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Extrusion of highly unsaturated wet powders: Stress fields in extruder barrels

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ABSTRACT

Wet glass bead powders with solid-to-liquid volume ratios between 2 and 3 were extruded through cylindrical dies by means of a ram extruder. The vertical stress exerted by the ram and the wall normal stress above the die were recorded. A continuum mechanical model was developed to calculate stress fields in the extruder barrel as a function of the load superimposed by the ram. The model predicts that the vertical stress, the wall normal stress and the wall shear stress decrease exponentially above the die entry region. A sudden increase in wall normal and wall shear stresses is observed where the powder in the die vicinity starts to be vertically accelerated. The stresses then decrease to zero from this point to the die which is in agreement with experimental observations. Calculated wall normal stresses are consistent with measured values.

1. Introduction

Solid particles in wet powders are bound by a liquid binder which exists as discrete liquid bridge or a continuous phase enclosing several particles. Air inclusions have a strong impact on the flow properties of wet powders. Frictional forces at particle contact points and capillary forces result in wet powders being more cohesive than saturated systems such as suspensions and pastes. However, the role of the binder is dual as it increases cohesion but also acts as a lubricant between the particles.

The pharmaceutical and food industries frequently extrude wet powders to make stable press agglomerates which are then further processed. If the amount of liquid is small, e.g. to reduce drying costs or obtain a definite texture, there is a risk of blocking by particle interlocking and interparticle friction in the die entry region [1–3].

Shear flow of wet powders (and pastes) leads to the formation of shear layers in which the relative position of particles remains unchanged [1,2,4,5]. Whilst extruding a wet powder into a rectangular die, stagnant zones in the die entry region are bounded by such shear layers [1,2]. These layers form an approximately conical exit geometry [1]. In this zone of initially highest acting shear stresses — also referred to as transition shear zone — the particles rearrange, thus reducing local porosity and increasing saturation. This triggers the reduction of gas inclusions and development of suspension structure with disappearance of Coulomb friction. This results in major shear deformation in this domain. The wet powder in the core flows as a plug with a less dense particle packing and a lower degree of saturation than in the transition shear zone. The transition shear zone propagates upwards with time and comprises more material in the vicinity of the barrel axis [6].

In ram extrusion of pastes and wet powders with large binder quantities, the relationship between the extrusion pressure exerted by the ram and the extrusion velocity is frequently described using an empirical approach from Benbow and Bridgwater [2]:

$$p = p_1 + p_2 = 2(\sigma_0 + \alpha v^m) ln(D_0/d_{die}) + 4(\tau_0 + \beta v^n)(l_{die}/d_{die}) \quad (1)$$

where D_0 is the barrel diameter, d_{die} the die diameter and v the ram velocity. σ_0, α, m, τ_0, β, and n are empirical parameters which are determined from a series of measurements with varying ram velocities and die lengths. The first term in Eq. (1) describes an uniaxial extension from a barrel into a die, the second term describes the pressure drop in the die land.

The model from Benbow and Bridgwater is based on the assumption that the wall shear stress is independent of the local pressure. Highly unsaturated systems, i.e. wet powders in the pendular or funicular state, require higher extrusion pressures than pastes and wet powders in the capillary state. Usually, a peak pressure has to be overcome before a steady state is reached, provided that no liquid phase migration [2,7–10] occurs.

So far there has been no description of the stress states in highly unsaturated wet powders being extruded. Based on approaches from bulk solid mechanics by Janssen [11], Schulze [12] and Motzkus [13], a semi-empirical model was developed to calculate vertical stresses, wall normal stresses and wall shear stresses in the barrel above the die during ram extrusion. Knowing these stresses, dies and die entry regions can be optimized for specific materials to minimize the risk of blocking and to ensure stable extrusion processes.

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2. Materials and methods

Wet powders were prepared by mixing glass beads with a Newtonian binder in different proportions. The glass microspheres Spheriglass® 3000 (Potters Indsutries Inc., Valley Forge, PA, USA) used are manufactured from a soda-lime (A) glass. They are hard, spherically shaped and have a volume weighted median diameter $x_{50,3}$ of 11.2 µm. We used Nutriose® FM06 (Roquette, Lestrem, France) as binder, a dextrin derived from maize available as an agglomerated powder. Aqueous solutions of Nutriose® FM06 show Newtonian flow behavior. The concentration of Nutriose in demineralised water was adjusted to 63.

In the first step, the glass beads were mixed with the Nutriose powder in a free fall mixer with a vessel volume of 0.5 l. The rotational speed was 60 rpm, the mixing time 60 min. The powder mixture was filled into a Duplex kneader (Laboratory Kneader MKD 0,6-H60, IKA-Werke GmbH & Co. KG, Staufen, Germany) where demineralised water was added dropwise at a kneader rotational speed of 50 rpm. Then the material was kneaded for 1 h at 50 rpm while the temperature of the kneader walls was kept at 20 °C by means of a circulating water bath. Subsequently, the wet powder mass was pressed through a sieve with a mesh size of 4 mm to make it granular so that it could be metered easily into the extruder barrel.

The relationship between the bulk density of the wet powders and the vertical normal stress in the extruder barrel was determined using a material testing machine (Zwick Z010, Zwick GmbH & Co. KG, Ulm, Gemany) equipped with a 10 kN load cell. A barrel made from brass with a diameter of 20 mm and a length of 210 mm was put into a double wall fitting which was tempered at 20 °C by a circulating water bath. The bottom of the barrel was closed by a temporary cover (Fig. 1b). A ram with a diameter of 20 mm was mounted on the traverse of the Zwick. 10 g of wet powder was placed in the barrel and compacted with a ram velocity of 0.01 mm/s until a compaction pressure of 255 bar was reached. During compaction the actual height of the ram and the force applied was recorded by the Zwick control software. Aspect ratios H/D_0 of the height of a powder compact and its diameter were smaller than unity at any compaction pressure such that a decrease of the acting vertical stress by wall friction could be neglected. This was verified by measuring compaction curves at different filling heights. During compaction, the wall normal stress was measured 8 mm above the cover by means of a 350 bar pressure transducer (MPS 01210-0000-3,5CB, Dynisco, USA) to determine the lateral stress ratio against the vertical stress in the active-elastic state of stress [14].

Before extruding the powders, they were pre-compacted in the Zwick at 20 °C. The aim of this step was to establish a well-defined mechanical prehistory of the wet powders and better reproducibility of the extrusion trials. 10 g of powder were compacted to a vertical stress of 32 bar at a speed of 0.5 mm/s. This step was repeated four times to have a total mass of 40 g. The proportioning of the powder in four parts guaranteed homogeneous compaction.

Wet powders were extruded through cylindrical dies in batch quantities. The trials were carried out in the Zwick, too. The vertical stress applied by the ram and the wall normal stress in the barrel 11.5 mm above the die were measured simultaneously. A technical drawing of the setup is shown in Fig. 1a. Double wall fittings maintained the temperature of the walls at 20 °C. The inner diameter of the dies was 12 mm. Their lengths were 10 mm, 15 mm or 20 mm. The ram velocity was 0.2 mm/s. The extrusion was stopped when the ram was about 3 cm above the die. The time at stoppage and the exact position of the ram were noted down for later reconstruction of the measured stresses as a function of the distance between the die and the ram. After extrusion the plunger was pulled out of the barrel with a tensile force being smaller than the compressive force during extrusion by two orders of magnitude. No remains were found on the plunger. Hence erroneous load signals by powder fines between the barrel and the ram were negligible. Extrusion trials were duplicated.

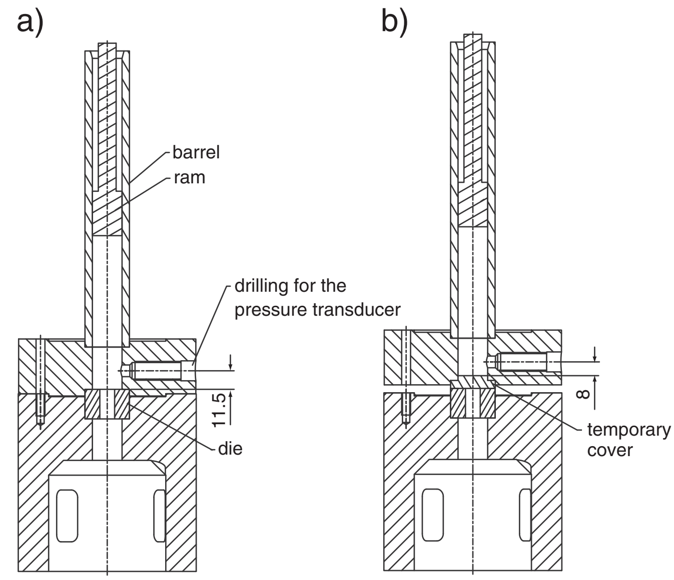

Fig. 1. Technical drawing of the setup: a) for the ram extrusion, b) for the compaction with the material testing apparatus.

A ring shear tester (RST-XS, Dr. Dietmar Schulze Schttgutmesstechnik, Wolfenbüttel, Germany) was used to measure yield loci and wall yield loci of the wet powders. The granular wet powder was pressed through a sieve with a mesh size of 0.25 mm with a spatula to exclude big air inclusions in the shear cell. The sieved powder was filled in a shear cell (type XS-Mr) with a cross section of 24 cm^2. Yield loci were measured at 12 pre-shear stresses up to 23 kPa, each yield locus determined by five measuring points. Commercially available shear cells reach stresses below the stresses during extrusion. The increase in bulk density by higher stresses would be minor. This is why it was assumed that the relationship obtained between the effective angle of internal friction and the bulk density can be extrapolated to higher stresses/bulk densities. Wall yield loci on a wall material sample of lathed, disk-shaped brass were determined in a wall shear cell, type WM. Wall shear stresses were determined for seven normal loads between 0.3 and 20 kPa. All measured wall yield loci were straight lines through the origin so that again an extrapolation towards higher normal stresses was assumed to be justified. All yield and wall yield loci were measured in duplicate.

3. Theory

The relationships described below are mainly based on approaches developed by Motzkus [13] and Schulze [14] for designing hoppers. Their models allow for the approximation of the stress fields in dry bulk solids during filling and emptying. To adapt these models to batch wet powder extrusion, the following assumptions are made:

- Gravitational forces are negligible.
- The stress state is two-dimensional. It is assumed that the largest and the smallest of the three principal stresses decide whether the powder flows, independent of the intermediate principal stress [15].
- The vertical stress is uniformly distributed over the cross section.
- The shear zone in the outer die entry region is considered as an axially symmetric conical wall. The apparent angle of wall friction is equal to the internal angle of friction at steady-state flow in the shear plane which follows from individual yield loci of the wet powder. The material above the die entry region is referred to as the cylindrical part.