Calibration of a Compaction Simulator for the Measurement of Tablet Thickness During Compression

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For the calibration of a compaction simulator for punch displacement measurements, the displacement of the punch must be related to the voltage output of a linear variable displacement transducer (LVDT) which is attached to the punch via its movable core, with correction for any deformation of the machine parts which are inherently incorporated in the LVDT readings. Contrary to common assumptions the relationship between the displacement of the movable core and the voltage output of the LVDTs used is not linear. Similarly, the deformation of the machine parts did not follow Hooke's law of linear elasticity but exhibited characteristics of nonlinear elasticity. The data demonstrate the need for careful validation of the calibration of a compaction simulator when accurate punch displacements are required.

KEY WORDS: linear variable displacement transducer (LVDT) calibration; punch deformation; compaction simulator; nonlinear elasticity.

INTRODUCTION

Powder compaction is governed by multiple factors. The roles the different factors play in powder compression and consolidation have been studied via the collection of force-displacement data during the entire compression cycle on instrumented single-station presses (1–6), multistation presses (7–12, and, more recently, compaction simulators (13–17). Force is commonly measured with strain gauges or piezoelectric elements, whereas punch displacement is usually monitored with linear variable displacement transducers (LVDT).

The displacement of the punch tips from a reference position can be monitored with LVDTs attached to the upper and lower punches of a press or compaction simulator. This facilitates the determination of the separation of the punch faces at any point in time. During compression the distance between the punch faces corresponds to the thickness/height of the powder mass, since the punch tips are in contact with the powder mass. Thus, changes in volume, porosity, and solid fraction of the particulate material can be deduced from the punch displacement measurements.

Machine parts undergo elastic deformation during compression. Such elastic deformations are incorporated in the LVDT readings, thus introducing an error in the thickness of the tablet determined from the raw LVDT data. The extent of the error depends on the position of attachment of the LVDT to the punch. The nearer the LVDT is attached to the punch tip, the less the degree of machine part deformation that will be incorporated into the LVDT readings (18). Whatever the point of attachment of the LVDTs to the punches, the goal is to compensate for the error introduced by the deformation of the machine parts.

The use of an LVDT for displacement measurement during compaction experiments involves the following: (i) establishing the relationship between the displacement of the LVDT rod and the corresponding voltage output of the transducer and (ii) correcting for the elastic deformation of the machine parts. For compaction simulators, which additionally use LVDTs as an aid to control punch movement, accurate calibration of the LVDT is critical to its proper function. If the calibration is inaccurate, the compaction simulator will not accurately follow the displacement–time profile fed to it by the microcomputer, thus defeating the purpose for which it is being used, namely, to mimic the displacement–time profile of a tableting machine.

The name "linear variable displacement transducer" suggests that a linear relationship exists between the displacement and the voltage output over the range specified by the manufacturer. The present work demonstrates that this is not always the case and the accuracy of the calibration is reduced by assuming linearity. Further, although the deformation of the punches and machine parts, in the range of forces normally used in powder compression, is reversible and instantaneous, the relationship between the deformation and the applied force is not necessarily linear. Hence the use of Hooke's law to describe the elastic behavior might lead to erroneous results.

METHODS

Determination of Displacement—Voltage Output Relationship of the LVDTs

The LVDTs used (Model D5/500, RDP Electronics Ltd., Wolverhampton, England) were specified by the manufacturer to have a linear range of ±12.5 mm, i.e., 12.5 mm on each side of the zero setting, giving a distance range of 25 mm. Figure 1 illustrates how the LVDTs were attached to the punches of the compaction simulator (Mand Testing Machines Ltd., Stourbridge, West Midlands, UK). The compaction simulator has been described in detail by Celik and Marshall (15). The LVDTs were calibrated *in situ,* i.e., in their mounted positions on the upper and lower punches of the compaction simulator, one at a time. A dial micrometer was used to measure the movement of the LVDT cores. The dial micrometer consisted of a magnetic base and a spring-loaded rod whose displacement effected a corresponding deflection of the dial pointer to indicate the exact distance the rod moved. The micrometer was attached to the frame of the compaction simulator, in close proximity to the LVDT, via the magnetic base. The micrometer rod was then aligned in the same axial plane as the core of the LVDT and adjusted such that its tip made contact with the LVDT core. As a

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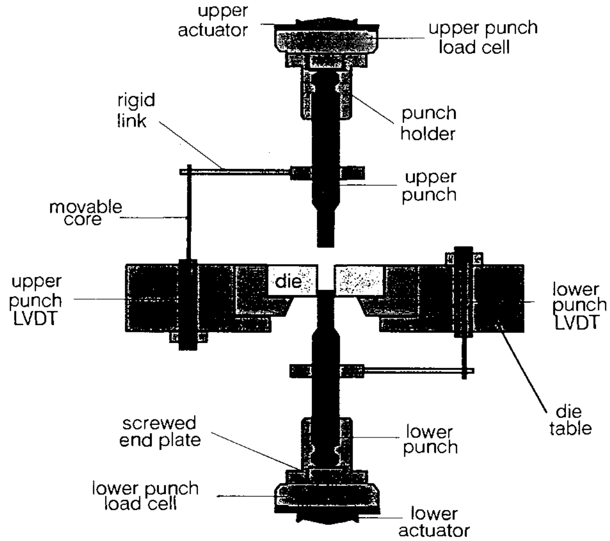

Fig. 1. An illustration of the attachment of the LVDTs to the punches.

result, the micrometer rod moved the same distance as the LVDT core.

The spring-loaded micrometer rod offered the advantage that the punches and therefore the LVDT core could be moved randomly as opposed to being displaced in increasing or decreasing magnitudes. The values so obtained were consequently not autocorrelated but were statistically independent of each other. A voltmeter, which was incorporated into the compaction simulator, was used to measure the voltage output.

The punches (and consequently the LVDT cores) were moved randomly according to a list of random numbers obtained from a random number table (19). The distances moved by the LVDT core and the corresponding voltage output were noted. The cores were moved over a distance of about 20 mm since the maximum envisaged compaction simulator punch stroke during compression in our laboratories was in the range of 15 to 20 mm. A minimum of 80 pairs of data was collected for each LVDT.

Determination of the Elastic Deformation of the Punches and Other Machine Parts

Using a "manual mode" (i.e., operator controlled), the upper and lower punches were moved toward each other until the punch tips came into contact. Contact was considered to be established when a force of circa 20 N was exerted on the punches. The force between the punches was manually increased while the upper and lower punch force signals as well as the corresponding upper and lower punch LVDT voltage readings were recorded on a digital oscilloscope (Nicolet 4094, Nicolet Instrument Corp., Marlton, NJ). The LVDT readings therefore correspond to the punch and machine parts deformation. The data were transferred to a VAX mainframe computer and converted into forces and distances using calibration constants previously determined. Three replicate determinations were carried out.

Data Analysis

An important step in the calibration procedure is the fitting of a function to the independent and dependent variables of calibration data collected with the aim of predicting the values of the independent variable by interpolation. We normally form an opinion by visual examination of a scatter plot of the dependent and the independent variables as to the function which will accurately fit the data collected for calibration purposes. The function which appears to fit the data should not be blindly accepted but checked for its validity. An inaccurate function would furnish erroneous predictions of the independent variable.

A check for the correctness of a postulated model (linear or nonlinear) involves the analysis or examination of residuals (20). The residuals, e_i, are the difference between what is actually observed and what is predicted by the regression equation and may be described mathematically as follows:

$$e_i = Y_{i(\mathrm{obs})} - Y_{i(\mathrm{pred})}, \qquad i = 1,2, \ldots, n \tag{1}$$

where

$Y_{i(\mathrm{obs})}$ = an observation
$Y_{i(\mathrm{pred})}$ = the corresponding fitted value obtained by use of the fitted regression equation

In performing any regression analysis, the following assumptions are made with regard to the residuals (20): (i) the residuals are independent, (ii) the residuals are random and have a zero mean, (iii) the residuals have a constant variance; and (iv) the residuals follow a normal distribution. Thus, if our fitted model is correct, the residuals should exhibit tendencies that confirm the assumptions made, or at least, not exhibit a denial of the assumptions.

The several ways of analyzing residuals are all graphical and revealing when the assumptions are violated (20). In this work a plot of the residual against the independent variable is used for the residual analysis.

The statistical analysis of the data in this work was done with the statistical program RS/1 (BBN Software Products Corp., MA).

Using the correlation coefficient as a check on the linear dependence of one variable on another is incorrect. The correlation coefficient is a measure of the degree of correlation of two variables which are linearly related and is not, by itself, a measure of linearity. In other words, the value of a correlation coefficient shows only the extent to which X and Y are linearly associated (20,21).

RESULTS AND DISCUSSION

The relationships between the displacement of the movable core and the voltage output of the LVDTs attached to the upper punch of the compaction simulator are shown in Fig. 2. The profile is also typical for the lower-punch LVDT. On casual examination of the plots, it appears that the variables are linearly related. The data were thus checked to verify whether a simple linear model in the form

$$Y = b_0 + b_1 X \tag{2}$$

where

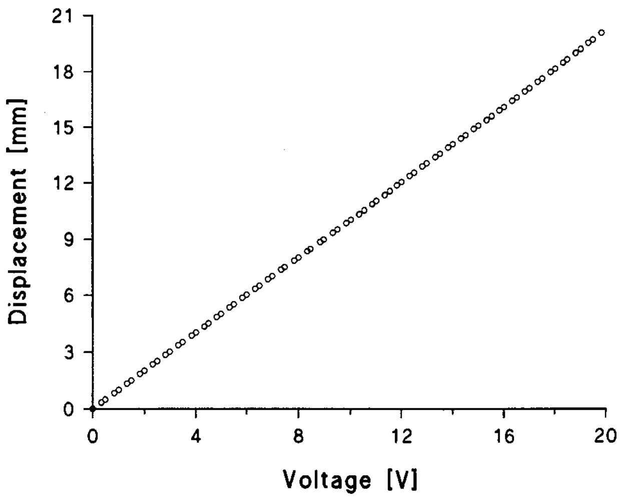

Fig. 2. Relationship between the displacement of the movable core and the voltage output of the LVDT attached to the upper punch.

Y = dependent variable (distance)
X = independent variable (voltage)
b_o, b_1 = constants

can be fitted to them.

The residual plot of the simple linear model for the upper-punch LVDT data set is shown in Fig. 3. The lower-punch LVDT data set exhibits a similar profile. The residual plots exhibit trends suggesting that the model must be rejected. This being the case, it is not correct to calculate regression parameters or the coefficient correlation based on Eq. (2) (20,21). If, however, the regression parameters were calculated based on Eq. (2) in spite of its being an improper statistical practice, Table I, depicting a correlation coefficient of 1.000, would have been obtained for both the upper- and the lower-punch LVDT data sets. Thus using the correlation coefficient as a measure of linearity is misleading.

The trends exhibited by the residual plots suggest that either extra terms are needed in the model or the dependent variable must be transformed (20). Consequently, a quadratic term was added to the model listed as Eq. (2) to yield a second-degree polynomial. That is,

Table I. Coefficient Table for Displacement–Voltage Data of the Upper-Punch LVDT Fitted with the Model $Y = b_0 + b_1X$

Term	Coefficient	SD	T value	Significance
b_0 (intercept)	−0.0202	0.0046	−4.366	0.000
b_1 (slope)	1.0065	0.0004	2503.059	0.000
			R^{2a} = 1.000	

[a] The multiple correlation coefficient, R^2, squared measures the proportion of the total variation about the mean of the dependent variable explained by the regression (20). For straight-line relationships the multiple correlation coefficient, R, is equal to the correlation coefficient (20).

$$Y = b_0 + b_1X + b_2X^2 \tag{3}$$

where b_0, b_1, and b_2 are constants.

The residual plot for the upper-punch LVDT data set for Eq. (3), which is similar to the lower-punch data set, is illustrated in Fig. 4. The residual plots from the upper- and lower-punch data sets using the second-degree polynomial show trends revealing that this model also does not adequately describe the relationship between the data sets. Consequently, a cubic term was added to Eq. (3) to render the model a third-degree polynomial in the following form:

$$Y = b_0 + b_1X + b_2X^2 + b_3X^3 \tag{4}$$

where b_0, b_1, b_2, and b_3 are constants.

The residual plots obtained by fitting the third-degree polynomial to the upper- and lower-punch LVDT data sets, as illustrated in Figs. 5a and b, respectively, show randomness, and therefore it can be concluded that the data sets are adequately described by the third-degree polynomial model. The estimates of the regressional parameters for this model [Eq. (4)], together with their standard errors, the corresponding T values (t statistics) for testing whether the true value of the coefficient is equal to zero, and the significance level for each T value for both the upper- and the lower-punch LVDTs, are listed in Tables II and III, respectively. It is shown that the regressional constants in the model are all significant ($P < 0.05$) except the y intercept, b_0, which is not significantly different from zero ($P > 0.05$); i.e., $b_0 = 0$. The

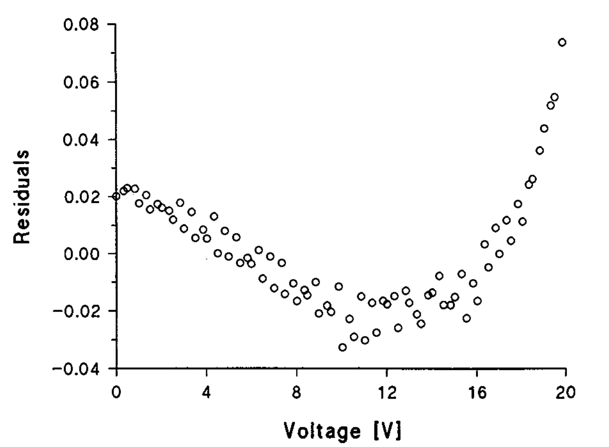

Fig. 3. Residual plot for the displacement of the movable core as a function of the voltage output of the upper-punch LVDT fitted with the model $Y = b_0 + b_1X$.

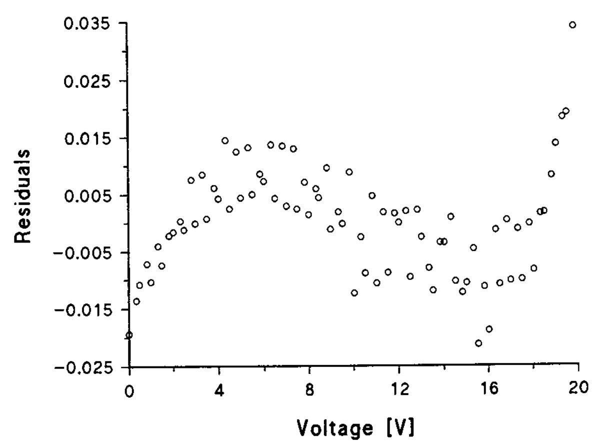

Fig. 4. Residual plot for the displacement of the movable core as a function of the voltage output of the upper-punch LVDT fitted with the model $Y = b_0 + b_1X + b_2X^2$.

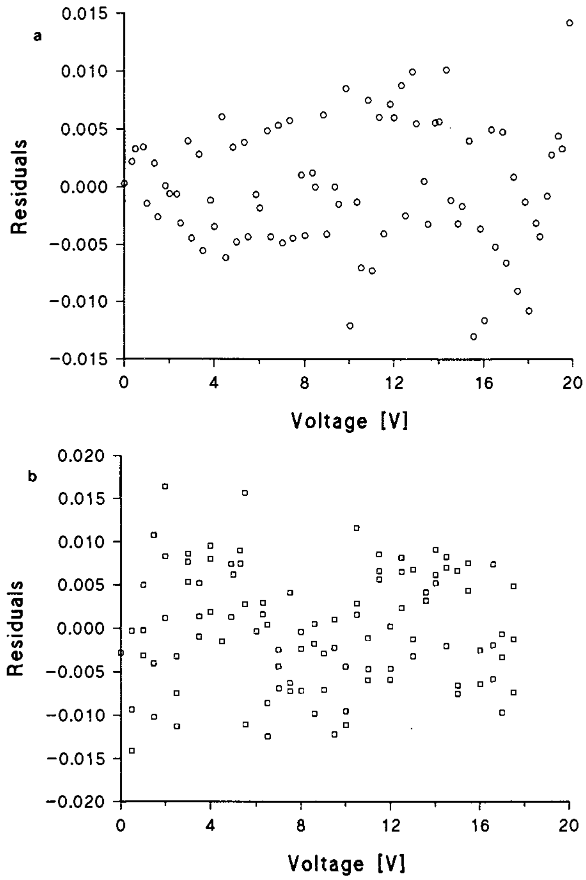

Fig. 5. Residual plots for the displacement of the movable core as a function of the voltage output of the LVDTs fitted with the model $Y = b_0 + b_1X + b_2X^2 + b_3X^3$. (a) LVDT attached to the upper punch; (b) LVDT attached to the lower punch.

high significance of the b_2 and the b_3 terms strongly indicates that there is curvature in the relationship. The nonsignificance of the y intercept, b_0, of Eq. (4) is logical and expected, since no change in voltage should be associated with a zero change in displacement. The y intercept of Eq. (2) in comparison is illogically significantly different from zero (see Table I).

Thus, in spite of the fact that the LVDTs were calibrated in the range of −10 to +10 mm, which is a narrower range than the manufacturer's specified linear range of −12.5 to +12.5 mm, the residual analysis indicates that the simple linear model in the form of Eq. (1) does not adequately describe the relationship between the displacement of the core and the voltage output of the LVDTs over the range calibrated.

In some ranges, narrower than −10 to +10 mm, the simple linear model describes the displacement–voltage relationship adequately. For example, the lower-punch LVDT exhibited linearity [as described by Eq. (1)] in the ranges 0 to −10 and 0 to +10 V (that is, about 10 mm on either side of the zero setting of the core at which point the voltage is zero). The upper-punch LVDT, on the other hand, showed linearity in the range −10 to 1.9 V. Consequently, the appropriate model can be chosen depending on the range of interest. For single-punch and rotary multistation tableting machines, the range of particular interest is that during which the punches are in contact with the powder. In the case of a compaction simulator, however, the whole punch stroke is of interest, since the hydraulic actuators, and, therefore, the punches follow the displacement–time profile fed to them by the microcomputer with the aid of the LVDTs.

The magnitude of error in using Eq. (2) instead of Eq. (4) may not be large. Thus when a high level of accuracy is not desired, Eq. (2) may be used. The decision to use Eq. (2) must be taken in full awareness that (i) the function is not accurate and (ii) the LVDT calibration constants are used in determining the magnitude of the punch deformation. Consequently, any errors made in the LVDT calibration will be propagated into the punch deformation data. Thus the error incorporated in the tablet thickness values which can be traced to inaccurate LVDT calibration will be greater than just the error which results from using Eq. (2) instead of Eq. (4) for the prediction of the displacement of the LVDT cores.

Table II. Coefficient and Analysis of Variance (ANOVA) Tables for the Model $Y = b_0 + b_1X + b_2X^2 + b_3X^3$ Fitted to Displacement–Voltage Data Obtained from the LVDT Attached to the Upper Punch

Coefficients

Term	Coefficient	SD	*T* value	Significance
b_0 (intercept)	−0.00030	0.00241	−0.123	0.902
b_1	1.00658	0.00105	954.941	0.000
b_2	−0.00093	0.00012	−7.539	0.000
b_3	0.00005	0.00000	12.636	0.000

ANOVA

Source	SS	df	Mean SS	*F* value	Significance
Regression	2709.909	3	903.30293	2.9×10^7	0
Residual	0.002	76	0.00003		
			R^2 = 1.000		
SD of regression			= 0.006		

Table III. Coefficient and Analysis of Variance (ANOVA) Tables for the Model $Y = b_0 + b_1X + b_2X^2 + b_3X^3$ Fitted to Displacement–Voltage Data Obtained from the LVDT Attached to the Lower Punch

Coefficients

Term	Coefficient	SD	T value	Significance
b_0 (intercept)	0.00281	0.00268	1.048	0.297
b_1	0.95064	0.00130	733.883	0.000
b_2	0.00111	0.00017	6.588	0.000
b_3	−0.00002	0.00001	−3.059	0.003

ANOVA

Source	SS	df	Mean SS	F value	Significance
Regression	2564.700	3	854.900	1.9×10^7	1.39×10^{-17}
Residual	0.005	102	0.000		
			R^2 = 1.000		
SD of regression			= 0.007		

Punch Deformation

The total deformation of the punches and other machine parts incorporated into the LVDT readings as a function of the force applied is shown in Fig. 6. This deformation behavior of the punches and machine parts exhibits nonlinear elasticity characteristics, thus Hooke's law which describes linear elasticity is not applicable. The relationship between the machine deformation and the applied force is adequately described with a second-degree polynomial as confirmed by the residual plot depicted in Fig. 7. The corresponding ANOVA and coefficient tables are summarised in Table IV. The y intercept, b_0, is significantly different from zero. This is due to experimental error and reflects the difficulty of experimentally determining when contact has been established between the punches.

The nonlinear elastic behavior may be attributed to the following factors.

Variability of the Actual Area of Contact Between the Punch Faces and Between the Punch Heads and the End Plates of the Punch Holders with Applied Force. The punch faces are not perfectly horizontal. As a result, only a fraction of the surface areas touches when the punch faces first make contact. With increasing force and deformation, the area of contact increases. In the case of the punch heads, the surfaces are curved and not flat. Consequently, the contact area between the punch head and the end plate of the punch holder increases with increasing force on the punch. The dependence of the contact area between the punch faces and between the punch heads and the end plate of the punch holders on the magnitude of the applied force results in a nonlinear stress–strain relationship. This condition is similar to the stress–strain relationship of an elastic sphere on a flat elastic plate and can be described with Hertz's classical elasticity laws (22).

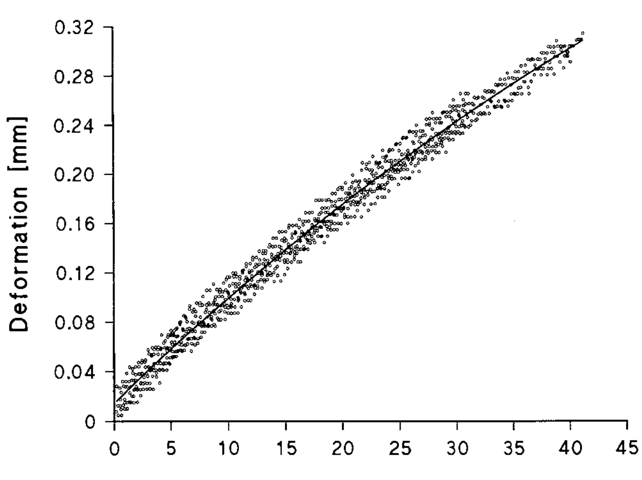

Fig. 6. Relationship between the total deformation of the punches and machine parts as a function of applied force. The data are a pool of four repeated experiments.

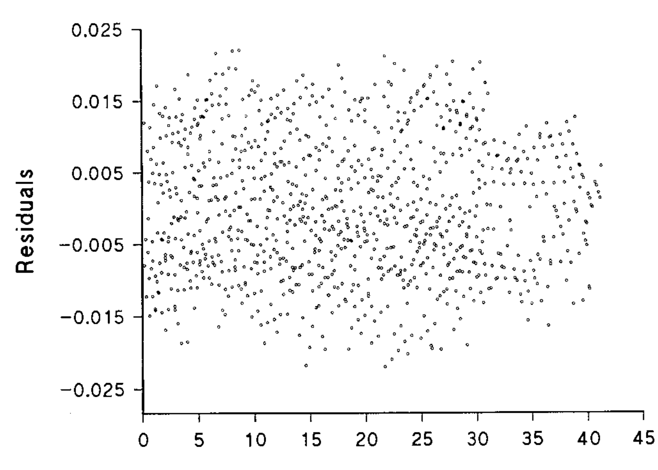

Fig. 7. Residual plot of the relationship of the total deformation of punches and machine parts as a function of the applied force fitted with a second-degree polynomial.

Table IV. Coefficient and ANOVA Tables for Total Deformation of Punches and Machine Parts as a Function of Applied Force Data Fitted with a Second-Degree Polynomial

Coefficients				
Term	Coefficient	SD	*T* value	Significance
b_0 (intercept)	0.01411	0.00090	15.650	0.000
b_1	0.00892	0.00011	84.234	0.000
b_2	−0.00004	0.00000	−16.356	0.000

ANOVA					
Source	SS	df	Mean SS	*F* value	Significance
Regression	6.099	2	3.050	3.3×10^4	0
Regression	0.091	975	0.000		
			R^2 = 0.985		
SD of regression			= 0.010		

The Nonlinear Elastic Deformation of Screw Threads. The punches are held in place in the punch holders with the end plate, which is tightly screwed onto the body of the punch holders. The punch holders together with the punches are in turn attached to the plattens of the compaction simulator via screws. During compression the screws are subjected to stress and correspondingly deform. However, the load is not transferred uniformly along the engaged thread length (23). At low axial loads, the screw threads near the loaded face bear the load. With increasing force application, increasing lengths of the screws are subjected to stress, albeit not uniformly. The maximum load per unit linear length of thread which occurs near the loaded face is several times greater than the average over the entire length. The stress concentration near the loaded face and the nonuniform load distribution along the length of the screws lead to a nonlinear stress–strain relationship. Further, the stress on the screws not only are normal (axial) but also are tangential. Thus, the screws are also subjected to bending forces. This adds to the nonlinearity of the stress–strain relationship of the screws.

Nonlinear relationships between the deformation of punches and machine parts have also been reported for eccentric machines (24) and rotary multistation machines (25). Oates and Mitchell (25), however, reported an initial nonlinear region followed by a linear region. Our data and those reported in Refs. 24 and 25 demonstrate the need to examine carefully the calibration data for the particular equipment being validated. A check on whether a model being used is correct or not can be done with a statistical program with a minimum of effort.

The importance of accurately calibrating equipment for the acquisition of compression data cannot be overemphasized. Inaccurate calibration introduces equipment-source errors and provides false information on the compression and/or consolidation characteristics of the materials being investigated. This will invariably lead to inconsistencies in results obtained from different equipment, thus rendering it impossible to compare data obtained from different laboratories or sources as exemplified by the report on comparison of data obtained from different compaction simulators (16).

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