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The bending strength of tablets with a breaking line—Comparison of the results of an elastic and a "brittle cracking" finite element model with experimental findings.

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ABSTRACT

The aim of this work was to ascertain the influence of the position of the breaking line of bevel-edged tablets in a three-point bending test. Two different brands of commercially available, flat-round, bevel-edged tablets with a single central breaking line were studied. Breaking line positions tested, relative to the upper loading roll, were 0°, 22.5°, 45°, 67.5° and 90°. The breaking line faced either up- or downwards during the test. The practical results were compared with FEM results simulating similar test configurations.

Tablets failed mainly across the failure plane, resulting in two tablet halves. An exception to this was found for tablets where the breaking line faced down and was positioned at an angle of 22.5° relative to the loading plane. Here the crack followed the breaking line in the centre of the tablets and only diverged towards the loading plane position at the edges of the tablets. The breaking line facing upwards resulted in a significantly higher tensile strength of the tablets compared to it facing downwards. However, with one exception, the orientation of the breaking line relative to the loading plane appeared not to affect the tensile strength values.

A fully elastic FEM model indicated that both the position of the breaking line relative to the loading plane and as to whether the breaking line faced up- or downwards during the bending test would result in considerably different failure loads during practical experiments. The results also suggested that regardless of the breaking line position, when it is facing down crack propagation should start at the outer edges propagating towards the midpoint of the discs until failure occurs. Failure should hence always result in equal tablet halves, whereby the failure plane should coincide with the loading plane. Neither predictions fully reflected the practical behaviour of the tablets.

Using a brittle cracking FEM model significantly larger tensile stresses for tablets with the breaking line positioned downwards at 0° or 22.5° relative to the loading plane were still predicted, but the differences between model and experimental values was greatly reduced. The remaining differences are more likely due to the inadequacy of the equation available to calculate the experimental tensile strength values. This equation cannot account for the presence of a breaking line and overestimates the thickness of the loading plane by the depth of the breaking line when in 0° or 22.5° position. If the depth of the breaking line is taken into account, the model predictions and the experimental findings are comparable. Also, in the brittle cracking FEM simulations the predicted crack propagation patterns were similar to those found in the experiments, and the model stress distributions across the lower surfaces were much more homogeneous and streamlined parallel to the loading plane. The brittle cracking model hence reflected the practicalities of the bending test more closely. The findings suggested that with the breaking line facing down fracture should always start in the centre of a tablet at its lower surface, initiated by the breaking line. Due to simultaneous development of larger stresses along the *y*-axis the tablet should still break into two equal halves along the loading plane, unless the position of the breaking line relative to the loading plane was 22.5°. In this case the tablet would fail by a mixed process, whereby failure would occur mainly along the breaking line, but due to simultaneous crack formation at the lower surface close to the bevel edge parallel to the loading plane the final breaking pattern would deviate from the breaking line about half-way from its centre, as seen in the practical experiments.

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1. Introduction

In an attempt to reduce chipping of the tablet edges during packaging, transport and handling, flat round tablets typically are bevel-edged. Frequently they also have a breaking line (“score line”), the purpose of which has been discussed by Van Santen et al. (2002). Under industrial manufacturing conditions, the breaking force of round tablets should be tested using the standard diametral compression test (Method 1217, USP38/NF33, 2014). Recently, Podczeck et al. (2014) investigated the influence of the position of the breaking line in terms of its angle relative to the loading plane during a diametral compression test of commercially available bevel-edged flat-round tablets. They compared their practical findings with theoretical investigations using finite element method (FEM). FEM results using both a fully elastic and an elasto-plastic model predicted that the tensile stress values at failure would be up to three times larger, if the breaking line was positioned at an angle of less than 45° relative to the loading plane, whereas at an angle of 45° or larger the failure loads should be similar. Newton et al. (1977) using photoelasticity measurements reported that the effect of the breaking line position depended on its depth, and if the depth was in the range of commercial tablet designs, a horizontal breaking line position caused compressive stresses at the tip of the breaking line and was associated with an increase in tensile stresses at the plane face. On the other hand, the vertical position of the breaking line resulted in increased tensile stresses at the tip of the breaking line and a reduction in the tensile stresses at the flat face. Similar effects, but more detailed due to the use of different breaking line positions in terms of angles relative to the loading plane, were found using FEM (Podczeck et al., 2014), and their theoretic work also predicted that not all breaking line angles would result in clean tensile failure. The practical results, however, only confirmed some deviations from a clean tensile failure due to the position of the breaking line, whereas the breaking forces as such were only marginally affected by the position of the breaking line relative to the loading plane. Since there were differences in the breaking pattern, they concluded that despite similar failure loads the failure mechanism varied with the angle of the breaking line and hence a conversion of a breaking load into the tensile strength using the Brazilian equation (Barcellos, 1953; Carneiro, 1953; Fell and Newton, 1968, 1970) was not recommended.

Mazel et al. (2014) suggested that pharmaceutical compacts of round, cylindrical shape should be tested using a three-point bending test, because it reflects the tensile failure stress more accurately than the diametral compression test. The advantages and disadvantages of this test in terms of its practical applicability under routine settings in the pharmaceutical industry can be found in previous reports (Podczeck, 2012; Podczeck et al., 2014). However, this test has not yet been studied in terms of its applicability and accuracy when breaking round-flat, bevel-edged tablets with a breaking line.

The aim of this work was to ascertain the influence of the position of the breaking line in a three-point bending test. Similarly to the previous paper (Podczeck et al., 2014) two different brands of commercially available, flat-round, bevel-edged tablets with a single central breaking line were studied. Breaking line positions tested, relative to the upper loading roll, were 0°, 22.5°, 45°, 67.5° and 90°. The breaking line was either facing down or upwards during the test. The down-facing position would be theoretically preferred due to tensile stresses developing only at the lower tablet face during a bending test and crack propagation leading to tablet failure should hence always start at the lower tablet surface. In this situation, fracture mechanics predicts a stress concentration at the tip of the breaking line (Irwin, 1957), and thus potentially an influence of the breaking line position on the failure stress. However, assuming that under industrial working conditions an automatic tablet positioning mechanism would be required to make such a test viable, an upwards orientation of the breaking line appears possible and hence should also be investigated. The practical results were then compared with FEM results simulating similar test configurations. Initially an elastic model was employed, followed by a brittle cracking model in an attempt to overcome discrepancies between the theoretical FEM results and the experimental findings.

2. Materials and methods

2.1. Software

Standard finite element methodology (FEM) was employed (Abaqus 6.12.3, Dassault Systèmes, Vélizy–Villacoublay, France). Cubic-spline interpolations were made using a Microsoft®-approved add-on to Excel 2013 (SRS1 Software, Boston, MA). Analysis of Variance (ANOVA) was performed using SPSS 20.0 (SPSS–IBM, Woking, UK).

2.2. Practical work

Bevel-edged tablets with a single central breaking line were purchased to be able to reflect the larger variability of tablet breaking loads of commercially produced compacts during testing: (1) Superdrug Diarrhoea Relief Tablets (DRT), Surepharm Services Ltd., Burton–Upon–Trent, UK, batch 4A222; (2) Boots Aspirin 300 mg Dispersible Tablets (ADT), Aspar Pharmaceuticals Ltd., London, UK, batch 140700.

According to the Patient Information Leaflet (PIL) the main ingredients of the DRT tablets are 400 mg light kaolin and 75 mg calcium carbonate. The remaining excipients are icing sugar, maize starch, magnesium stearate, erythrosine, clove-, cinnamon- and nutmeg oil. The estimated powder particle density of the mixture is 2150 kg m^{-3}. The ADT tablets contain 300 mg of acetylsalicylic acid, plus lactose monohydrate, sodium saccharin, maize starch, citric acid, sodium lauryl sulphate, talc and calcium carbonate as excipients (based on updated “Summary of Product Characteristics”, dated 27 April 2015). The estimated powder particle density of the mixture is 1470 kg m^{-3}.

The breaking load of the tablets was determined using a CT6 tablet strength tester (Engineering Systems, Nottingham, UK), equipped with a 50 kg load cell, at a test speed of 1 mm min^{-1}. A three-point bending rig was used, which had freely rotating lower rolls and a fixed upper roll, each of 3 mm diameter. The distance between the midpoints of the lower rolls was 10.5 mm. The breaking load was recorded with an accuracy of ±0.005 kg. The tester was linked to a laptop (Dell Latitude D505, Dell UK, Bracknell, Berkshire) via a USB cable. Machine inherent plotter software (Graph Plotter®, V2.09; Engineering Systems, Nottingham, UK) was installed and used to control the tester remotely from the computer. Force versus displacement curves were recorded for each tablet using a recording frequency of 1000 Hz. They were exported into Windows Excel 2007 (Microsoft®) and further processed to obtain the slope of the linear portion of the force–displacement curves. The tensile failure stress of the tablets was calculated from (Hertzberg, 1996):

$$\sigma_t = \frac{3PL}{2DW^2} \quad (1)$$

where P is the breaking load, L is the distance between the midpoints of the lower rolls, and D and W are the diameter and thickness of the tablet, respectively.