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DETERMINATION OF A RELATIONSHIP BETWEEN FORCE-DISPLACEMENT AND FORCE-TIME COMPRESSION CURVES

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ABSTRACT

A series of experiments were conducted to evaluate and compare force-displacement and force-time compression curves. A Stokes B-2 sixteen station rotary tablet press was instrumented with piezoelectric transducers to monitor compression and ejection forces (in addition to punch proximity) and interfaced with a microcomputer. Processing and material variables were examined for their effects on the direct parameters (*ie.* height and area) and derived parameters (*ie.* area:height ratio and maximum slope:height ratio) of the force-time compression curve. Upper punch displacement was estimated and force-displacement curves were plotted. The force-time curve was then divided into three segments pertaining to the three stages of upper punch movement: compression, relaxation, and decompression. The "relaxation" stage was defined as the portion of the compression force-time curve corresponding to the interval when the upper punch displacement was held constant. The total, net and elastic works of compaction were calculated and their relationships with the areas of the individual phases of the force-time compression curve were examined. It appears that the area under the compression force-time curve can be related satisfactorily to the work of compaction.

INTRODUCTION

Many papers have been published showing the value of the compaction parameter, work. Historically, research which has been conducted, measuring the parameter work, has been limited to the single station eccentric type tablet machine. Recently though, several workers have calculated, and one has actually measured punch displacement during the compaction event on the rotary tablet press [1-3]. T.M. Jones *et al.* developed a system using short range radiotelemetry to acquire the punch position signal measured by a linear variable differential transformer mounted on the tablet press [2]. Rippie and Danielson [3] and Charlton and Newton [1] have attempted to calculate the punch displacement using the geometric dimensions of the rotary tablet presses. These mathematical methods are slightly inaccurate and complex, and the data obtained has lacked reproducibility.

Since, it becomes very time consuming for the scientist to convert force-time experimental data into force-displacement measurements, a more reasonable approach would involve the use of the compression force-time diagram in compression studies. The use of the microcomputer as a monitoring device in compression studies will conveniently allow the integration of the compression force-time curve and enable the use of the area under this curve as a parameter in compression studies.

The present paper describes the research performed in the authors' laboratory to calculate punch displacement and determine if work done in compacting a tablet could be related to the area under the compression force-time curve. It is hoped that by investigating this relationship between the two parameters that a more practical technique can be developed for studying formulation and processing variables on the rotary tablet press, and the usefulness of the rotary tablet machine will be enhanced.

EXPERIMENTATION

Materials and Methods

The instrumentation of a Stokes B-2 sixteen station rotary tablet machine using integral coupled piezoelectric transducers located in the eye bolt (to monitor compaction forces) and the ejection cam (to measure ejection forces) has been described previously [4]. Force measurements were acquired from the tablet press by an Apple II plus computer interfaced to the tablet press through an Interactive Structures Inc. AI13 fast analog-to-digital converter. Data points were acquired at a rate of one kilohertz.

Four direct compression matrices consisting of Avicel PH-102, Emcompress, anhydous lactose and Starch 1500 were used in the study. Each matrix was blended for five minutes in a WAB Turbula T2C shaker/mixer with an amount of magnesium stearate which would result in a 0.5% w/w concentration of lubricant in the final blend. The materials were then compressed on the Stokes B-2 tablet press, operating at the speed of thirty revolutions per minute using four of the sixteen compressing stations on the press.

Punch Displacement

Tablet compaction on a rotary machine is effected by a pair of punches running between two rollers. On the Stokes B-2, the upper roller is fixed and the lower pressure roller is raised or lowered to control the amount of lower punch travel and hence the compaction pressure. The processes occurring at the upper and lower rollers are considered to be similar. The movement of the punch will be dependent upon: (1) the height of the roller above the cam track, (2) the radius of the pressure roller and (3) the profile of the punch head. The punch speed will then be determined by this movement and the angular velocity that the turret is turning.

Because of the difficulty in mounting a linear variable differential transformer (LVDT) on the press and acquiring the signal telemetrically, it was determined that actual measurement of punch movement was not economically feasible. Instead, punch position, as a function of time, was calculated from tablet press dimensions of the Stokes B-2. Manufacturing specifications of the flap radius (also known as the circle of dies radius), compression

TABLE I

Measurements of Several Tablet Press Dimensions (of the Stokes B-2) Needed to Calculate Punch Displacement

Parameter	Measurement
Pressure roll radius	101.60 mm
Flap (or circle of dies) radius	114.30 mm
Punch head flat radius	6.35 mm
Punch head curvature radius	9.73 mm

roller radius, and the radius of the flat section on the head of the tablet punch were obtained from the machine's manufacturer. Table I shows the tablet press dimensions which were obtained from the manufacturer of the Stokes B-2 machine. Additional measurements needed included punch diameter, and velocity of the tablet press rotation. Fig. 1 show the measurements used in the calculation and the resultant parameters.

The center of the punch head radius was used as the reference level for making calculations of the height of the punch, then the position of the punch before the punch reaches the roller will be r_c. Trigonometry allows the calculation of a third side of a triangle if the other two sides are known. Therefore, the vertical displacement of the upper punch, Z, as it passes under the compression roller is:

$$Z = [R^2 - X^2]^{1/2} \qquad \text{[Eqn 1]}$$

(A)

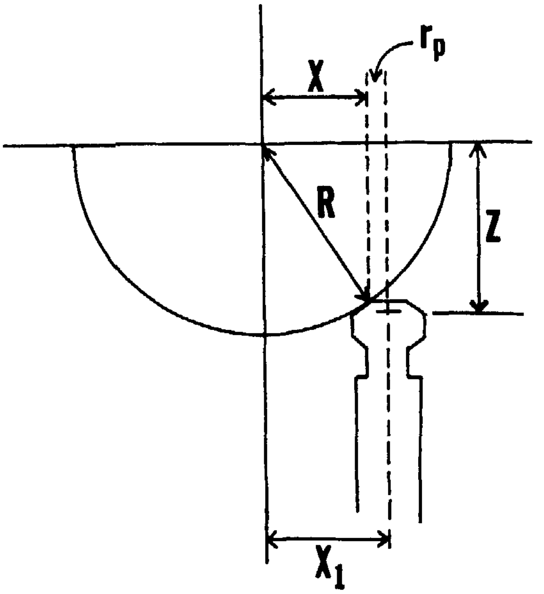
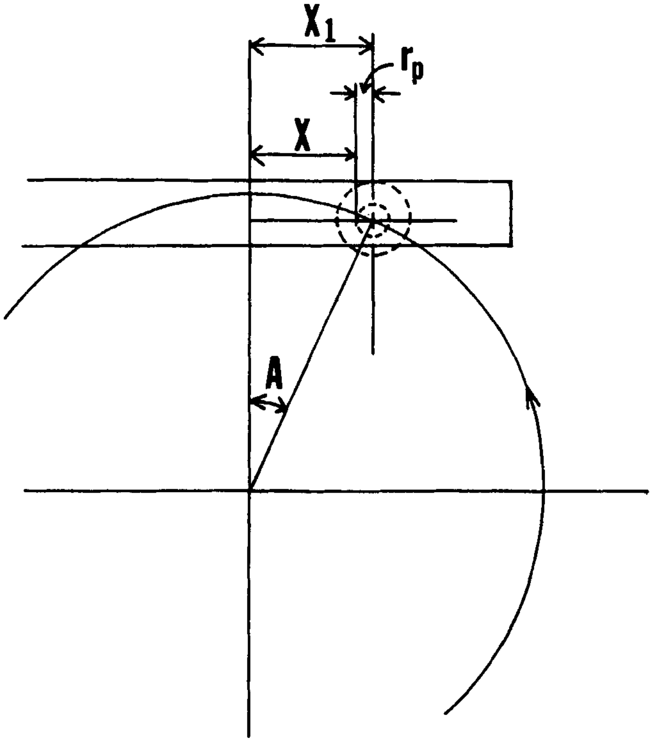


FIGURE 1

Diagrams of the geometric relationships between (a) the center of the pressure roll and the center of the upper punch, (b) the angle A, the punch and pressure roll; and (c) the punch head curvature and punch head flat radius.

where R is the sum of the radii of the compression roller and X is the horizontal distance between the vertical center line of the compression roller and the near edge of the flat portion on the head of the upper punch (Fig. 1c). This distance can be redefined as the distance to the vertical center (X_1) of the upper punch

(B)



(C)

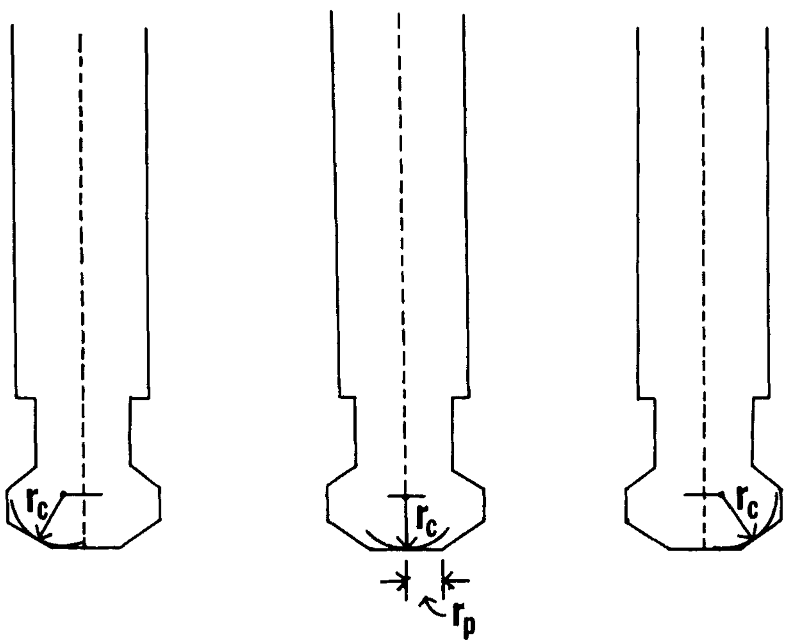


FIGURE 1. continued

minus the radius of the punch head flat (r_p):

$$X = X_1 - r_p \quad \text{[Eqn 2]}$$

The distance that the punch is from the centerline of the compression rollers is a function of the horizontal angle, A:

$$X = r_d \sin A - r_p \quad \text{[Eqn 3]}$$

where r_d is the flap radius (or the radius of the circle of dies), r_p is the radius of the punch head flat.

The horizontal angle, A, measured from the punch axis to the vertical centerline of the roller is a function of the turret angular velocity, w, and the time of compression. Thus from substituting Equation 3 into Equation 1, the upper punch displacement at any time prior to the position of greatest penetration into the die is:

$$Z = [(R + r_c)^2 - (r_d \sin(wt) - r_p)^2]^{1/2} \quad \text{[Eqn 4]}$$

The above equations hold true until the roller comes in contact with the punch head flat, at which point the height of the punch will be constant, until the opposite curved lip of the punch comes into contact with the compression roller and the punch drops away from the compact. The position of the punch at maximum displacement will be:

$$Z = R + r_c \quad \text{[Eqn 5]}$$

The time at which the punch flat comes into contact with the pressure roller will be a function of the angular velocity, flap radius and the radius of the punch flat. This occurs at a particular time interval prior to the distance between the vertical centers of the pressure roll and punch of becoming zero. This time interval (t_c) can be calculated mathematically using:

$$t_c = \arcsin (r_p/r_d)/w \qquad \text{[Eqn 6]}$$

If we know the time point at which the maximum force (t_f) is obtained, and are able to calculate the time at which maximum displacement (t_d) occurs, $t_d = t_f + t_c$, then Equation 4 can be simplified to:

$$Z = [(R + r_c)^2 - (r_d \sin(w(t_d-t))^2]^{1/2} \qquad \text{[Eqn 7]}$$

When $t > t_f$, the punch head flat is running over the roller, and the vertical punch velocity and the punch displacement are constant at 0 ms^{-1} and Z mm respectively. While the punch flat is traveling over the roller, the punch displacement is constant, for a time period, t_m, calculated from any of the following equations:

$$t_m = r_p/ \quad r_d f \qquad \text{[Eqn 8]}$$

$$t_m = r_p/ \quad r_d(s/60) \qquad \text{[Eqn 9]}$$

$$t_m = 2r_p/V_h \qquad \text{[Eqn 10]}$$

where the terms are defined as:

f = the frequency of turret rotations per second

s = the number of revolutions per minute

V_h = the horizontal speed of the press (in units of millimeters per second).

(A)

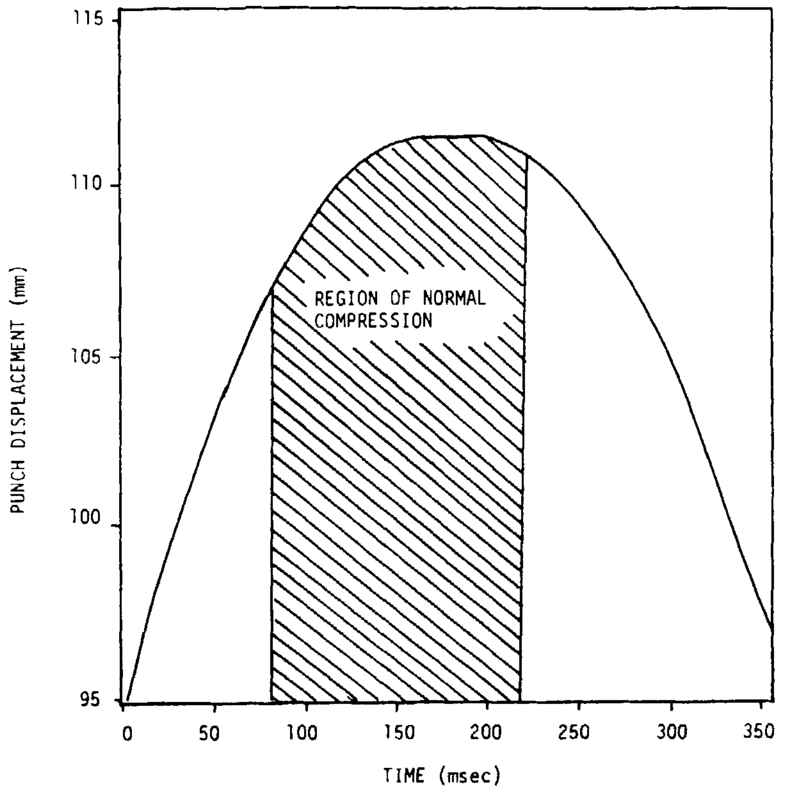


FIGURE 2

Typical profiles of (a) upper punch displacement and (b) upper punch vertical velocity calculated from Equations 4 and 11.

The theoretical profile of punch displacement is shown in Fig. 2a.

If we try to calculate the punch diplacement using Equation 3, and not take the punch flat dimension into consideration than the calculated displacement of the punch will actually seem to decrease because of r_d sin (wt) > r_p. It is physically

(B)

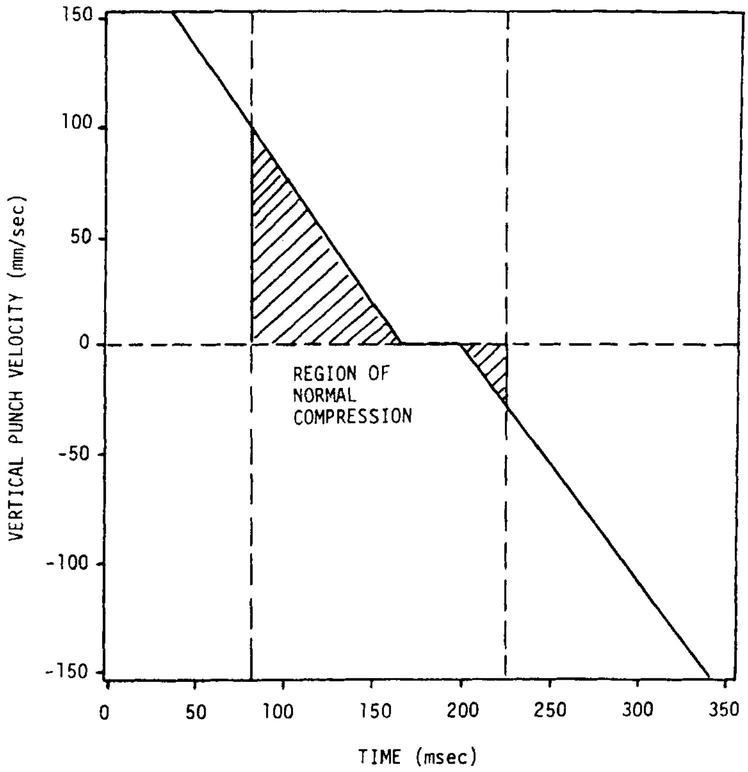


FIGURE 2. continued

impossible for the punch displacement to decrease at this moment, and therefore we must use Equation 5 when $t_f < t < (t_f + t_m)$, to calculate the punch displacement.

The derivation of the above mathematical expressions to calculate upper punch position is similar to that used by Danielson and Rippie [ ], but with several differences. The major difference exist in the second term used in Equation 3. In calculating punch position, Danielson and Rippie erronously used a term which they defined as "the horizontal distance between the vertical center of the compression roller and upper punch, respectively, and the center of vertical curvature of the punch head rim" [3]. The use of this term instead of the radius of the punch head flat will reduce the time span that the upper punch is at maximum displacement, and also place error into the calculation of the punch position.

The vertical velocity of the punches can be calculated from the derivative of Equation 7 versus time. This yields the following differential equation:

$$dZ/dt = 1/2\ [(R + r_c)^2 - (r_d \sin(wt) - r_d)]^{-1/2} * (-2(r_d \sin(wt) - r_p) * (r_d \cos(wt)) * w \qquad \text{[Eqn 11]}$$

A plot of this function is shown in Fig. 2b.

The analysis of the compaction process on a rotary tablet press is complicated further by the relative movements of the two punches. If one punch travels a considerable distance farther than the other, then the compaction process will be harder to specify exactly. However, it is common to assume that the movement of the lower punch upward by the lower roller will not affect the compaction process until the upper punch is in contact with the powder bed and the upper pressure roller. At this point, the two punches move equal amounts in opposite directions [3], and this greatly simplifies any calculations to be made.

RESULTS

The energy used during the compaction of powders can be calculated from punch force and punch displacements. Energy in compaction can be used in particle rearrangement, interparticle friction, die-wall friction, elastic deformation, fragmentation of particles and formation of bonds. Typically, the amount of work calculated from a simple force-displacement measurements can be divided into several processes: compaction work, work recovered in the form of elastic work (work performed by the compact on the tablet press), and the net work of compaction. The net work is quite simply the total work of compaction minus the elastic work. A typical force-displacement curve is shown in Fig. 3.

Using the equations above, the force-time curve was divided into three segments according to the movement of the upper punch: compression, force relaxation, and decompression. The compression phase is defined as the segment of the actual compaction of the powder to the maximum compression force. The relaxation phase is best explained as the time period where the punch displacement is held constant for T_m time during which we observe a decrease in the measured compression force. Finally, the decompression (or recovery segment) is the period where the upper and lower punches are allowed to move away from the compact and the punch displacement will decrease. Fig. 4 illustrates the segmentation of the compression force-time curve. Accordingly, the areas under the various segments of the force-time curve will be used as parameters for relating the work calculated to various areas.

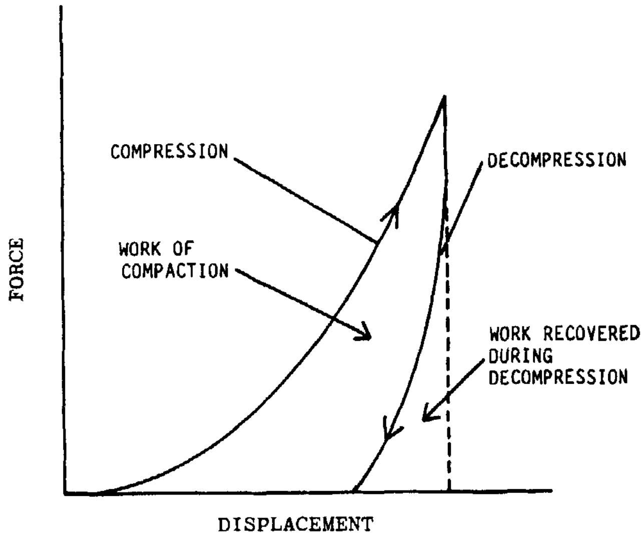


FIGURE 3

Typical force-displacement curve with the measurements obtained form it.

Several assumptions are made when segmenting the force-time curve: (a) that the maximum compression force represents the time at which the punches first reach maximum displacement, t_c, and (b) that the force relaxation segment will contain some elastic work done by the compact on the tablet press.

A computer program (FT-FD) was written in BASIC computer language for use on the Apple II series of personal computers. The program inputs the compression force data from a sequential data file stored on floppy disk and calculates the punch displacement according to the time prior to the point of maximum compression force, also assumed to be the point of maximum

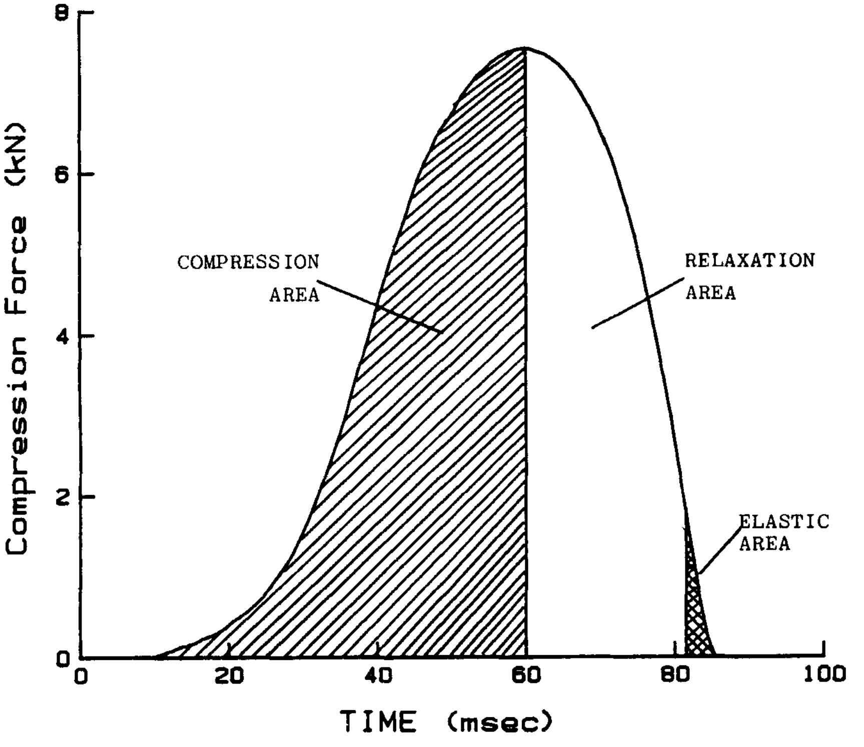


FIGURE 4

Compression force-time curve divided into the segments (or phases) of compression.

displacement. The program uses the equations shown above. The user is able to chose several options of output of the data: results of the segmentation analysis, force-displacement analysis, and displacement-time analysis. The results of the force-displacement analysis and displacement-time analysis can be stored to a disk so that they can be recalled by a graphics program and plotted.

```
]RUN
******** FILE TO BE ANALYZED ********
ENTER FILENAME: MEAN.EA
SLOT <6>: 6
DRIVE <2>: 2

PEAK= 6.69 AT 100 MSEC

****** INPUT PRESS DIMENSIONS ******
DEFAULT <VALUES> ARE FOR STOKES B-2
OPERATING AT 30 RPMS
(Press RETURN to keep default value

R (compression roll radius) <101.6>: 101.6

Rc (punch head radius) <9.73>: 9.73

Rd (flap (die) radius) <114.3>: 114.3

W (turret angular velocity) <180>: 180

Rp (punch radius) <6.35>: 6.35
-----------------------------------------
Time at Max. Displ.= 35.3677612 msec

DISPLAY RESULTS ON S(CREEN) OR P(RINTER
P


FILENAME: MEAN.EA
-----------------------------------------
PEAK OF MEAN CURVE=  6.69 kN
  PEAK TIME= 100 msec
FORCE AT RELAX.= .11 kN
  RELAX. TIME= 135.3 msec

NET WORK DONE =      9.229 kN-mm
COMPACTION WORK =    9.229 kN-mm
ELASTIC WORK =       0 kN-mm
AREA OF F-T CURVE = 361.91 N-sec
COMPRESSION AUC =    199.95 N-sec
RELAXATION AUC =     161.96 N-sec
DECOMPRESSION AUC = 0 N-sec


DO YOU WANT PRINT OUT OF THE
DISPLACEMENT RESULTS (Y/N)? Y
```

FIGURE 5

Example of the output obtained from the FT-FD,

force-time/force-displacement conversion program

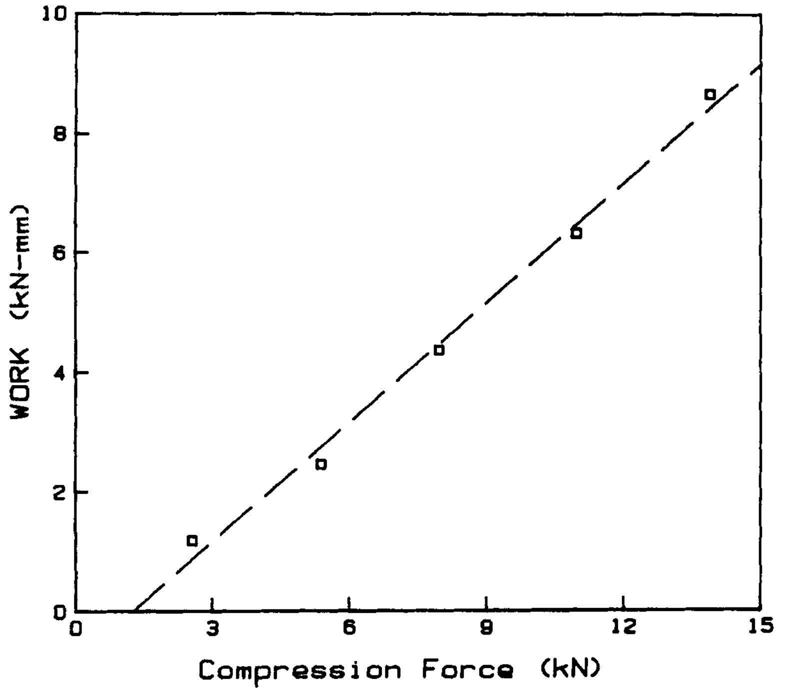


FIGURE 6

Typical plot of the total compaction work versus the compression force. This plot was obtained from the conversion of force-time data to force-displacement data

After calculating punch displacement, the FT-FD computer program will calculate the areas under the segmented compression force-time curve and the values of work performed. A sample output from this program appears in Fig. 5.

The amount of elastic work recovered was negligible in most of the tablet compressions. Fig. 6 shows a typical plot of the total work of compaction calculated versus the height of the

TABLE II

Results of Regression Analysis Results of Data from Force-time and Force-Displacement Calculations including those of the Segmented Curve.

Material	Slope*	Intercept*
Anhydrous Lactose	41.80 (1.71)	9.0 (10.9)
Avicel PH-102	47.71 (2.68)	-12.4 (14.1)
Emcompress	44.86 (2.04)	20.4 (10.9)
Starch 1500	44.45 (1.82)	14.7 (10.9)

*Note: Standard errors of the estimates are in parentheses.

compression curve. Table II shows the results of all the compactions and their mean segmented areas and amounts of work calculated. As can be determined from this table, all of these work-force relationships possessed a coefficient of determination greater than 0.99 and therefore were highly significant.

The areas under the total and segmented compression force-time curves are plotted in Fig. 7. As can be seen in these plots, all the relationships were very linear.
This observation was not quite expected, and raised some questions about which of the areas would be more appropriate to compare to the amount of work performed. It was hypothesized that the Compression Area would most closely be correlated with the total

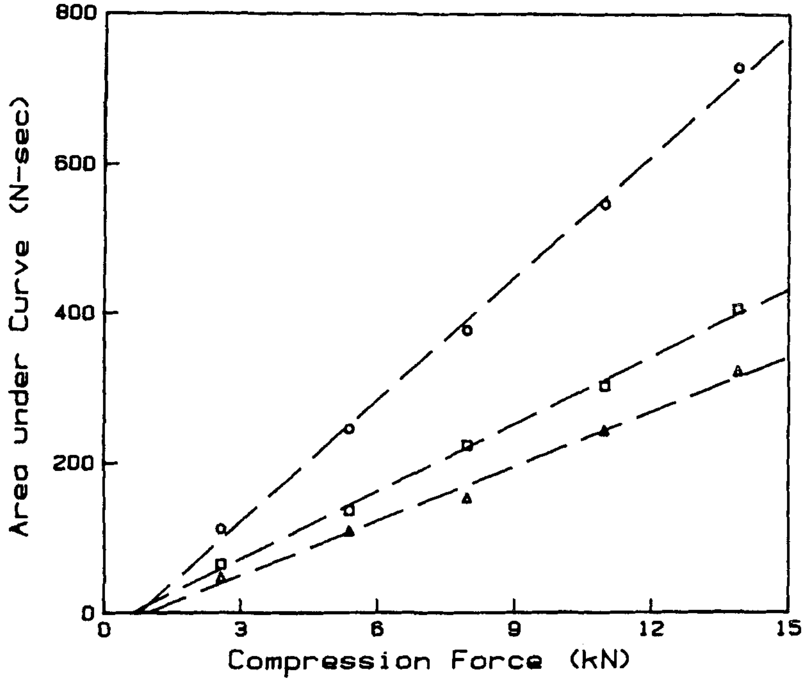


FIGURE 7

Plots of the segmented areas, (O) total, (□) compression and (Δ) relaxation, of the compression force-time curves against the compression force for Emcompress.

amount of work done. The area representing the force relaxation (or unloading) phase corresponded to no amount of work performed since it is assumed that the upper and lower punches do not move during this phase. As mentioned previously, the amount of elastic work calculated and the area under the decompression segment of the force-time curve were negligible. This observation raises the question if any elastic work could be recovered during the force relaxation phase as a result of the action of the overload spring.

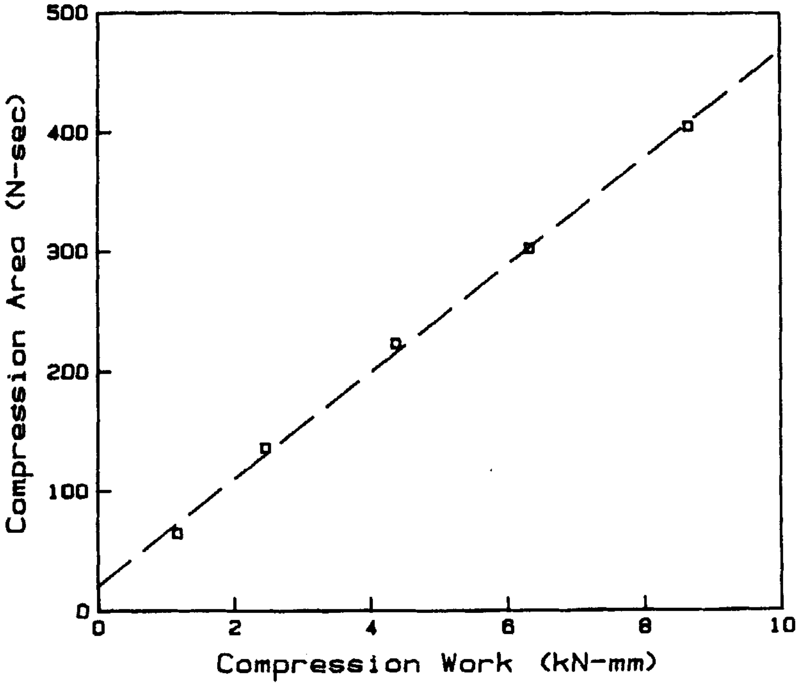


FIGURE 8

Plot showing the linear relationship of the Compression Area and the Total Compaction Work for Emcompress.

If this is so then, the amount of total work performed on the powder would be the only portion of work that could be correlated to the area under the compression segment. This relationship is one of several illustrated in Fig. 8 for Emcompress. The correlation of the relationship for this material and the other materials was better than 0.99.

The question arose if each material had a relationship between the area under compression segment and the total work that was material dependent or independent. Fig. 9 shows a plot of the

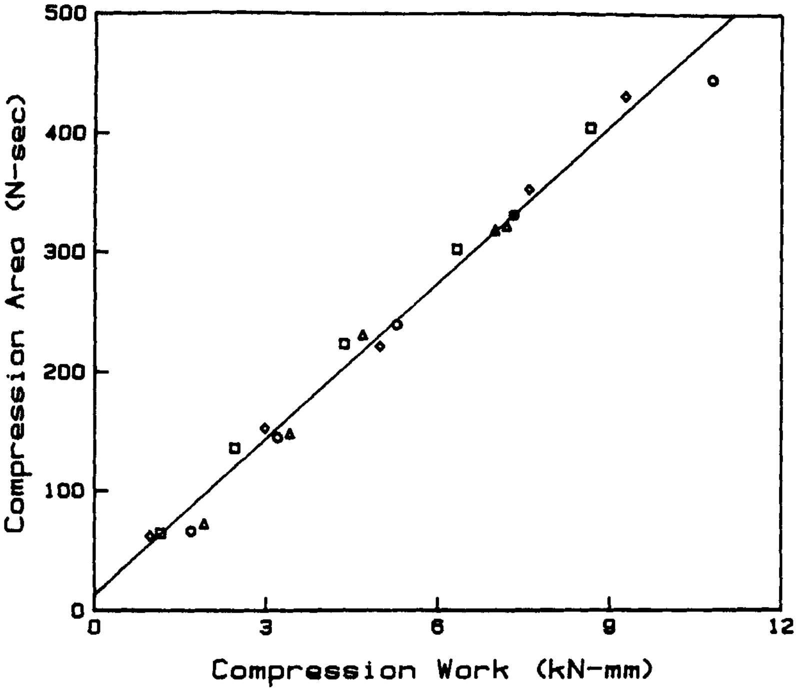


FIGURE 9

Plot of Compression Area as a function of Total Compaction Work for (△) Avicel PH-102, (□) Emcompress, (◇) anhydrous lactose and (○) Starch 1500. A single regression line has been drawn for all the experimental results.

mean values for all four materials with only one regression line going through the experimental points. This regression line showed excellent correlation and it was decided to test for the heterogeneity of slope of the relationships between materials. Using the SAS statistical software package, a heterogeneity of slope model was constructed in the general linear models procedure

TABLE III

Results of a Heterogeneity of Slope Model*

Analysis Using the GLM Procedure of SAS

SOURCE	DF	SS	MS	F	PR>F
Material (intercepts)	3	543.6	181.2	1.1	0.3481
Height (due to regression)	1	271240.4	271240.4	1811.5	18E-15
Height*Material (slopes)	3	566.3	188.8	1.3	0.3317
ERROR	12	1796.8	149.7		
CORRECTED TOTAL	19	301977.3			

R-SQUARE 0.9941

*Note: The model specified in the analysis was:

MODEL CAREA = MATRIX TWORK TWORK*MATRIX

which would test for differences in slopes and intercepts. The results of this procedure appear in Table III, and prove that there is no statistical significance between the slopes and the intercepts. It thus appears that the relationship between the amount of work done and the area under the compression segment of the force-time curve is independent of any material effects, and may well be a machine dependent effect.

The tableting differences seen between the single punch machine and the rotary tablet press may be the result of the extended "dwell time" of the rotary tablet press. Extended "dwell time" implies that the compression force is applied at the maximum level for a longer period of time. This would allow plastic flow in any of the components of the material to exhibit plastic properties and absorb the energy of elastic strain recovery before the force is released. *This may be sufficient to prevent the* excessive strain relaxation in the compact which is considered to be a cause of capping [5]. As observed, if any plastic flow occurs, the value of the force on the punch will not be held constant during this period, but rather will decrease with time, as the compact is held at a constant volume. Thus this portion of the segmented compression curve has been defined as the relaxation phase.

Many pharmaceutical scientists, through experience, know that a formulation may run satisfactory on the laboratory single punch machine but will show capping when compressed on the rotary tablet press. Charlton and Newton calculated that "contact" times to form the same dimensional tablet on a single punch machine and a rotary machine operating at similar cycling speeds [1]. They showed that even with the additional "dwell" time, the contact time of the rotary tablet press was only 83% of that for the single punch machine, and may not be sufficient to overcome the elastic strains built up in the compact. Hiestand has written that the a formulation of an elastic behavior drug should include a plastic acting excipient, which upon mixing with the elastic drug will result in a harder compact due to plastic deformation increasing the bonding surface area [5]. The resultant compact should withstand the release of stored elastic energy in the compact.

It should be noted that the calculations of punch displacement could be subject to some minor errors due to certain assumptions. The calculations have been made on the basis of no significant deformation of various components of the tablet press involved (e.g. punches, cams, etc.) and relief settings. A second assumption is that the upper punch of the rotary machine follows the profile of the upper roller. In practice, the upper punch is allowed to fall from the upper cam track onto the powder bed under its own weight. This occurs before the upper punch is running under the upper pressure roller. Thus there will be some tapping and precompression of the powder bed due to the weight of the upper punch, and a thinner sample than assumed will result.

Another premise is that the lower punch follows the same displacement profile as the upper punch. However, this is known not to be always an entirely precise premise since the position of the lower pressure roll in relation to the upper pressure roll is varied as the lower pressure roll is lowered or raised to adjust the compression force. The segmentation of the compression curve described in this paper, will still be essentially valid if the maximum compression force occurs at the point where the net displacement (of the upper and lower punches) is at its maximum value. Observations of force-time curves can be used, indirectly, to support this assumption. As the tablet press speed was increased, it was noted that the time at which the maximum compression force was observed appeared to be a linear function of the press speed. When the tablet press was operated at a press speed of 30 rpms (179.5 mm/sec), a maximum compression force was observed at 100 milliseconds into the compression data acquisition cycle. Then, when the press speed was adjusted to 60 rpms (359.0 mm/sec), a maximum compression force was observed at 50 milliseconds of the acquisition cycle. The time at which the maximum force was observed could be stated to be inversely proportional to the press speed, and this would support the assumption.

In efforts to gain data and opinions on the points discussed above, the authors contacted with several scientists involved in related research. Recent correspondence between J.R. Hoblitzell and Dr. T.M. Jones, of The Wellcome Foundation Ltd, England, indicated that his research group is in the initial stages of a comprehensive compaction study designed to clarify the relationship between force, time, and punch position. According to preliminary compaction data, obtained using a compaction simulator, Dr. Jones believes that there may be some differences between the type of materials and the occurrence of the peak compression force. The data indicates significant material dependence in the "lag time" and a possible relationship between it and the fundamental compaction mechansisms of the material.

The term "lag time" was defined by J.R. Hoblitzell, in his correspondence to Dr. Jones, as a time delay between the occurrence of maximum displacement and the appearance of the maximum compression force. This effect probably certainly result in some significant error in the above assumption for some systems. Dr. Jones emphasised that these findings were preliminary results, and that more comprehensive conclusions are still a year or so away.

In a recent telephone conversation, J.R. Hoblitzell discussed the same premise with Dr. L.L. Augsburger, of the University of Maryland [6]. Recently, Dr. Augsburger and his coworkers have instrumented a tablet press to enable the direct measurement of punch displacement [7]. Dr. Augsburger indicated that he subscribed to the view that the time of maximum force was very close to, if not identical with, the time at which maximum upper punch displacement was observed. A perusal of recent experimental data published by Dr. Augsburger, suggests that the nature of the punch displacement-time and powder bed thickness-time curves are somewhat complex [6,7]. These results would seem to support the authors' assumption, that if minor differences do exist, it seems probable that the overal effect of the error introduced by the above assumptions will not be large.

CONCLUSIONS

It is premature to make a definitive conclusion regarding the relationship of Area under the force-time and Work obtained from the force-time curves. Yet, the results of this study clearly show a good correlation between Area and Work.

The results show that calculations of upper punch displacement can reliably be done but it must be noted that due to the position of the lower pressure roll not being fixed it is much less reliable to calculate the displacement of the lower punch. A study using radio-telemetric devices to record the exact position of the upper and lower punches will be advantageous and needed to clearly substantiate the conclusions reached here on the relationship between Area and Work.

ACKNOWLEDGEMENTS

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GLOSSARY OF TERMS

Z upper punch displacement from the center of the upper pressure roll

R the radius of the compression pressure roll.

X the distance to the edge of the upper punch's flat section on the head

X_1 the distance between the vertical centerline of compaction and the near (flat) edge of the punch head.

r_p the radius of the punch head flat

r_d the radius of the die flap (the circle of dies)

A the angle of the vertical centerline of compaction and the center of the punch.

w the angular velocity of the turret rotation

r_c the radius of the punch head curvature

t time of the compaction cycle

t_d the time during the compaction cycle at which the center of the punch is in line with the centerline of the compression pressure rolls.

t_c the time period, prior to t_d, where maximum displacement of the upper punch occurs

t_f the time during the compaction cycle at which maximum force is achieved ($t_f = t_d - t_c$)

t_m the length of time at which geometric maximum displacement is maintained (equivalent to $2t_c$)

f the frequency of turret rotations (rotations per second)

s the number of turret revolutions per minute

V_h the horizontal turret velocity

V_v or dZ/dt is the vertical punch velocity

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