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Predicting the density and tensile strength of viscoelastic soy powder compacts

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ABSTRACT

The key physical property of compact density is important for scale up and influences the mechanical properties such as tensile strength. Hence, the objective of this research was to develop semi-mechanistic models for density of Soy Flour (SF) and Soy Protein Concentrate (SPC) powder compacts as a function of viscoelastic properties and process variables. Each of the powders was compacted at a punch speed of 5 mm/min to final compaction pressures in the range of 26–230 MPa, in a 13 mm diameter cylindrical die. Pressure–time profile during compaction, compact height and force relaxation during the dwell time were also recorded. Compact density was measured under maximum pressure and 24 h post ejection. The force relaxation data was fit to linear Maxwell model for a viscoelastic solid ($R^2 > 0.99$) and the viscoelastic properties of the pure solid material were estimated by extrapolating to zero compact porosity. Although density of compacts from either powder was found to be comparable, SPC produced much stronger compacts compared to SF due to higher bonding. The estimated compact density under pressure was found to be much higher than the pycnometer measured true densities. The contact area between two particles was estimated using Lum and Duncan-Hewitt (1999) using median particle size, pressure–time during compaction and material viscoelastic properties. Power law and logarithmic models that express the compact density and tensile strength respectively as a function of total viscoelastic contact area in a compact were successfully developed ($R^2 > 0.95$–0.99). Thus, viscoelastic properties were found to influence compact density and tensile strength through their influence on the contact area.

1. Introduction

The density of a powder compact is a very important physical property since the scale-up and final tablet mechanical properties depend on it (Rowe and Roberts, 1996; Hancock et al., 2003). The tensile strength of a compact is a measure of its resistance to attrition. Density and hardness measurement which is a measure of tensile strength are classified as a Critical Quality Attributes (Yu, 2008).

The density and tensile strength of compacts made from viscoelastic materials are dependent on the area of contact between particles and their bonding strength. Being composed of amorphous carbohydrates and proteins, defatted soy powders undergo viscoelastic deformation during compaction. Although a few reports are available in the pharmaceutical tablet compaction literature, research dealing with the influence of viscoelastic properties on food powder compact properties of density and tensile strength is almost non-existent. Only more recently, Palzer (2009) proposed a model to predict the tensile strength of dextrose syrup solids based on the complex viscosity of liquid from which they were produced. It must be noted that powder compaction involves large deformation of particles. Hence, consideration of viscoelastic properties at the actual compaction pressures and strains might is necessary to predict these properties. For several soy protein isolates, Caillard and Subirade (2011) investigated their compaction properties using the Walker Equation which does not consider the influence of viscoelastic properties on compressibility.

The tensile strength and density of a powder compact are largely determined by two factors: interparticulate contact area and the bonding strength between individual particles. The contact areas, during and after compression depend on the time-dependant flow of material, which occurs in conjunction with instantaneously responding elastic deformation (Patel et al., 2006; Rumpf et al., 1978). The time dependent deformation of particles in turn is described by the viscoelastic properties of a material. Bonding between particles depends on the type and magnitude of bonding mechanism which is dependent on chemistry of molecules (Nystrom et al., 1993; Ferrari et al., 1996). Thus in case of dry compaction of powders, adhesion forces are mainly linked to increasing Van der Waals forces caused by increased contact area resulting from viscoelastic deformation of the particles (Palzer, 2009). Hence, interparticulate contact area is a critical property

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that directly affects the density and tensile strength of any powder compact. Experimentally, the influence of increasing contact area between bronze particles (whose compaction is rate independent), on compact density has been shown by Fischmeister et al. (1978). Lum et al. (1998) quantitatively modeled the density of compacts made from MMA/PMMA copolymer as a function of compaction speed and pressures, based on contact area. However, no published research has focused on studying the influence of viscoelastic contact area on density or tensile strength of real powder systems occurring due to large deformations. Hence, the objective of this research is to model compact density and tensile strength of soy powders as a function of estimated contact area between particles, which is influenced by material viscoelastic properties and process variables.

2. Materials and methods

2.1. Materials

Defatted Soy Flour and Soy Protein Concentrate (*henceforth abbreviated as SF and SPC respectively*) used in this research were obtained from Solae LLC (Remington, IN). The powders were equilibrated in glass jars for two days in order to minimize the effect of environmental humidity changes on powder properties and prevent further moisture uptake. The containers were stored at ambient conditions (~23 °C and 50% RH). Although the actual process of the powder manufacture was not known, the general manufacture process is described by Endres (2001).

2.2. Methods

2.2.1. Powder bulk and particle physical property measurements

The following physical properties of both powders were characterized:

1. Moisture content: loss on drying in a convection oven (DN-84, Scientific American products) at 105 °C for 24 h ($n = 3$) following methodology of Fitzpatrick et al. (2004).
2. Particle morphology: Scanning Electron Microscopy (Quanta™ 3D DualBeam™, FEI, OR) (500–2000× for SF and 250–500× for SPC). Magnification was chosen based on qualitative assessment of particle sizes.
3. Particle size distribution: laser diffraction (Mastersizer 2000E equipped with Scirocco 2000 manual dry dispersion unit, Malvern instruments, Westborough, MA) ($n = 2$).
4. Bulk and tapped densities: 10 g of each powder was taken in a 50 mL measuring cylinder and tapped until an equilibrium volume was reached (150 taps) following the procedure of Hollenback and Peleg (1983). The bulk and tapped densities were calculated based on volume after pouring and at the end of tapping respectively.
5. True density: Nitrogen Pycnometer (Accupyc 1330, Micromeritics, GA) ($n = 20$ for Soy Flour, $n = 10$ for Soy Protein Concentrate) (Fitzpatrick et al. 2004).

2.2.2. Soy powder compacts density, tensile strength and volumetric expansion

The variation of Soy Flour and Soy Protein Concentrate powder compact density, tensile strength and volumetric expansions (elastic and viscoelastic) was studied as a function of compaction pressure. A sample size of 0.475 g each of Soy Flour and Soy Protein Concentrate powders was compacted in the cylindrical die with 13 mm diameter and 11 mm height at a constant speed of 5 mm/min ($n = 2$). Compression was started at initial powder bed heights of 5 and 8.5 mm for SF and SPC respectively. A large range of pressures was chosen to prepare compacts from the lowest to highest possible solid fractions for each of the powders. Preliminary experiments were conducted by decreasing the compression force from highest pressing force possible for the current load cell (42.26 kN) to the critical pressing force (lowest possible compaction force that could produce a compact with a measurable breaking force of 1 N). The highest pressure chosen to make compacts to characterize the density, tensile strength and relaxation was 30.69 kN, beyond which the increase in pressure yielded negligible increase of density/tensile strength. The following final compression forces were used to make compacts (kN):

A. Soy Flour: 8.9, 11.12, 13.34, 17.79, 30.69.
B. Soy Protein Concentrate: 3.34, 6.68, 13.34, 17.79, 22.24, 26.69, 30.69.

Once the pre-set force was reached, the punch was kept constant for 1 min (dwell time) before decompressing with a speed of 508 mm/min. The compacts were then ejected and the thickness measured immediately within 1 min (h_{in}) using calipers with a resolution of 0.01 mm (Absolute series 500, Mitutoyo, USA). The initial compact density was calculated based on these measurements (ρ_{in}). The compacts were then stored in glass vials for 24 h for further volumetric expansion following which they measured for weight and thickness (h_{fin}) to calculate the final compact density (ρ_{fin}). The compact tensile strength (σ_t) was measured using the diametral method, based on the breaking force (F), compact diameter (D) and thickness (h_{fin}) (Eq. (1)) using at a testing speed of 0.6 mm/min. The testing speed was chosen based on the work of Sun and Hou (2008).

$$\sigma_t = \frac{2F}{\pi D h_{fin}} \tag{1}$$

Three phases of volumetric expansion of a powder compact have been reported (Haware et al., 2010; Hein et al., 2008; Van der Voort Maarschalk et al., 1997):

a. Elastic relaxation following decompression.
b. Elastic relaxation following compact ejection.
c. Viscoelastic relaxation during storage.

Although the overall elastic relaxation (combining phases a and b) and viscoelastic relaxation were measured, they will only be briefly discussed in this research. These relaxations result in a decrease of density consequently also decreasing the contact area (Sun, 2011).

2.2.3. Measurement of soy powder compaction and viscoelastic properties

The compaction response (stress–time relationship during compaction) was characterized based on the raw force–time data obtained during making the compacts ($n = 2$). For stress relaxation studies, 0.475 g of the powder was compacted to the same pressures as the ones used to make compacts. However, the maximum pressure was limited to a force of −17.79 kN since the instrument was unable to hold the punch constant beyond this pressure. Only one experiment was possible for each powder at a given pressure. Once the preset force was reached, the punch was held constant for 3000 s and the relaxation in force with time was recorded. The force, time and displacement data recorded by the instrument was used to calculate the relaxation modulus.

2.2.4. Estimation of viscoelastic contact area between particles

Lum and Duncan-Hewitt (1999) developed a relationship (Eq. (2)) to calculate the contact area between two particles resulting from small viscoelastic deformations taking into account both