Journal of Pharmaceutical Sciences xxx (2016) 1-4

Contents lists available at ScienceDirect

Journal of Pharmaceutical Sciences

journal homepage: www.jpharmsci.org

Note

Note on the Use of Diametrical Compression to Determine Tablet Tensile Strength

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ARTICLE INFO

Article history:
Received 5 May 2016
Revised 6 July 2016
Accepted 5 August 2016

Keywords:
powder technology
compression
compaction
materials science
mathematical model
mechanical properties

ABSTRACT

The diametrical compression (DC) test, as defined in United States Pharmacopeia <1217> and in American Society for Testing and Materials testing standard D 3967, has been used extensively to derive the tensile strength (TS) of pharmaceutical tablets from the measured breaking force. DC-derived TSs provide a good approach to measuring the consistency of tablet mechanical properties from one batch to the next. For these quality control type applications, method precision is required, but accuracy is not. In addition, DC has been used to calibrate parameters of the Druker Prager Cap model, a yield criterion expressing the failure of a powder compact under arbitrary 3D loading conditions. For this application, the DC method must not only provide suitable precision but also provide accuracy. In this work, we explore the accuracy of the DC method by comparing TS results to those of the 3-point bend test method (also defined in United States Pharmacopeia <1217>). We conclude that the true TS of a powder compact is approximately double the DC-derived value. Although historical literature assumes that tablets fracture under tension along the centerline of the tablet, analysis of the stress state suggests that tablets are likely to fracture under shear. The impact of this ~50% error should be considered when accuracy of the TS result is required.

Introduction

The diametrical compression (DC) test is depicted in Figure 1 both schematically (upper left) and in practice (lower left).

Fell and Newton[1] explored the use of the DC method to determine tablet tensile strength (TS). They concluded that the tablet must break along the centerline for a valid measurement. Even in that case, the authors claim that the DC method does not provide an absolute TS.

Hertz derived the equations for stress states in elastic disks subjected to diametrical compression.[2,3] Procopio et al.[4] provided finite element method simulations showing good agreement with the Hertz equations near the center of the tablet, but contact flattening caused localized deviations near the platens.

Both Hertz[2] and Procopio et al.[4] show that the 3D stress state associated with DC varies dramatically at different locations within the tablet. This is true even for points along the centerline, as shown schematically for points a and b in Figure 1. In both locations, the tensile σ_{xx} stress represented by horizontal arrows is the same. However, the compressive σ_{yy} stress (vertical arrows) varies substantially along the y-axis. For points along the centerline of the tablet, the 3D stress state is expressed as

$$\sigma_{DC} = \begin{pmatrix} \dfrac{2\cdot F}{\pi\cdot D\cdot h} & 0 & 0 \\ 0 & \dfrac{-2\cdot F}{\pi\cdot h}\cdot\left[\dfrac{1}{\left(\dfrac{D}{2}-y\right)}+\dfrac{1}{\left(\dfrac{D}{2}+y\right)}-\dfrac{1}{D}\right] & 0 \\ 0 & 0 & 0 \end{pmatrix} \tag{1}$$

where *F*, *D*, and *h* denote the applied force, tablet diameter, and tablet thickness, respectively, and the origin of the coordinate system is taken as the center of the tablet. The equation shows that the magnitude of the compressive σ_{yy} stress is 3 times the tensile σ_{xx} stress at the origin (point a, where $y = 0$). At points closer to the compression platens (e.g., point b), the compressive σ_{yy} stress grows nonlinearly. The imbalance of normal stresses in the x and y directions produces shear stresses at inclined planes within the tablet. For example, at $y = 0.4D$, the compressive σ_{yy} stress is more than 10 times the tensile σ_{xx} stress, and the overall deviatoric or "Mises" stress component of the 3D stress tensor is more than 18 times the magnitude of the tensile σ_{xx} stress.[5] Therefore, at some point close to the compression platens, fracture becomes much more likely to initiate as the result of shear stress than from the tensile σ_{xx} stress. This concept is supported by high-speed imaging from Procopio et al.[4]

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http://dx.doi.org/10.1016/j.xphs.2016.08.004

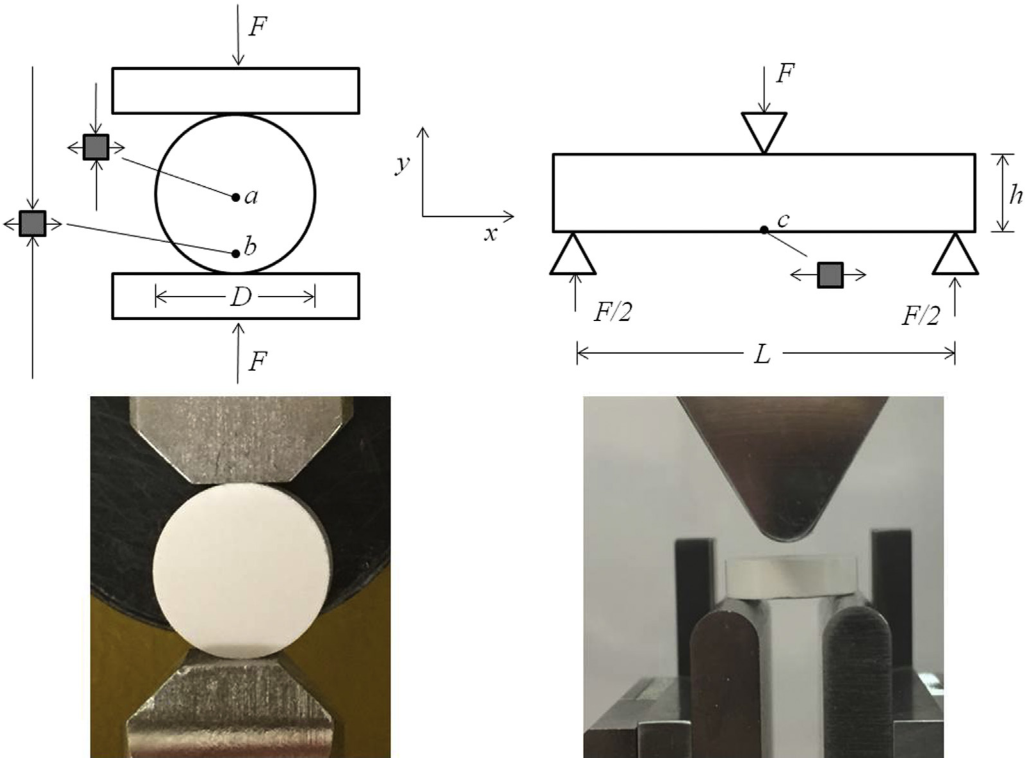

Figure 1. The diametrical compression and 3-point bend tests.

DC is often used to derive TS values used in the calibration of the Druker Prager Cap model,[6-10] which predicts that fracture can occur as a combination of tensile and shear stresses. In these calibrations, the stress state associated with fracture is assumed to be

$$\sigma = \begin{pmatrix} \frac{2\cdot F}{\pi\cdot D\cdot h} & 0 & 0 \\ 0 & \frac{-6\cdot F}{\pi\cdot D\cdot h} & 0 \\ 0 & 0 & 0 \end{pmatrix} \tag{2}$$

which strictly applies at the center of the tablet (point a in Fig. 1) and is obtained by substituting $y = 0$ into Equation 1. Unfortunately, this is the point of lowest deviatoric stress along the centerline of the tablet, and assuming that fracture initiates here is therefore questionable.

Fortunately, alternative tests have also been developed to determine the TS of cylindrical tablets. Of specific interest is the 3-point bend test[11,12] as depicted in Figure 1 both schematically (upper right) and in practice (lower right). As for the DC test, the stress state in bending also varies substantially at different points within the tablet. However, the stress state is the largest where the fracture initiates, point *c*. The 3D stress state imposed at point c is described by:

$$\sigma_{3PB} = \begin{pmatrix} \frac{3\cdot F\cdot L}{2\cdot h^2\cdot D} & 0 & 0 \\ 0 & 0 & 0 \\ 0 & 0 & 0 \end{pmatrix} \tag{3}$$

where *L* is the span defined in Figure 1. The stress state is one of simple uniaxial tension, and failure under this loading geometry therefore provides a true measure of the tablet TS.

Mazel[13] has reviewed the validity of Equation 3 using finite element method and provides combinations of *L*, *h*, and *D* for maximum accuracy. Mazel also uncovers an error in the calculations performed by David and Augsburger[11] and a typo in the equation reported [but not used (confirmed via personal correspondence; Podczeck, 2016)] by Podczeck.[12] In the latter 2 references, TS values were determined by both DC and 3-point bending.

In this work, we compare the TS values of identical materials using both DC and 3-point bending. We compare our results with the results of Podczeck and the corrected results of David and Augsburger. Results show that TS values derived from the 3-point bend test are constantly twice as high as those derived from the DC test for all materials in the study.

Materials and Methods

Tablets were created using microcrystalline cellulose (Avicel PH102, FMC Biopolymer, Philadelphia, PA) and lactose monohydrate (Fast-Flo 316, Foremost Farms, Baraboo, WI). These materials were chosen for consistency with David and Augsburger.[11] The microcrystalline cellulose was compressed as-is while the lactose was first mixed with 0.75 wt% magnesium stearate (vegetable source; Mallinckrodt, St. Louis, MO) for 5 min at 50 RPM using a Turbula® bottle mixer (Glen Mills Inc., Clifton, NJ). For both materials, tablets were compressed at 2 different solid fractions (approximately 0.77 and 0.86) using 11-mm round, flat-faced tooling. Tablet solid fractions were calculated from tablet geometry and weight as described elsewhere.[9] The breaking forces under diametric compression were measured using a Key Tablet Tester® (Key International, Cranbury, NJ) ($n = 10$ per sample). The tablets were also characterized by a 3-point bend test ($n = 10$ per sample) using a Texture Analyzer (TA-HDplus®; Texture Technologies, Hamilton, MA). The tablets were placed in a 3-point bend