Powder Technology 315 (2017) 410–421

Contents lists available at ScienceDirect

Powder Technology

journal homepage: www.elsevier.com/locate/powtec

Characterizing the powder punch-face adhesive interaction during the unloading phase of powder compaction

Shrikant Swaminathan [a], Brian Ramey [b], Jon Hilden [b], Carl Wassgren [a, c, *]

[a] School of Mechanical Engineering, Purdue University, West Lafayette, IN 47907, USA
[b] Eli Lilly & Company, Corporate Center, Indianapolis, IN 46285, USA
[c] Department of Industrial and Physical Pharmacy, Purdue University, West Lafayette, IN 47907, USA

ARTICLE INFO

Article history:
Received 30 November 2016
Received in revised form 23 March 2017
Accepted 1 April 2017
Available online 5 April 2017

Keywords:
Adhesion force
Picking and sticking
Instrumented punch

ABSTRACT

The objective of this work is to measure the adhesive force profile between a punch face and powder compact in-die during the decompression phase of a tablet compaction process. An instrumented punch was developed for use on a single station compaction fixture mounted on a universal testing machine. Adhesive force-displacement profiles were measured for acetaminophen, ibuprofen, lactose, mannitol, several grades of micro-crystalline cellulose, and blends containing magnesium stearate as a lubricant. The degree of compact solid fraction, the number of compacts produced in a row, and concentration of magnesium stearate in the blends affected the adhesive force profiles. The maximum adhesive force increased with increasing compact solid fraction and decreasing magnesium stearate concentration. An increase in the number of compression cycles increased the adhesive force between the powder compact and punch face, but the compression speed did not play a significant role in the adhesive interaction, at least over the range of speeds examined in the current work.

1. Introduction

One of the difficulties faced during the production of pharmaceutical tablets is powder sticking to the punch faces during compaction [1]. Sticking occurs when the adhesive stresses between the punch face and powder exceed the powder cohesive stresses within the tablet. The adhesive stresses have been shown to be a function of the tablet formulation and process parameters [1]. For a commercial tableting operation in which tablets are formed in rapid succession, sticking can be particularly problematic as powder accumulated on the punch face is repeatedly compressed [2].

In addition to sticking, powder-punch adhesion can also lead to picking. Picking is a special case of sticking where powder is pulled away from the compact in the vicinity of debossed features on the tablet face such as letters, numbers, symbols, and breakaway lines. Picking and sticking lead to poor tablet quality and incomplete or missing identification features on the tablets, potentially leading to rejection of those tablets [3]. Furthermore, if picking and sticking does occur, tablet press tooling must be cleaned before the tableting process can be resumed, which is time consuming and costly.

In most cases, picking and sticking problems are addressed using a trial and error approach. Having measurements of the adhesive force time history during a powder compaction process would be useful in order to quantify and compare the degree of adhesion for different formulations, punch surface finishes, and processing parameters. In addition, these adhesive measurements could be incorporated into numerical models such as finite element method (FEM) simulations for powder compaction [4], in order to predict sticking and picking and thus decrease the reliance upon extensive experimentation.

2. Background

This section begins with a brief review of the phenomena that can lead to powder-punch adhesion. The following sub-sections describe methods used to measure adhesion at particle and bulk powder scales.

2.1. Mechanisms leading to adhesion

The adhesive force between a punch face and powder is a result of several different mechanisms including van der Waals forces, electrostatic forces, capillary forces, and contact melting [2,5,6]. Van der Waals forces occur between all materials and are most significant

* Corresponding author.
E-mail address: wassgren@purdue.edu (C. Wassgren).

http://dx.doi.org/10.1016/j.powtec.2017.04.003
0032-5910/

when surfaces are in close proximity (< 100 nm) [7]. Capillary forces arise from liquid bridges in the gap between contacting bodies. In dry, cold compacted powders, liquid bridging can be neglected as a significant source of adhesion [8]. Electric double layer forces can arise from charged surfaces with an intermediate liquid layer, but are only relevant for separation distances smaller than approximately 5 μm [9]. Electrostatic (Coulomb) forces arise from an accumulation or depletion of charge on materials. Tribocharging of materials, especially non-conductive ones in low humidity environments, is a common source of charge transfer [10]. This force is most relevant for particles under 100 μm [9,11]. Contact melting occurs for materials that have a low melting point. The heat generated during powder compaction may partially melt particles onto a punch face resulting in adhesion.

Clearly, the mechanisms leading to adhesion are complex and, hence, are the focus of many research studies. In the current work, rather than identify the specific mechanisms causing adhesion, the net adhesive force between powder and a punch surface is measured directly. The following sub-section describes some of the adhesion force measurement techniques currently used in the pharmaceutical industry.

2.2. Techniques for measuring adhesion

The adhesive interaction between a punch face and powder has been characterized by measuring the maximum adhesive force, F_{adhesion}, the work required to separate two adhered surfaces, W_{adhesion}, or the mass of powder adhered to a punch face, M_{adhered}. These quantities can be measured at the particle or bulk scales. In general, the techniques used to measure the force at the particle level require more complex equipment in comparison to methods used to measure the force at the bulk level. Several of the experimental measurement techniques used to characterize powder-punch face adhesive interactions are discussed in this section. A more comprehensive discussion of the different measurement techniques is given by Podczeck [12].

2.2.1. Characterization of adhesion by particle-level measurements

Two common measurement techniques used to characterize the adhesive interaction at the particle-level are force microscopy techniques and centrifuging techniques. Force microscopy involves the measurement of the interaction force between a sample and a probe as a function of their separation distance. The interaction force is measured using a calibrated spring or, more recently, a piezoelectric crystal. The separation distance between the two surfaces is monitored using an optical technique such as light interferometry. The Surface Force Apparatus (SFA) was the first technique based on force microscopy [13]. Force microscopy techniques have been improved to create more specialized techniques such as atomic force microscopy (AFM), lateral/frictional force microscopy (LFM), and ultra high vacuum atomic force microscopy (UHV-AFM) [14–17]. A comprehensive discussion on force microscopy techniques may be found in Wang et al. [2] and Podczeck [12]. Force microscopy is becoming a standard technique for particle-level adhesion measurements since it allows for precise measurement of contact force and separation distance. However, these techniques are time consuming and require specialized equipment.

A popular technique used in the pharmaceutical industry to measure adhesion forces for individual particles is centrifuging [18]. In this technique, particles are adhered to an outward facing surface and subject to rotation, for example in a centrifuge. The particle will leave the surface when the centrifugal force on the particle equals the adhesion force,

$$F_{\text{adhesion}} = m\omega^2 R, \tag{1}$$

where m is the particle mass, ω is the rotation speed, and R is the distance between the particle and the axis of rotation. This technique is much simpler and easier to perform than force microscopy methods; however, information on the adhesion force as a function of surface separation distance is unavailable.

These particle-level techniques have two significant limitations. First, a large number of particle-level measurements are needed for statistical significance, especially since irregularly shaped particles may have different adhesive force profiles depending on their orientation. And second, these particle-level techniques are not typically performed for particles undergoing significant elasto-plastic deformation or fracture, which would be the case during a compaction process. Hence, powder adhesion is also studied at the bulk level [1,8,19-23].

2.2.2. Characterization of adhesion by bulk-level measurements

There have been several methods described in the literature for characterizing the adhesive force during the detachment phase of tablet compaction [1,20-22]. Naito and Nakamichi [21] measured the "slipping force" between an upper punch face and a tablet, which was defined as the force applied at a given radius required to twist the upper punch off the tablet surface. Tablets were made in a single station tablet press fitted with a modified upper punch and split die assembly. After the tablet was compressed, the upper punch was not retracted from the die and instead the punch and die assembly was transferred to a separate device to measure the slipping force. This slipping force was measured three times. If sticking occurred, then a larger force value was measured during the first punch rotation. The force measured in the second and third rotations was due to friction as opposed to adhesion. A challenge with this method is that the measured force is dependent on the fidelity of the transfer to the slipping force assembly. If the equipment is jostled, then it is possible that the adhesive bond between the powder and punch face may change. More significantly, the torque required to twist a compact from a punch surface is not a measure of the force required to pull a tablet off the punch surface in a perpendicular direction, as would be the case in a tableting operation.

An alternate approach to measuring the adhesion force was devised by Mitrevej and Augsburger [22]. These authors measured the sweep-off force between the lower punch and the tablet, and thus characterized adhesion between the tablet and lower punch surface. As with the previously described measurement method, there are practical concerns with this technique. First, the measured force is a combination of the adhesion force and the inertial force required to sweep the tablet off the punch (the sweeping action occurs rapidly). Second, the adhesion force is measured after ejection from the die. Frictional interactions with the die wall during ejection and elastic rebound of the tablet after the upper punch retracts, especially upon leaving the die, may cause changes to the stress state at the lower punch-compact interface. Lastly, as with the "slipping force" technique described previously, the sweep-off technique is not a direct measure of the perpendicular pull off force, but instead is a shear force measure.

Mullarney et al. [20] used a punch with a detachable face in a rotary tablet press. The mass of powder stuck to the punch surface was measured periodically during a tableting operation to determine sticking tendency. Although measuring the mass of the powder stuck to the punch can give a good estimate of sticking tendency in practice, it does not provide adhesion force information that can be used in a predictive manner such as in a finite element method model.

Waimer et al. [1] developed a punch with a central bore to accommodate an instrumented pipe for measuring the adhesion force in a single station tablet press. A schematic of the punch is shown in Fig. 1. The instrumented pipe contained strain gauges capable of measuring very small strains. A steel disk was attached to the end