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Reevaluation of the diametral compression test for tablets using the flattened disc geometry

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ABSTRACT

Mechanical strength is an important critical quality attribute for tablets. It is classically measured, in the pharmaceutical field, using the diametral compression test. Nevertheless, due to small contact area between the tablet and the platens, some authors suggested that during the test, the failure could occur in tension away from the center which would invalidate the test and the calculation of the tensile strength. In this study, the flattened disc geometry was used as an alternative to avoid contact problems. The diametral compression on both flattened and standard geometries was first studied using finite element method (FEM) simulation. It was found that, for the flattened geometry, both maximum tensile strain and stress were located at the center of the tablet, which was not the case for the standard geometry. Experimental observations using digital image correlation (DIC) confirmed the numerical results. The experimental tensile strength obtained using both geometries were compared and it was found that the standard geometry always gave lower tensile strength than the flattened geometry. Finally, high-speed video capture of the test made it possible to detect that for the standard geometry the crack initiation was always away from the center of the tablet.

1. Introduction

Mechanical strength is an important critical quality attribute for tablets. As such, especially in the development of a Quality by Design approach, a precise and reliable quantification is required. For pharmaceutical tablets, the main technique to study the mechanical strength of the tablet is the diametral compression test (also known as Brazilian test)(EUP, 2016; USP, 2013). This test was developed during the 40's to study the mechanical strength of concrete cylinders (Carneiro, 1943; Fairbairn and Ulm, 2002). It measures the tensile strength in an indirect manner when direct tensile tests are difficult to perform due to the mechanical properties of the tested material. Firstly used for concrete or rocks, it was introduced, during the 60's for the characterization of pharmaceutical tablets (Fell and Newton, 1968, 1970). The cylindrical shape is indeed easy to obtain when performing die compression and the test is thus well suited for tablets.

Nevertheless, as mentioned before, the diametral compression test is an indirect test. A cylindrical sample is submitted to a compressive force along its diameter by diametral compression between two flat platens. This promotes the development of tensile stresses at the center of the compact. These tensile stresses are supposed to cause the failure. Then, by supposing an elastic behavior of the compact and by using the elastic theory, in a 2D plane stress, it is possible to prove that the maximum tensile stress is located at the center of the compact and is given by:

$$\sigma = \frac{2F}{\pi D h} \quad (1)$$

where σ is the maximum tensile stress, F is the applied force and, D and h are respectively the diameter and thickness of the cylinder. Thus, considering a failure criteria based on the maximum principal stress, the tensile strength is calculated by substituting, in Eq. (1), the force that caused the failure.

The main problem of this test geometry is the contact in the loading area. The contact area between the platens and the cylinder is very small, and it promotes the development of high stresses. If the contact is too small, the failure could be caused by shear effects in this area, leading thus to incorrect failure pattern.

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To avoid this problem, several authors have recommended the use of loading strips that are positioned between the platens and the sample (Li and Wong, 2013).This makes it possible to increase the contact area avoiding shear or compressive failure. Other techniques to increase the contact area have been proposed like the use of curved platens (ISRM, 1978) or the use of flattened discs instead of cylinder (Wang et al., 2004). This last solution, will be further developed below in the article.

In the pharmaceutical field, the problem of contact was studied since the test was introduced. In the article which is the reference for the use of the diametral compression for tablets, Fell and Newton (1970) studied the influence of inserting sheets of blotting paper between the platens and the tablet. Diametral compression tests with and without blotting papers were carried out for five different lactose batches (one crystalline, and four spray-dried noted A–D). For the samples B–D they found that, without blotting paper, the failure was due to compression and shear at the contact with platens. For this case, the use of blotting paper was mandatory to obtain a tensile failure. For the case of crystalline and spray dried A samples, the results were different. For these two products, with and without blotting paper, the failure occurred in tension, i.e. no failure at the contact point was observed. Nevertheless, the values obtained in the two cases were different. They then stated "this raises the question as to which of the values represent the tensile strength of the tablet". The contact problem was also studied more recently by Davies et al. (2007). In their study, they used two contact configurations. The first one was classical flat platens and for the second configuration, they attached semi-circular steel rods to the platens. As it is proved in the article, this last configuration made it possible to have a smaller contact width. In all the cases, they observed failure in tension, but they also observed that for smaller a contact width, they obtained a lower tensile strength by using Eq. (1) which is exactly the same trend obtained by Fell and Newton (1970). Again, due to the failure pattern observed, shear or compression failure at the contact point could not be taken as responsible of the difference in tensile strength obtained for different contact widths. And the question mentioned above about which one of the value is the correct value of the tensile strength has still, to our knowledge, no real answer in the literature.

Nevertheless, the problem of contact is generally discarded in the pharmaceutical field. A great number of studies have been published about the failure of pharmaceutical compacts using the diametral compression test, studying for example the effects of the shape of the compact (Pitt et al., 1988; Podczeck et al., 2013, 2014; Razavi et al., 2015; Shang et al., 2013a, 2013b) or on the effect of the material properties (Procopio et al., 2003). But in nearly all the studies, flat platens are used. The regulatory texts also advise for the use of flat platens (EUP, 2016; USP, 2013) and the value of the diametral compression test is taken as the value of the tensile strength and used, for example, as a material characteristic to calibrate numerical models (Brewin, 2008; Cunningham et al., 2004; Wu et al., 2005).

Another interesting point is that the theoretical development that leads to Eq. (1) is a 2D one. It thus supposes that there is no 3D effects. Nevertheless, 3D effects were demonstrated by both analytical developments (Wijk, 1978) and numerical simulation using the Finite Element Method (FEM) (Ehrnford, 1981; Li and Wong, 2013). All these studies show that, if the contact surface between the cylinder and the platen is too small, high tensile stresses develop on the outer surface of the compact away from the central axis. The value and position of these stresses depend on the contact area, on the thickness of the compact and also on Poisson's ratio. The quantitative determination of these stresses is thus complicated, but as stated by Ehrnford a long time ago, due to these stresses, "peripherical fracture initiation must be regarded as a possibility" (Ehrnford, 1981). Surprisingly, the presence of the maximum tensile stress away from the center is generally not considered and studies about 3D effects in the diametral compression test mainly focus on the variation of the tensile stress at the center along with the thickness of the compact (Podczeck et al., 2013; Yu et al., 2006).

After this brief review, it is clear that the diametral compression test is still not fully understood and that the results obtained (i.e. tensile strength) must be taken with caution if they are to be used as material constants. It is also clear that the problem comes from the contact area between the platen and the compact. Among the solutions to increase this contact, the flattened Brazilian disc was proposed for rocks. It consists in introducing two flat ends to the disc (Wang et al., 2004). This technique has the advantage of introducing a well-defined and quantified contact area between the platens and the tablet. By choosing the correct contact surface, it should thus be possible to suppress the problem of shear failure and also the peripheral tensile stresses due to 3D effects. In the case of pharmaceutical tablets, by designing tools with the proper shape, it is easy to produce flattened tablets.

In this work, we wanted to study, in the case of pharmaceutical tablets, the effect of using the flattened Brazilian disc on the value of the tensile strength obtained by diametral compression. In a first part, FEM studies were performed to show that the use of specially designed punches to obtain flattened compact did not have an impact on the stress and density distributions inside the compact. This part was necessary to prove that the flattened and round tablet had the same density distribution and that their failure behavior could thus be compared. In a second part, the stress and strain distributions inside the compact during the diametral compression were evaluated numerically (FEM). FEM simulations were compared to the results already published in the literature. Then, Digital Image Correlation (DIC) was used to compare the experimental strain fields to the ones obtained numerically in order to prove that FEM simulations gave a good representation of the actual behavior of the compact. Afterwards, the diametral test was performed for both normal and flattened disc geometry to compare the values obtained for the tensile strength. Finally, a high speed camera was used to locate the crack initiation during the test on both geometries.

2. Material and method

2.1. Powders

Five different powders were used to produce compacts: anhydrous calcium phosphate (aCP) (Anhydrous Emcompress®, JRS Pharma, Rosenberg, Germany), calcium phosphate dihydrate (DCP) (Emcompress Premium®, JRS Pharma, Rosenberg, Germany), spray-dried lactose monohydrate (SDLac) (Flowlac® 90, Meggle, Wasserburg, Germany), granulated lactose monohydrate (GLac) (Tablettose® 80, Meggle, Wasserburg, Germany) and spray-dried mannitol (SDMan) (Pearlitol® 200SD, Roquette, Lestrem, France). To perform the compaction experiments, the products were mixed with 1% (w/w) of magnesium stearate (Cooper, Melun, France) to minimize the frictions in the die. The blending was performed at 50 rpm for 5 min using a turbula mixer (Type T2C, Willy A Bachofen, Muttenz, Switzerland).

2.2. Compression

All the compacts were produced using a compaction simulator Stylcam® (Medelpharm, Bourg-en-Bresse, France). This tableting press is a single station press. It is equipped with force sensor (accuracy 10 N) and the displacements of the punches are monitored with an accuracy of 0.01 mm. Two different sets of flat-faced euro B punches were used (ACM, Avilly-Saint-Leonard,