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Characterisation of the flow behaviour of pharmaceutical powders using a model die–shoe filling system

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Abstract

In the pharmaceutical industry it is important to understand the factors that influence the flowability of a powder and to identify suitable experimental procedures which can be used to evaluate powders and guide the selection of process parameters. This paper determines the flowability of seven pharmaceutical powders using a model die–shoe filling rig. Dimensional analysis is employed to provide a framework for the interpretation of experimental results and to guide the development of procedures to extrapolate the results to other die and shoe geometries. It is demonstrated that the variation of mass delivered into a standard die as a function of shoe velocity provides a measure of flowability. Qualitative and quantitative studies were undertaken using a high-speed video system and by determining the critical shoe velocity, i.e. the shoe velocity above which incomplete filling occurs.

Keywords: Measurement of powder flow; Die–shoe filling rig; Critical velocity; High-speed video; Rotary press

1. Introduction

The flow properties of a powder are important in pharmaceutical tablet manufacturing as most processes, such as mixing, granulation, drying, milling and compaction involve powder flow. The transfer of powders between these unit operations involves flow in hoppers, pipes, chutes etc. The flow of powders can be divided into 1) slow (i.e. hopper flow) where energy is dissipated through inter-particle friction [1,2] and 2) rapid flow (i.e. avalanches) which are dominated by collisions between particles [3]. Parameters such as dimensionless shear rate [4] were proposed to describe various flow regimes. During a manufacturing process, these different types of flow can occur concurrently, with the details of the flow behaviour influenced by the loading experienced by the particles (gravity, vibration, body forces, interaction with air flow and the container boundaries etc.), the fundamental powder characteristics (such as particle size and size distribution, morphology, material composition and density), operating conditions (i.e. moisture, temperature, static charge) and the current state of the powder (i.e. tapped, consolidated, aerated, free flowing etc.). The flow behaviour of a powder is brought about by a combination of the factors listed above; it is therefore difficult to characterise the flow properties in a universal way for all applications. Indeed, most flow characterisation techniques focus on specific aspects, such as measuring: flow rates through orifices of different size; the angle of repose; the energy to stir a powder bed; cohesion and internal angle of friction of the powder; bulk and tap densities; the formation of avalanches, etc. There is a vast amount of literature, patents, standards and specialised books and monographs dedicated to detailed descriptions of powder flow measurement methods, for example [5].

The above techniques are based on different principles and it has been recognised that the choice of flow characterisation technique should be made in relation to the process under investigation [6]. Yet, correlations have been found between techniques based on tapped density measurements, orifice flow, angle of repose and avalanching behaviour [6–8]. These techniques have been simplified and adapted to evaluate pharmaceutical powders, i.e. Refs. [9,10].

From a die fill point of view, however, Schneider et al., [11] have demonstrated that different flow measurement techniques

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can provide a different rank order of flowability. It is therefore important that flow characterisation is carried out using a device that captures the physical phenomena involved in die fill. Hopper flow has been investigated extensively and many of the flow characterisation methods described above are employed to assist hopper design. Flow into closed dies has, however, received less attention. In this paper we focus on powder flow through the delivery system of a rotary tablet press; more specifically on the flow mechanisms involved in die filling. We begin by describing the filling mechanisms of various tablet presses and examine a particular rotary press in detail. A model die–shoe filling system is used to capture the effects of gravity feed and the effect of air pressure on die fill. Experiments on typical pharmaceutical excipients and powder mixtures are presented. The flow is described qualitatively using high-speed video recording. A flowability metrics is introduced, which extends procedures developed to examine hopper flow [1] to account for the velocity of the feed shoe. The results are discussed in relation to the parameters of a rotary tablet press and the scaling of powder flow from the experimental system to the rotary press is analysed. This work builds on previous studies of die filling in the context of powder metallurgy and ceramic processing [12–14,11] and pharmaceutical powders [15].

2. The feed system of tablet production presses

The tablet compaction sequence typically includes die fill, tablet compression and ejection. Tablet presses can be divided into two broad categories: single station presses, which are driven by an eccentric cam, and multi-station presses, where the dies and punches are mounted on a rotating turret. We examine these machines from a die fill perspective.

In single station presses, the powder is fed into the die from a hopper and a feed shoe. The bottom punch is stationary during filling. The feed shoe is moved above the die opening, executes a number of shakes and is withdrawn. During this process, the powder is deposited into the die cavity. Depending on make or model, the powder movement in the feed shoe may be facilitated by a feeding mechanism (helical screws or rotating paddles, for example). The filling mechanism described above is very similar to typical presses employed in metal powder compaction, where a shoe executes a number of passes in order to fill the die. The filling process is essentially gravity assisted and the principal parameters are the shape and size of the feed shoe and feed shoe kinematics. For older presses, the mechanisms executing punch motion and die fill are usually mechanically interlinked and the shoe kinematics is dictated by the operating speed of the press. Modern presses allow for a more independent adjustment of fill parameters.

Single station presses are mostly used in research and development; while rotary presses are used for high volume production (i.e. hundreds of thousands of tablets per hour). Die fill on rotary presses is more complex. The operating cycle of a rotary tablet press is illustrated in Fig. 1. As the die Table 1 rotates as indicated in Fig. 1a, each tooling station passes successively through the die fill mechanism (4), compression rollers (6 and 7) and ejection cam mechanism (8). The characteristics of die fill are described below. The filling system consists of a mass flow hopper connected to a feed frame. In modern feed frames the powder is circulated using up to three rotating paddles to ensure uniform die fill and powder movement through the fill system.

In the following, we discuss the feed shoe of a Fette 1000 production press (Manufactured by FETTE GMBH, Schwarzenbek, Germany), to identify the feed mechanisms and the typical ranges for the operating parameters. The feed frame (Fil-o-matic type) consists of three wheels, for dispensing, feeding and metering, which are interlinked by interchangeable gear mechanisms; we consider the case where all wheels operate at the same speed. Each wheel is composed of 12 paddles.

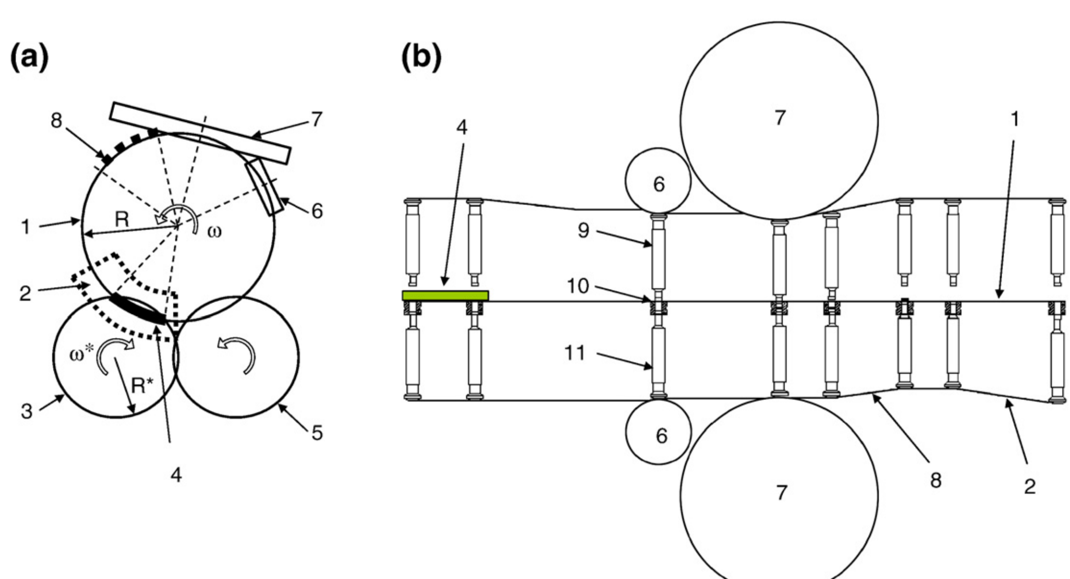

Fig. 1. Rotary press production cycle a) top view, b) unfolded view; 1 — die table, 2 — fill cam, 3 — feed wheel, 4 — die fill area, 5 — metering wheel, 6 — pre-compression roller, 7 — main compression roller, 8 — ejection cam, 9 — upper punch, 10 — die, 11 — lower punch.