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Research paper

The determination of the mechanical properties of elongated tablets of varying cross section

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Abstract

The mechanical properties of elongated tablets of different thickness prepared at a range of pressures with surfaces that are flat or curved, have been determined by application of a flexure test. The strength of the tablets in terms of breaking load increases with an increase in the cross-sectional area of the tablets. To provide an improved method of comparing the strength of the tablets, the tensile strength of the specimens has been calculated from equations based on stress analysis. While an allowance for a change in thickness was provided by such a system there was a clear indication that the specimens tensile strength was still dependent on their dimensions. For equivalent central core thickness strength increased with the face curvature. This could be due to changes of the structure within the specimen during the formation process and there were clear indications that tablet porosities for equivalent compaction pressures were not equal. The differences could, however, be utilised to provide optimum conditions of curvature and central core thickness to give maximum tablet strength.

Keywords: Mechanical properties; Elongated tablets; Flexure test

1. Introduction

The mechanical strength of tablets is used as a quality control procedure to ensure that the tablets prepared are reproducible and can withstand the subsequent handling procedures. In addition the mechanical properties of tablets are also measured to evaluate some functional properties of the material in terms of their ability to form tablets. The method of determining the mechanical properties is usually undertaken by the application of a mechanical stress until the tablet breaks and recording this value, or by subjecting the tablets to small repeated tumbling interaction and monitoring the loss in weight due to abrasion (friability test). The value at which a tablet breaks is dependent on the type of stress applied, its manner of application and the shape and dimension of the tablet. For tablets that are circular, it is more usual to subject them to diametral loading. This has the added advantage that under appropriate conditions of stress, the breaking load can be converted into tensile strength if the tablet is in the form of a right circular cylinder [1], or has a regular convex surface [2], from consideration of the physical dimensions of the tablet. This allows a more readily comparable value if the tablets have grossly different dimensions [3] although even here, tablets with different dimensions do not always have equivalent tensile strength, even when corrected for volume and brittleness [4], presumably because changing the dimensions of the specimen changes the flaw distribution within the compact.

When tablets are produced in elongated beam form, diametrical compression tests are no longer appropriate. The obvious way to overcome this is by subjecting the tablets to a flexure test and Stanley and Newton [5] have shown how such a system can be used to derive a tensile strength of both beams with a flat and a curved surface. The former takes the form for a flat faced beam of

$$\sigma_f = \frac{3Fl}{2bd^2}$$

where σ_f is the tensile strength, F is the load to cause failure in a three point flexure test, b and d are the width and the thickness of the beam, respectively, and l is the distance between the lower supports. For a beam with curved faces, the equation takes the form

$$\sigma_f = \frac{3Fl}{2d^2}\left(\frac{d + 2a}{6A + bd}\right)$$

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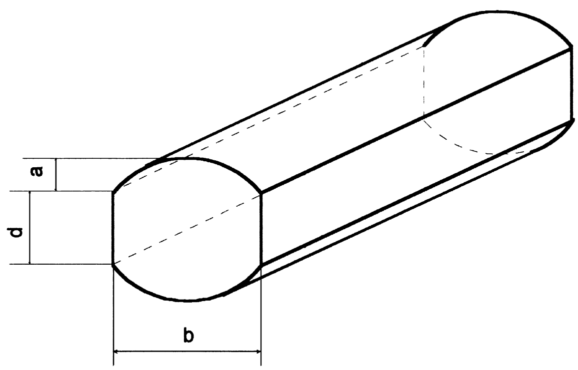

Fig. 1. Definition of dimensions of an elongated tablet. (a) Height of the curved segment (a = 0 for flat faced tablet); b, tablet width; d, tablet thickness.

where d is the central thickness of the beam, a is the height of the curved surface above the central thickness and A is the area of the curved segment (see Fig. 1).

While Stanley and Newton [5] showed how such expressions could be used to determine the tensile strength of such tablets the same question arises as with the diametral crushing strength. Do the different dimensions of tablets impose restrictions on the use of this approach? To test whether or not this is the case, flat-faced beam specimens of different thickness and beams with curved surfaces of different radii and with differing central thickness were prepared and their flexure strength determined.

2. Materials and methods

2.1. Materials

The model test materials were the directly compressible dicalcium phosphate dihydrate (DCPD) (Emcompress, Forum Chemicals, Woking, UK), microcrystalline cellulose (Avicel PH102, FMC, Philadelphia, USA) and acetylsalicylic acid (crystalline grade, Rhône–Poulenc Rorer Ltd., Dagenham, UK). The powders were stored for at least 2 weeks at ambient room conditions prior to compaction.

2.2. Methods

Tablets were prepared with punches and dies made to fit a single-punch tablet machine and manufactured by Hollands (Long Eaton, UK). The punches were 25 mm in length and 10 mm in width and were either flat-faced or curved to provide tablets with the cross section shown in Fig. 1, and values of $a = 0.1$ and 0.2 cm. The compacts were prepared by compression of a known weight of powder between the faces of the punches which were mounted between the platens of a mechanical testing instrument (Instron floor model, Instron, High Wycombe, UK). Compaction at a cross-head speed of 10 mm/min was used. At least 10 tablets were prepared for testing at each applied pressure. The quantity of dicalcium phosphate dihydrate which was required to prepare tablets with punches which had a curved height segment of 0.2 cm and a thickness to width ratio of 0.6 exceeded the capacity of the die and hence could not be prepared.

Tablets were stored at 53% relative humidity in desiccators filled with saturated salt solutions of magnesium nitrate (BDH, Poole, UK) for 7 days prior to testing. Before fracturing, the dimensions and weight of the tablets were determined to 0.01 mm and 0.001 g, respectively. The porosity of the tablets was determined by calculation of the volume from their dimensions and weight (Mettler AE160 analytical balance, Greifensee, Zürich, Switzerland) and their

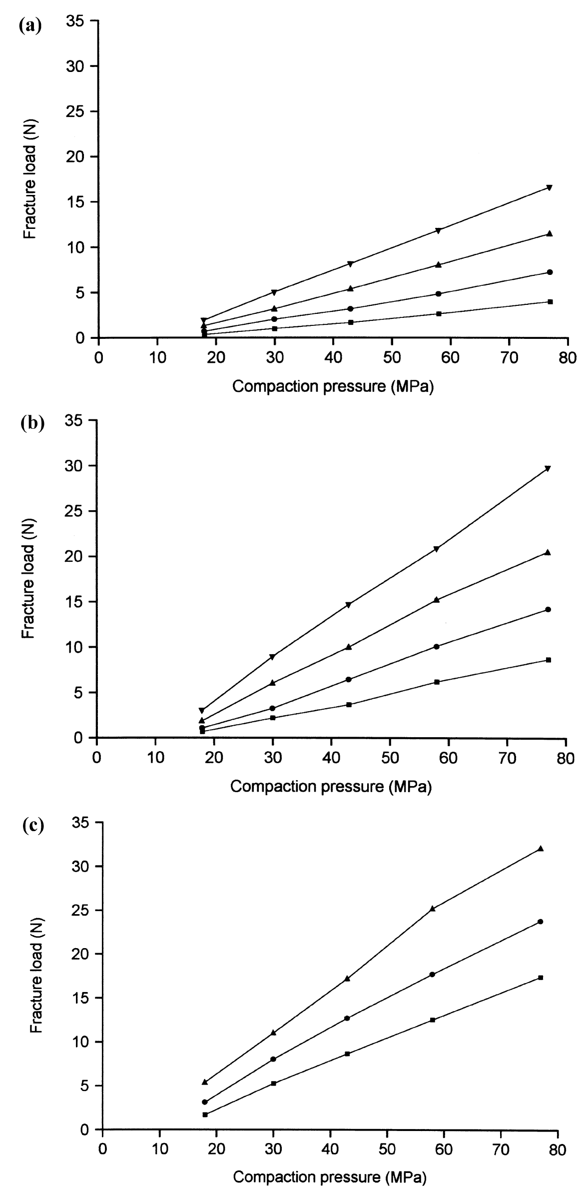

Fig. 2. Breaking load of elongated emcompress tablets with different thickness to width ratio 'd/b' (■, d/b = 0.3; ●, d/b = 0.4; ▲, d/b = 0.5; ▼, d/b = 0.6) and different height of the curved segment 'a' ((a) 0.0 cm; (b) 0.1 cm; (c) 0.2 cm) as a function of the compaction pressure.

apparent particle density was determined by an air compression pycnometer (Model 930, Beckman Instruments, Inc., CA). The flexure strength of the tablets was determined (10 replicates) with a tablet strength tester (CT-40 Engineering systems, Nottingham, UK) fitted with a 3-point bending device. The load was applied at a rate of 1 mm/min and the load at failure was recorded by the peak hold device fitted to the CT-40. The specimens were examined for the mode of failure and only those which broke from a central point of the lower surface were used to derive tensile strength values. The coefficient of variation of the breaking load for the samples ranged from 3–25%, and was independent of tablet dimensions, materials or compaction pressure.

3. Results and discussion

Subjecting all the tablet specimens to the flexure test resulted in failure propagating from the centre point below the point of load, indicating that they had indeed failed in tension. Expressing the breaking load as a function of compaction pressure clearly shows (Fig. 2a,b,c) that for a given beam thickness, the breaking load increases in a linear manner with the compaction pressure. As the beam thickness increases, so does the breaking load as there is a greater resistance to bending induced by the greater cross-sectional area. For a given beam thickness, the breaking load also increases as the radius of curvature increases, providing an increase in the height of the curved segment, and an increase in the cross sectional area of the specimen.

Conversion of the breaking load into tensile strength for the flat-faced beams shows that the values are nearly independent of the thickness of the beam (Fig. 3a). Hence expressing results of tablet strength as a tensile strength as a function of pressure would clearly provide a better comparison of the mechanical properties of such tablets, especially as the rate of increase in tensile strength with pressure is constant, at 0.018 for the depth to breadth ratios (d/b) of 0.3, 0.4 and 0.5. The gradient is slightly higher (0.023) for the thickest beams.

These results indicate that for a plane faced compact, comparison of tablet strength as a derived tensile strength value gives a satisfactory comparison of mechanical properties. The question arises does this apply for tablets with different face curvatures? For curved faced specimens, the change in central core thickness shows slight variation in the value of the tensile strength, but far less than exists with the breaking load (Fig. 3b). The rate of change of strength with pressure for the curved-faced specimens is 0.024, 0.024, 0.025 and 0.025 for beams with d/b ratios of 0.3, 0.4, 0.5 and 0.6, for those whose curved segment the height is 0.1 cm, (Fig. 3b) values of 0.024, 0.024, and 0.23 for beams with d/b ratios of 0.3, 0.4 and 0.5 and a curved segment height of 0.2 cm (Fig. 3c).

If the results are replotted to illustrate the influence of face curvature on the derived value of the tensile strength

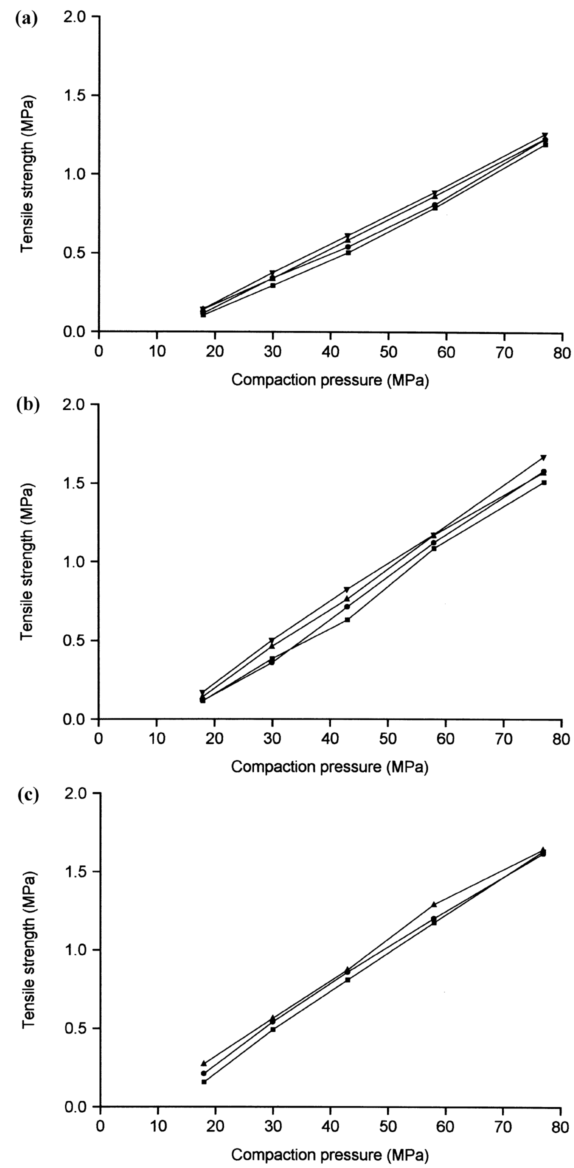

Fig. 3. Tensile strength of elongated emcompress tablets with different thickness to width ratio ‘d/b’ (■, d/b = 0.3; ●, d/b = 0.4; ▲, d/b = 0.5; ▼, d/b = 0.6) and different height of the curved segment ‘a’ ((a) 0.0 cm; (b) 0.1 cm; (c) 0.2 cm) as a function of the compaction pressure.

(Fig. 4a–d) it can be seen that in all cases the value of the tensile strength is higher as the face curvature increases for all central core thickness to width (d/b) ratios. Thus it is not possible to relate accurately the value for the tensile strength of the specimens if the face curvature changes. There are several sources for this difference. One is associated with the fact that when the face curvature of the punch is changed, the internal structure of the tablet may change. The changes in internal structure of the tablet are very difficult to evaluate. Gamma-ray attenuation has been used to identify density changes within compacted powders [6], but has not been applied to tablets with the particular shape reported here. It is certainly likely that the specimens here show similar non-homogeneous behaviour to the round tablets

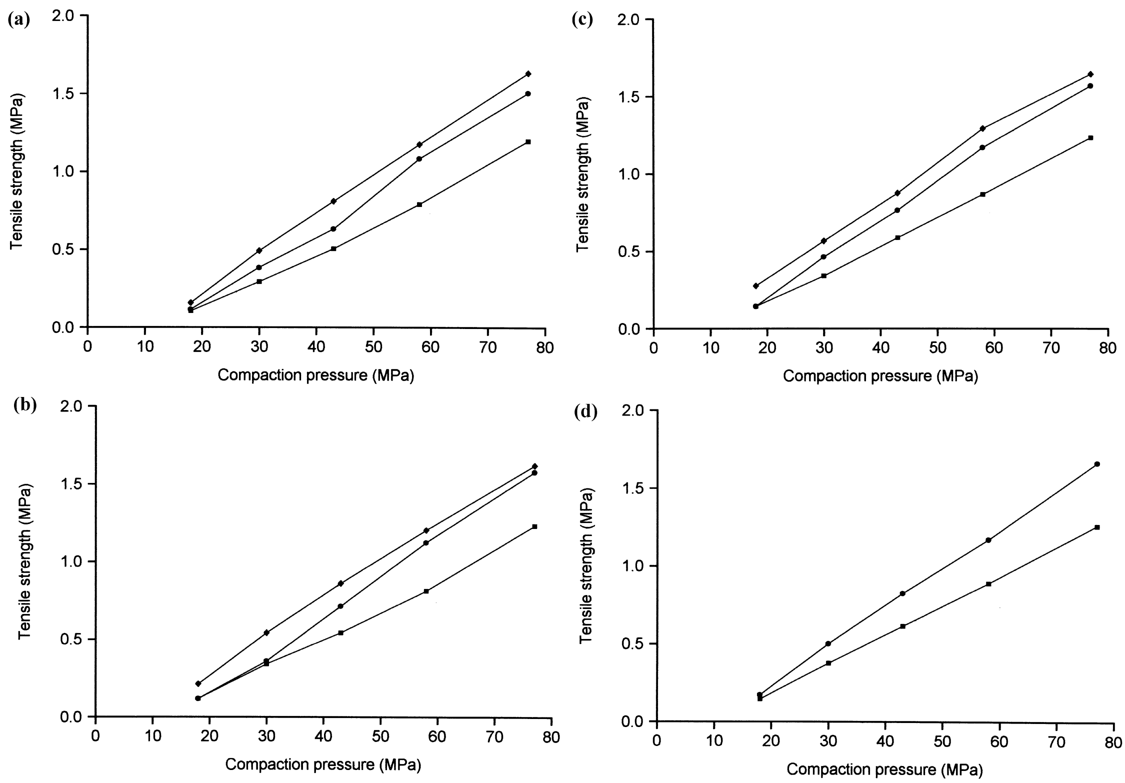

Fig. 4. Tensile strength of elongated emcompress tablets with different height of the curved segment 'a' (■, a = 0.0 cm; ●, a = 0.1 cm; ◆, a = 0.2 cm) with different thickness to width ratio 'd/b' ((a) 0.3; (b) 0.4; (c) 0.5; (d) 0.6) as a function of the compaction pressure.

studied by Charlton and Newton [6]. It is possible to estimate if there is an overall change of the tablet structure by the calculation of the porosity of the tablet. This was possible from measurements of tablet dimensions, weight and the apparent particle density of the dicalcium phosphate dihydrate. The value of tablet porosity as a function of the natural logarithm of the applied pressure is shown in Fig. 5a–d. Especially in Fig. 5a–c it appears that a lower porosity was obtained for the same compaction pressure when the curved punches were used to produce the tablets. Thus the higher tensile strength of the tablets could be due to the lower porosity of such specimens. For tablets with the highest central core thickness plain-faced and curved-face punches produce tablets of approximately equal porosity for application of equivalent pressure (Fig. 5d). This does not mean that the internal structure is in fact the same, as porosity is an overall average value and provides no information on the nature of the size and the distribution of the spaces which contribute to the porosity. Nor does it mean that tablets of the same porosity will have the same strength. Just how porosity is involved in the strength is complex but it is more likely that the shape and size of pores is more important in terms of strength than the average value of porosity [7]. It has been previously observed that crack propagation starts from a pore and then follows grain boundaries [8]. The tensile strength of tablets which are of equal porosity is compared, e.g. Fig. 6a,b,c and clearly tablets of equal porosity do not always have the same strengths. The flexure test should be particularly associated with the flaws at the lower surface of the specimen because the maximum tensile stress occurs at the lower surface of the specimen, and the orientation of the flaw that propagates would be perpendicular to the surface. The internal structure of the tablet will, however, contribute to the total structure of the tablet which is certainly associated with the resistance to the bending of the specimen.

To illustrate that the findings were not only applicable to dicalcium phosphate dihydrate, which is a material which is brittle in nature, tablets were also prepared at different compaction pressure from microcrystalline cellulose (Avicel PH102) and acetylsalicylic acid (crystalline grade). The latter materials are considered as ductile [9]. The results in Figs. 7 and 8 again illustrate for the same central core thickness, that tablets prepared with punches with a curved surface have a higher tensile strength than those with a flat surface. Again, this was not related to the porosity of the tablets. Tablets of the same porosity but with different face curvature did not have equivalent strength values. The similarity of the results produced for DCPD and the two ductile powders confirms that tablet strength purely reflects the properties of the compacted specimen, which is brittle in nature.

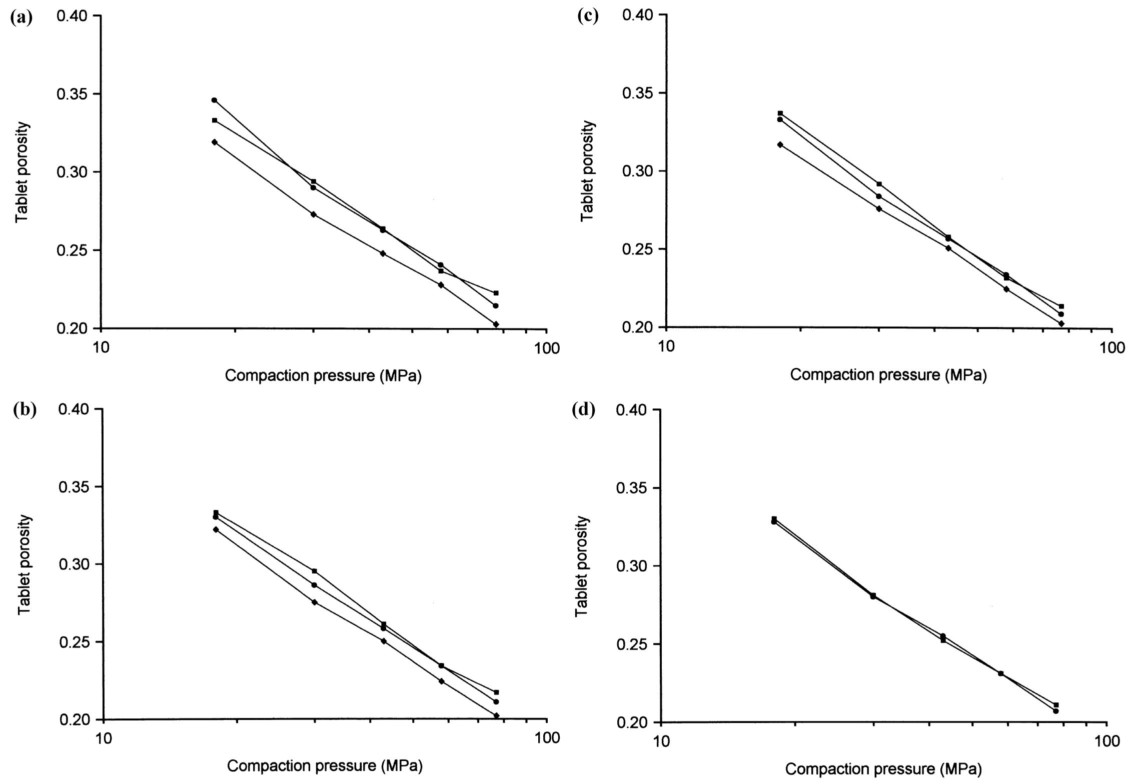

Fig. 5. Tablet porsity of elongated emcompress tablets with different height of the curved segment 'a' (■, a = 0.0 cm; ●, a = 0.1 cm; ◆, a = 0.2 cm) with different thickness to width ratio 'd/b' ((a) 0.3; (b) 0.4; (c) 0.5; (d) 0.6) as a function of the natural logarithm of the compaction pressure.

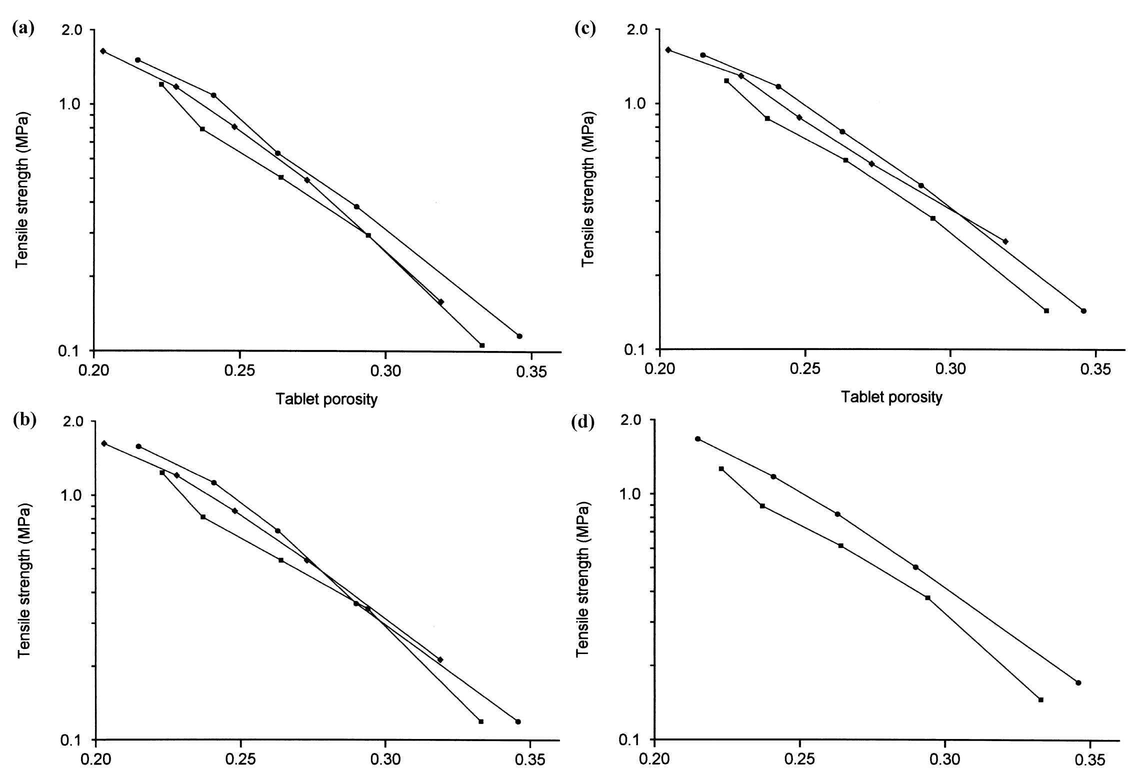

Fig. 6. Natural logarithm of the tensile strength of elongated emcompress tablets with different height of the curved segment 'a' (■, a = 0.0 cm; ●, a = 0.1 cm; ◆, a = 0.2 cm) with different thickness to width ratio 'd/b' ((a) 0.3; (b) 0.4; (c) 0.5; (d) 0.6) as a function of the tablet porosity.

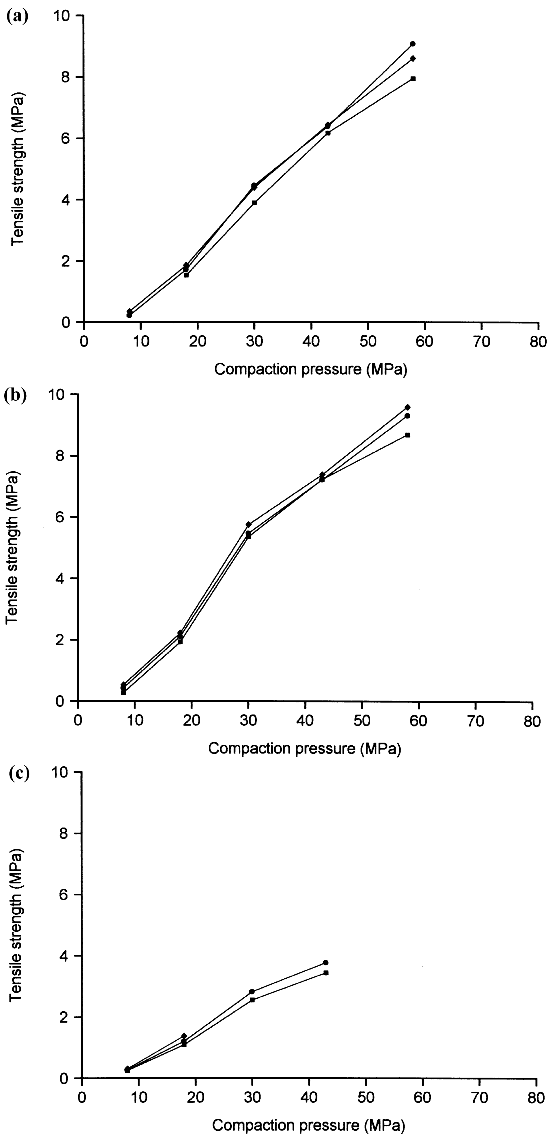

Fig. 7. Tensile strength of elongated Avicel PH102 tablets with different thickness to width ratio 'd/b' (■, d/b = 0.1; ●, d/b = 0.2; ◆, d/b = 0.3) and different height of the curved segment 'a' ((a) 0.0; (b) 0.1; (c) 0.2) as a function of the compaction pressure.

4. Conclusions

The determination of the mechanical strength of elongated tablets, e.g. those of capsule shape, is best evaluated by a flexure test. This allows the derivation of the tensile strength of the tablets which is a more representative value than breaking load, if tablets of different central core thickness and face curvature are to be compared. That the dimensions of the tablet still have some influence on the magnitude of the derived tensile strength is associated with the formation of the tablets and the inhomogeneous nature of the structure.

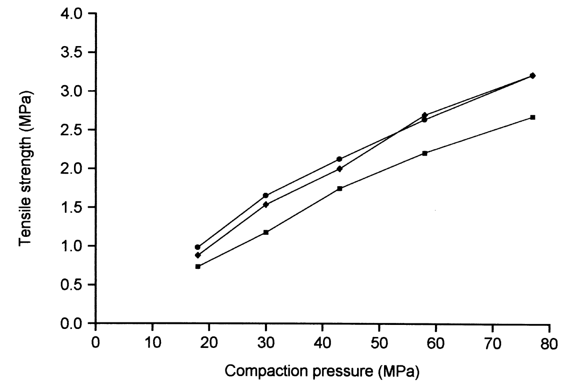

Fig. 8. Tensile strength of elongated Acetylsalicyclic acid tablets with different height of the curved segment 'a' (■, a = 0.0 cm; ●, a = 0.1 cm; ◆, a = 0.2 cm) as a function of the compaction pressure.

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