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Effect of water content on tensile fracture force and deformability of ram extruded cylinders

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Abstract

Wetted powder masses with varying amounts of water were used to study mechanistically the extrusion process in terms of brittleness and deformability of ram extruded cylinders. Mixtures of dicalcium phosphatedihydrate (DCPD) and Avicel PH 101 (70/30%) were massed with water (moisture contents 50, 60, 70, 80 w/w%). The masses were extruded in a ram extruder (25.0 mm in diameter) with a 10.0 mm diameter and 10 mm long die. The moist extrudates were cut to isodiametrical cylinders. The porosity and degree of liquid saturation were determined. The extruded, isodiametrical cylinders were glued on to small brass cylinders and connected to a material testing instrument. The force as a function of displacement was recorded during tensile testing of the extruded cylinders at a loading rate of 1 mm/min. The tensile fracture force (force at failure) and the deformability during the measurement, i.e. an inverse number of the slope of the force displacement curves, were determined. The tensile fracture force of the extruded cylinders decreased with increased porosity. At the same time the deformability of the cylinders increased for all the extruded masses, except the one with the highest porosity. © 1997 Elsevier Science B.V.

Keywords: Ram extrusion; Microcrystalline cellulose; Dicalcium phosphate dihydrate; Water content; Porosity; Mechanical properties; Fracture force (force at failure); Deformability

1. Introduction

From a previous study (Jerwanska et al., 1995) it is known that the degree of compression of a wet powder mass before extrusion and the force needed to extrude the material are related to the porosity and degree of liquid saturation of the mass during the extrusion. It was shown that extrusion occurred at high degree of liquid saturation (near 100%), and thus that an increased water content of the powder mass increased the porosity of extruded cylinders.

The extrudate produced must be able to break into short segments that are sufficiently plastic to

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be rounded by spheronization. This requires a wet powder mass which shows a balance between brittleness and deformability. Such mechanical properties of moist extrudate formed are related to the porosity of the specimen.

Physically and chemically different powders and different levels of moisture have different effects on mechanical properties of mixtures used in pharmaceuticals. The moisture can cause either an increase or decrease in mechanical strength (Schubert, 1975; Kristensen et al., 1985a,b). Different Avicel grades with different moisture levels present in the formulation have also been found to exhibit different tensile properties (Khan and Pipel, 1986) and rheological properties (Raines et al., 1990). The range of levels of water required to enable successful spherical pellets to be prepared was found to be dependent on the ratio of microcrystalline cellulose and excipient (Bains et al., 1991). Lustig et al. (1995) demonstrated that, for a fixed level of microcrystalline cellulose the quantity of water required to prepare the best spherical pellets was controlled by the solubility of the other ingredients.

The aim of the present paper was to characterise the mechanical properties of the ram extruded cylinders in terms of brittleness and deformability as a function of water content. An axial tensile testing method was used to determine the tensile fracture force and deformability of extruded cylinders.

2. Materials and methods

Dicalcium phosphate dihydrate, DCPD (Calipharm, Albright and Wilson, Ph. Eur., UK) and microcrystalline cellulose (MCC), Avicel PH 101 (FMC Corp. USA) in proportion 70/30% w/w were used as a test materials.

2.1. Primary characteristics of the test materials

Measurements of the apparent particle density and the weight-specific surface areas are described in the preceding paper (Jerwanska et al., 1995).

2.2. Dry and wet mixing and ram extrusion

Blending and extrusion were performed as described previously (Jerwanska et al., 1995) using a laboratory ram extruder (Harrison et al., 1985) fitted with a die of diameter 10.0 mm and 10 mm length. The extrudate was cut immediately after extrusion into isodiametrical cylinders.

2.3. Determination of mechanical properties of extruded cylinders.

Ten isodiametrical cylinders (10 × 10 mm) of each water content were tested by adhesion method (Schubert, 1975). The isodiametrical cylinders were glued between a pair of adapters (small brass cylinders) by cyanoacrylate adhesive (Cyanolit 203, Ean Benetter AB, Stockholm Sweden) and connected to the material testing instrument as described by Nyström et al. (1977). By the application of tensile force to the adapters the

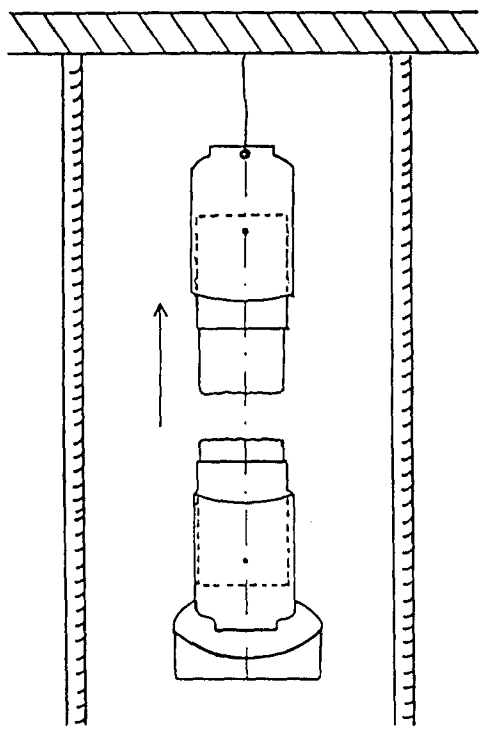


Fig. 1. Axial tensile testing of extruded cylinders.

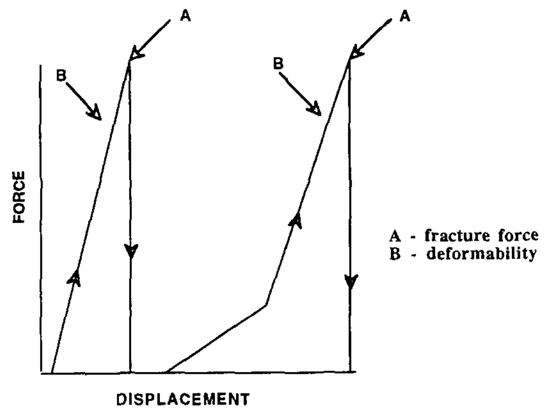


Fig. 2. Tensile fracture force as a function of displacement: (A) force at failure (fracture force); (B) deformability (slope from force-displacement profile).

cylinders were strained at a loading rate of 1 mm/min until they broke (Fig. 1).The force as a function of displacement was recorded. The tensile fracture force, i.e. force at failure (A in Fig. 2) and deformability, i.e. an inverse number of the slope of the force displacement curves obtained during tensile testing (B in Fig. 2) were determined. The mechanical properties of extruded cylinders are listed in Table 1.

3. Results and discussion

3.1. Mechanical properties of extruded cylinders

One method to describe the plasticity of the moist mass is to measure the tensile strength of the extruded cylinders, and to describe the deformability by determining the slope of the force curve obtained during tensile testing.

3.1.1. Force-strain relationships

Moist agglomerates possess a degree of brittleness. When a brittle extrudate is strained, the granule packing undergoes rearrangements until the fracture occurs. An axial tensile testing of the extruded cylinders gave force-displacement curves with two phases (curve to the right in Fig. 2). Initially only a slight increase in the force occurs with appreciable displacement. In the next phase the force rapidly increases, in an almost linear manner until the fracture occurs (A in Fig. 2). The first stage is usually a rearrangement of the particles within the extrudate. As the applied strain stress increases the rearrangement ceases and the system deforms elastically or plastically. Elastic deformation is a reversible process while plastic deformation and fracture is an irreversible process. As a consequence of a further increase in strain stress the fracture of the extrudate occurs. The slope of the linear part of the curve was used to determine deformability (B in Fig. 2). In addition, it seems that the force displacement curves have a different appearance depending on the formulation. For the extruded cylinders with the lowest water content the initial phase of the curve was entirely missing (curve to the left in Fig. 2)

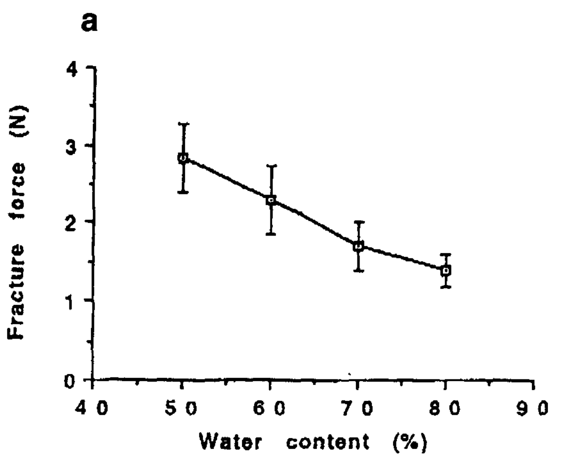
3.1.2. Tensile strength

The tensile strength is of interest in connection with spheronization, where different forces induce strain to moist extrudates causing the breakage of the extrudate strands to short segments on the friction plate. According to the tensile strength studies by Schubert (1975) and Schubert et al. (1975), the tensile strength is defined as the maximum tensile force per unit of the plane cross-sectional area of the bulk material at right-angles to

Table 1
Mechanical properties of extruded cylinders of diameter 10.0 mm[a]

Water content (%)	Extrusion force at steady state (kN) $n = 3$	Porosity (%) $n = 3$	Force at failure (N) $n = 3$	Deformability (μm/N) $n = 10$
50	4.8	51.06	2.84 (0.73)	57.62 (10.32)
60	2.5	56.53	2.28 (0.69)	67.48 (20.43)
70	1.4	58.21	1.69 (0.48)	74.62 (18.55)
80	0.9	62.56	1.40 (0.33)	73.17 (24.45)

[a] Standard deviations are given in parentheses.



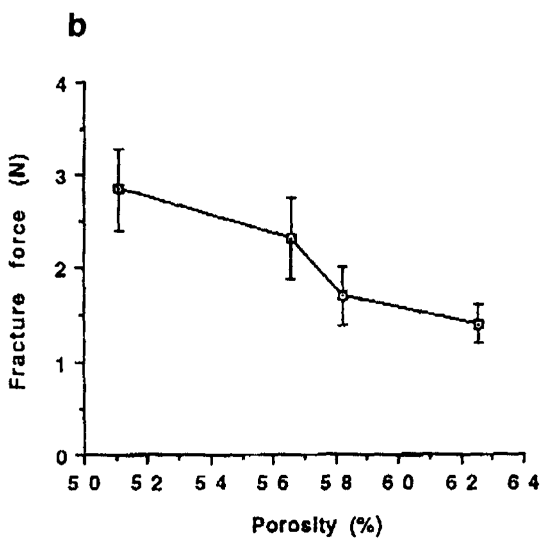


Fig. 3. Tensile fracture force as a function of (A) water content; (B) porosity. The error bars represents the 95% confidence interval for the mean, using Student's t-factor.

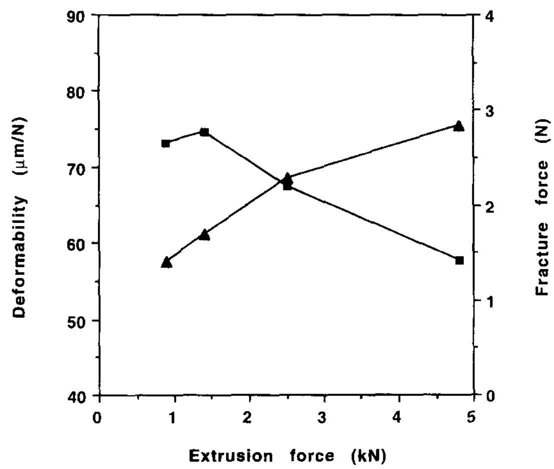


Fig. 4. Deformability and fracture force as a function of extrusion force at steady-state. (■) Deformability; (▲) fracture force.

the direction of the tension. This definition indicates the difficulties of measuring the tensile strength of the moist extruded cylinders. These cylinders must be strong enough to be bonded to the adapters without damage. Furthermore, the fracture face (cross-sectional area) should be in a plane and at right-angles to the direction of the tensile force. An axial tensile testing of the extruded cylinders indicated that the actual fracture face was not in a plane, but followed the surface of the particles and was angled rather than occurring at right-angles to the direction of the tension. For these reasons it was not possible to estimate accurately the area of the cross section of the extruded cylinders for calculation of the tensile strength, as it is defined. It was therefore decided for the present study to record the tensile fracture force, i.e. force at failure, instead.

The tensile fracture force was as low as 1.40–2.84 N and decreased with increased moisture content (Fig. 3).

A possible explanation for this is that increased moisture content reduced particle-particle interactions in the mass, and tensile fracture force of the extruded cylinders decreased. In the previous paper (Jerwanska et al., 1995) it was shown that the porosity of the extruded cylinders increased with increased water content. The increase in porosity could decrease the contact area between particle surfaces and accordingly weaken the particle-particle interactions, resulting in the observed behaviour.

3.1.3. Deformability

During spheronization the initial break down of the extrudate is dependent on its brittleness (fracture force), while subsequent rounding is a function of the deformability.

Deformability of the moist extrudate compromises its ability to be strained without breaking.

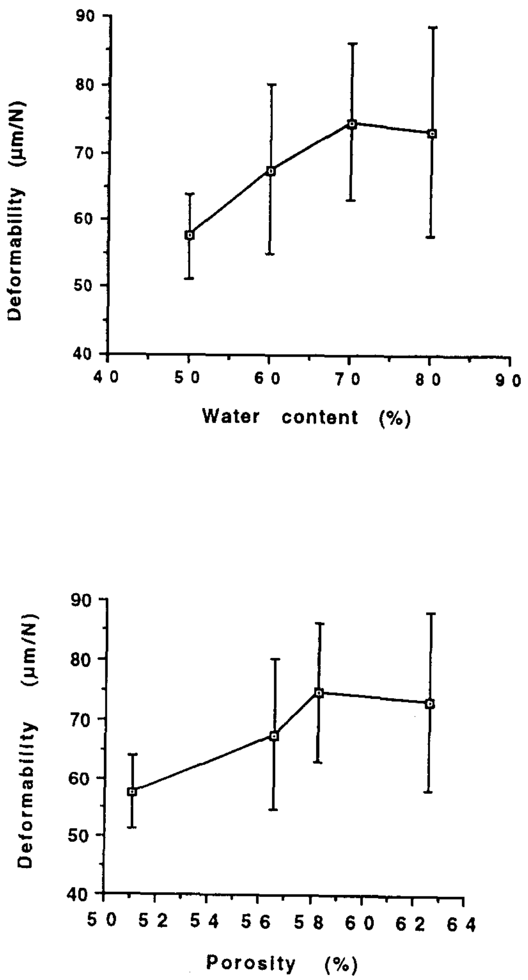


Fig. 5. Deformability as a function of (A) water constant; (B) porosity. Confidence interval as in Fig. 3.

When a force is applied to the mass particles rearrange in the mass and deformation takes place. The deformability of the moist extrudate can be described as the inverse value of the slope of the force displacement curve obtained during tensile testing. The force required to maintain steady-state flow was earlier used to characterise the mechanical properties of ram extruded cylinders in terms of porosity and degree of liquid saturation (Jerwanska et al., 1995).

The results in this study showed that the values of the steady-state force, i.e. extrusion force, were of importance both for the tensile fracture force and deformability of the moist extruded cylinders. An increasing extrusion force gave an increased fracture force and decreased the deformability of the cylinders (Fig. 4). The more porous the extrudate, the smaller the extrusion force. The deformability of the extruded cylinders increased with increased porosity for all extruded masses, except the one with the highest porosity (Fig. 5). This might be because a plateau level was reached above which there were limited changes in deformability with porosity. The changes in porosity were obtained by changing the water content in the mixture. There are, however, other possible mechanisms for changing porosity which could influence the effectiveness of the process. Such factors as the type of microcrystalline cellulose, particle size and shape of the particles require further investigation.

4. Conclusions

The purpose of this study was to characterise the extrusion properties of wet powder masses by ram extrusion and to obtain the mechanical characteristics, the tensile fracture force and the deformability of the resulting extrudates.

For extrudates containing 30% of Avicel PH 101 and 70% of dicalcium phosphate, it was found that an increasing porosity caused by change in the water content of the extrudates decreased the tensile fracture force and increased the deformability. An increased porosity increased the separation distance between the particles and decreased the contact area between particle surfaces, thus reducing particle-particle interactions.

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