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Dependence of ejection force on tableting speed—A compaction simulation study

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ABSTRACT

Using a compaction simulator, it has been shown that tablet ejection force increases with increasing tableting speed during compression of powders lubricated with 1% magnesium stearate. Faster tableting speed corresponds to higher coefficient of friction, μ, but does not affect residual radial die wall (RDW) stress. The higher μ suggests the presence of less lubricant at the tablet–die wall interface due to the shorter time available for migration of lubricant to the interface. This mechanism can also explain the sudden occurrence of powder sticking problem upon formulation scale up. The elevated ejection force due to higher μ also results in higher RDW stress at the moment of slip initiation, which proportionally increases ejection force.

1. Introduction

To successfully manufacture a large batch of tablet, ejection force, i.e., force required to push tablet out of die after compaction, must not be too high. A high ejection force is indicative of high friction at the tablet–die wall interface. Excessive friction can damage tablet and reduce tooling life by wearing. A small weight percentage of boundary lubricant is routinely added to pharmaceutical formulations to reduce ejection force during tableting, a practice known as "internal lubrication". The internal lubrication strategy is also effective in dealing with progressive accumulation of powder on tablet tooling, a phenomenon known as "punch sticking". Severe sticking compromises pharmaceutical elegancy of the final drug products, causes tablet weight variations, and leads to inaccurate dose. The addition of lubricant is, however, a double-edged sword because it also results in detrimental effects to tablet quality, such as lower mechanical strength, longer disintegration, and slower dissolution [1–3]. A successful formulation must contain an optimum amount of lubricant to harvest the beneficial effects of lubrication, i.e., reduced ejection force and sticking-free manufacture, without causing other unwanted effects. Owing to its important roles on the quality of tablet, lubrication has been extensively studied in the field of pharmaceutical tablet formulation [4].

The level of lubricant in a formulation is typically determined by trial and error within the typical range of 0.5%–2% (wt%), depending on the type of lubricant employed. Magnesium stearate is by far the most commonly used lubricant in tableting. It has been observed that a powder with acceptable tableting performance on a laboratory tablet press may become problematic on a high speed tablet press upon scale up, e.g., low tablet mechanical strength [5]. It has also been observed that a well-behaving formulation at the laboratory scale may suddenly have punch sticking problem during large scale tablet manufacturing at a high speed. Clearly, a reliable prediction of changes in compaction properties upon scale up is practically important. In this study, we investigate the effect of tableting speed on ejection force using a compaction simulator.

2. Materials and methods

We used microcrystalline cellulose (MCC, Avicel PH102, Philadelphia, PA) and compressible sugar (DiPac, Domino Sugar, Baltimore, MD) to represent plastic and brittle materials, respectively. A co-processed MCC and mannitol powder (Avicel HFE102, Philadelphia, PA) was used for studying ejection properties in absence of lubrication.

Powder lubrication was carried out by blending 198 g of each powder with 2 g of magnesium stearate (USP, Mallinckrodt, NJ) for 10 min at 25 rpm in a V-shaped blender (Patterson-Kelley, East Stroudsburg, PA, 1 qt volume). Powder compaction was performed using a compaction simulator (Presster, Metropolitan Computing Corporation, East Hanover, NJ). Punches and die assembly passed between two compaction rolls at linear speeds of either 0.126 m/s (100 ms dwell time) or 1.265 m/s (10 ms dwell time). The dwell time corresponds to contact time between the compaction roll and flat region of the punch head. The compaction rolls were 250 mm in diameter. Flat round toolings with a diameter of 10 mm were used to make tablets. The ejection

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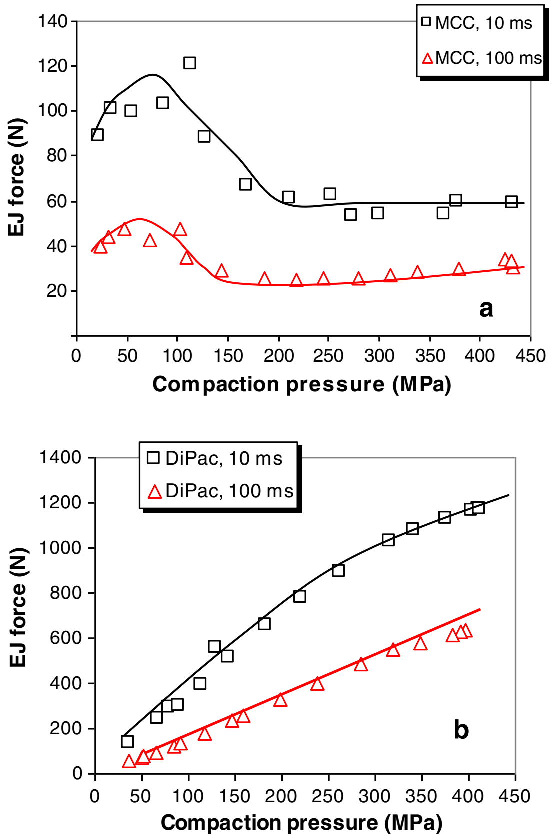

Fig. 1. Faster tableting speed corresponds to higher ejection force for lubricated powders: (a) microcrystalline cellulose and (b) compressible sugar. Black squares, 10 ms dwell time; red triangles, 100 ms dwell time.

angle was 7.0°. Effect of punch inertia on ejection force was quantified by running empty die at identical speeds, and appropriate correction was made (52.4 ± 3 N for 10 ms dwell time and 14.3 ± 2 N for 100 ms dwell time). In-die separation between the upper and lower punch tips and force on both punches were recorded as a function of time. Radial die wall (RDW) stress was obtained by using an instrumented die. Two bonded strain gages, arranged in a fully active Wheatstone bridge, covered the portion of the die where compaction took place, allowing a maximum tablet thickness of 7 mm. The calibration of the RDW stress measurements was performed using a rubber tablet under the assumption that RDW stress is equal to the axial loading stress for the rubber tablet, which behaves liquid-like under stresses [6,7]. In this study, tablet thickness ranged from 2.5 to 5 mm for all tablets. The coefficient of friction, μ, between tablet and die wall is calculated using Eq. (1),

$$\mu = \frac{F_{ej}}{\sigma_r \times \pi \times d \times h} \quad (1)$$

where F_{ej} is the peak ejection force, σ_r is the residual RDW stress at the end of decompression phase and before ejection, d is the diameter of the die (10 mm in this study), and h is tablet thickness at the end of decompression or unloading step.

Before studying ejection properties of unlubricated powders, the die was first thoroughly cleaned with isopropanol to remove any trace amount of lubricant. Then a number of tablets were made at ~100 MPa until the measured ejection force was nearly constant. The unlubricated powder was then filled to the die and compressed.

3. Results and discussion

On average, ejection force at 10 ms dwell time is approximately two times that at the 100 ms dwell time for lubricated (with 1% magnesium stearate) MCC and compressible sugar (Fig. 1) [8]. This behavior is similar to that of MCC and lactose lubricated with 0.5% magnesium stearate [9] but differs from that of a powder (8% MCC and 92% dicalcium phosphate dihydrate) containing 0.2% magnesium stearate [10]. Ejection force of compressible sugar is MCC under identical tableting speed (Fig. 1).

To gain some insights to the observed effect of tableting speed on ejection force, both σ_r and μ are examined as a function of tableting speed. We use μ, instead of ejection force, to characterize lubrication efficiency because it is not affected by variations in tablet thickness (Eq. (1)). We have found that the σ_r does not depend on tableting speed for all powders tested (Fig. 2). Thus, σ_r the observed effect of tableting speed on ejection force. However, μ is higher at faster tableting (Fig. 3). Therefore, the change in ejection force is attributed to the change in μ. Interestingly, μ falls within the same range for MCC and DiPac (Fig. 3), which significantly differ in both chemical compositions and mechanical properties. This suggests that very different ejection forces for the two materials (Fig. 1) are largely driven by the different residual RDW stress not by μ. The similar μ suggests that the relevant interface properties that determine μ are similar even for very different powders during ejection. A sensible explanation is that the lubricant, which is shared by different powder systems, plays a dominant role in tablet ejection behavior.

One possible explanation to the elevated μ at higher tableting speed is the presence of less lubricant at the tablet–die wall interface. It is consistent with the observation that a higher level of lubricant leads to lower ejection force. This mechanism is plausible only when the lubricant possesses certain mobility, allowing its time-dependent redistribution to the interface, which was suggested previously.[11–13] We

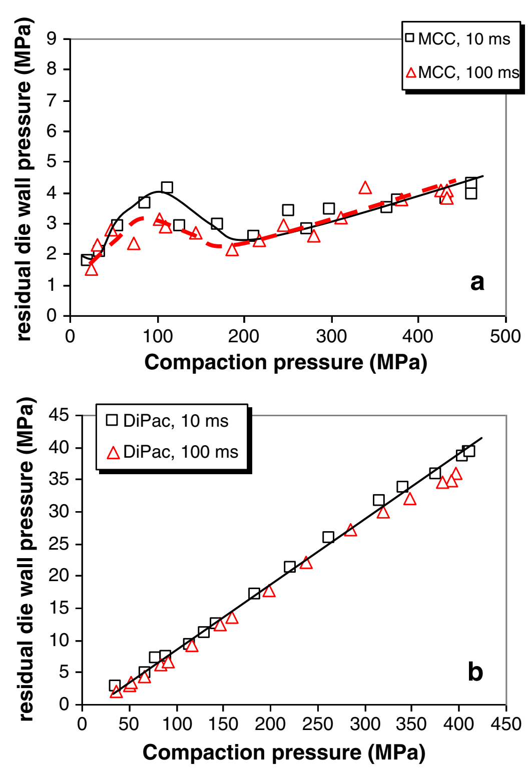

Fig. 2. Lack of effect of tableting speed on residual die wall stress for lubricated powders: (a) microcrystalline cellulose and (b) compressible sugar. Black squares, 10 ms dwell time; red triangles, 100 ms dwell time).