NOTE

Study of the Validity of the Three-Point Bending Test for Pharmaceutical Round Tablets Using Finite Element Method Modeling

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ABSTRACT: Mechanical strength is an important quality attribute of the tablets produced in the pharmaceutical industry. The three-point bending test is one of the methods described by the United States (US) pharmacopeia to test this property. In this work, finite element method modeling was perform to study the stress distribution in a round, flat tablet submitted to this test and to verify whether the equation given by US pharmacopeia to calculate the tensile strength could be used without restrictions. For this test, the center of the lower face of the tablet was submitted to the highest tensile stress and, at this point, the stress state was nearly uniaxial. This test is thus well suited to measure the tensile strength of pharmaceutical tablets. Moreover, simulations were performed with a large range of geometrical dimensions using the dimensionless parameters D/L and h/D (where D is the tablet diameter, h is the tablet thickness, and L is the distance between the supports). In order to obtain the value of the tensile strength with a good precision when using the equation given by the US pharmacopeia, the measurements should only be performed in a restricted area of the domain defined by D/L and h/D. © 2014 Wiley Periodicals, Inc. and the American Pharmacists Association J Pharm Sci 103:1305–1308, 2014

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INTRODUCTION

Mechanical strength is an important quality attribute of the tablets produced in the pharmaceutical industry. Appropriate strength makes it possible to maintain the physical integrity of the tablet throughout the whole process of production, from compaction to packaging.

To test this property, failure tests are routinely used in the industry. The most used is the diametral compression test, also known as Brazilian test, which was introduced in the pharmaceutical industry after its development in other fields.[1,2] The United States (US) pharmacopoeia[3] also mention the possibility to study the strength of tablets by using the three-point bending test.

The three-point bending test (Fig. 1) is a classical test in material science, and is generally performed on beams. During the test, the sample is subjected to a compressive stress in the upper part resulting in a tensile stress in the lower part. The highest tensile stress is located on the lower face of the sample, and is considered to cause the failure. In the case of the bending of a round, flat tablet, the US pharmacopoeia[3] gives the following equation for the calculation of the tensile stress (MPa) on the lower face:

$$\sigma_f = \frac{3FL}{2h^2D} \tag{1}$$

where F is the applied force (N), D is the tablet diameter (mm), h is the tablet thickness (mm), and L is the distance between the supports (mm), as represented in Figure 1. The peak tensile stress is considered to be the tensile strength.

It is worth noting that two other equations can be found in the pharmaceutical literature for the same test. The first one is given by David and Augsburger[4] in the paper that introduced this test for round tablets in the pharmaceutical field. The equation they provided is Eq. 1 divided by two. The reason for this scalar treatment has no justification from a theoretical point of view as already mentioned by Stanley and Newton.[5] The other equation was given by Podczeck,[6,7] and is written as:

$$\sigma_f = \frac{8LF}{\pi D^3} \tag{2}$$

where all the terms are as defined above. In fact, Eq. 2 does not apply to the flexure of tablets but to the flexure of beams with circular section.[8]

Equation 1 is an adaptation of the equation used for the flexure of beams with rectangular section normal to the x-axis.[5] Nevertheless, it has to be borne in mind that for the case of the tablet, contrary to the case of the beam, the cross-section normal to the x-axis does not have a constant area when moving along the x-axis. As a consequence, the stress distribution in the tablet is expected to differ from the one in the beam. The direct application of Eq. 1 could thus be an approximation. In all cases, even for beams, the assumptions made for the determinations of Eq. 1 lead to geometrical restrictions concerning the beam dimensions and the distance between the supports.[9] In the US pharmacopeia, no restrictions are given regarding these parameters. In this study, numerical modeling using the finite element method (FEM) was utilized to study the stress distribution inside the tablet and to check the validity of Eq. 1 when varying the different geometrical parameters.

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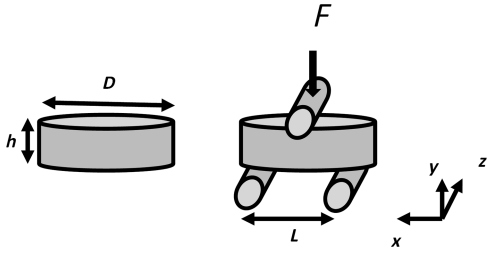

Figure 1. Schematic representation of the three-point bending test.

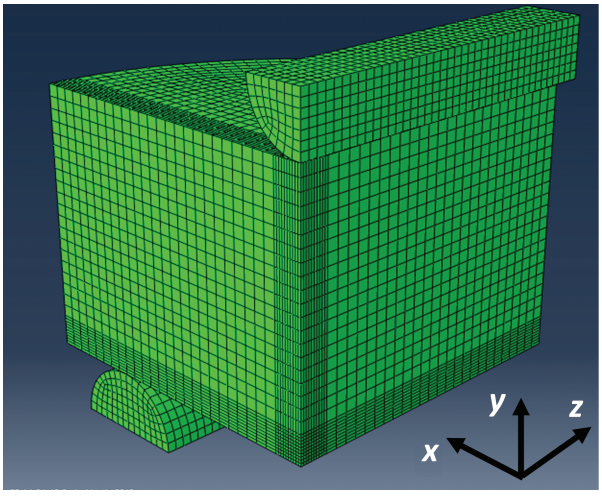

Figure 2. Numerical modeling setup for the test (in this example $D/L = 1.4$ and $h/D = 0.4$).

MATERIAL AND METHOD

Numerical Analysis Setup

The FEM modeling was performed using Abaqus® Standard 6.10 software (Dassault Systèmes, Vélizy-Villacoublay, France). The setup of the numerical modeling can be seen in Figure 2. Using the symmetry of the system, only a quarter of the compact was modeled. The whole system can be reconstructed by using the planes of symmetry (y,z) and (x,y).

The compact was considered as an elastic material with a Young's modulus (E) of 3.4 GPa and a Poisson's ratio (ν) of 0.23. These values were based on those found, in a former study,[10] for a compact of anhydrous calcium phosphate with a porosity level of 38%. The supports were also taken as elastic with elastic constants equal to those of steel ($E = 200$ GPa and $\nu = 0.3$). The stress was applied by moving down the upper support along the y-axis at the constant velocity of 0.05 mm/s.

As it can be seen in Figure 2, the mesh was refined at the center of the lower face of the compact to increase the accuracy.

Choice of the Geometrical Parameters

D/L and h/D were chosen as dimensionless parameters to study the influence of the geometric parameters on the validity of the test. Because L is always lower than D, D/L was varied from 1.1 to 2. The other parameter h/D was varied from 0.1 to 0.5 to cover a range of sizes compatible with a pharmaceutical tablet. Twenty five simulations were performed to cover the whole geometrical range.

RESULTS AND DISCUSSION

Stress Distribution in the Compact

The first step of this work was to study the stress distribution inside the tablet during three-point bending. The case of $D/L = 1.1$ and $h/D = 0.3$ was chosen as an illustrative example. The distribution of the normal stress along the x-axis in the compact can be seen in Figure 3. As expected, the compact is submitted to compression stresses on the upper surface, and to tensile stresses on the lower surface (tensile stresses are taken as positive). The maximal tensile stress is located on the lower face of the compact. The main difference with the distribution that would have been obtained on a beam with a square section normal to the x-axis is the distribution obtained when moving along the z-axis. For the beam, the stress is invariant with the translation along z-axis.[5] On the contrary, for the cylindrical tablet, the tensile stress on the lower face decreased when moving from the center of the face toward the external part. As a consequence, the maximal tensile stress was located at the center of the lower face of the compact. Thus, the value of the stress at this point was considered below to test the validity of Eq. 1.

Another interesting point was to check the value of the other stresses (normal and shear) at the node of highest tensile stresses. During the whole simulation, all the other stresses at the center of the lower face were always less than 1.5% of the value of the tensile stress along the x-axis. As an example, for a value of tensile stress along the x-axis of 3.5 MPa, which is in the order of magnitude of the value obtained experimentally for pharmaceutical compacts,[11] the complete stress tensor at the central point was:

$$\begin{pmatrix} 3.5 & 0.01 & -0.001 \\ 0.01 & -0.01 & 6\times10^{-5} \\ -0.001 & 6\times10^{-5} & -0.04 \end{pmatrix}$$

As a consequence, the stress state at the center of the lower face of the compact is nearly uniaxial. The value obtained for the stress at the failure can thus be considered as a value of the tensile strength of the material.

In the pharmaceutical literature, such as for example in the US pharmacopeia,[3] the value of the failure stress obtained using the diametral compression test is generally taken as the tensile strength of the material. Nevertheless, in the case of the diametral compression, it was shown that the compressive stress at the center is three times higher than the tensile stress, which is supposed to cause the failure.[8,12] The three-point bending test is thus much closer to a real tensile test than the diametral compression test. Moreover, several articles showed that the diametral compression gave values two to three times lower than the flexure test.[11,13] The results shown in the present study thus indicate that, in order to obtain the tensile strength of the compact, the three-point bending test should be preferred to the diametral compression test.

Validity of the Test

The second part of the work intended to check whether Eq. 1 made it possible to calculate the actual value of the maximal tensile stress in the tablet. For this purpose, various simulations were performed by varying D/L and h/D as described above. For each simulation, the evolution of the maximal

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