sci., 60(1971)758-763.

8. W. Strickland, T. Higuchi, and L. Busse, The physics of tablet compression X: Mechanism of action and evaluation of tablet lubricants. J. Am. Pharm. Assoc., 49(1960)35-40.

9. D. Tabor, A simple theory of static and dynamic hardness. Proc. Royal Soc., London, A192(1948)247-274.