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Compaction of food powders: The influence of material properties and process parameters on product structure, strength, and dissolution

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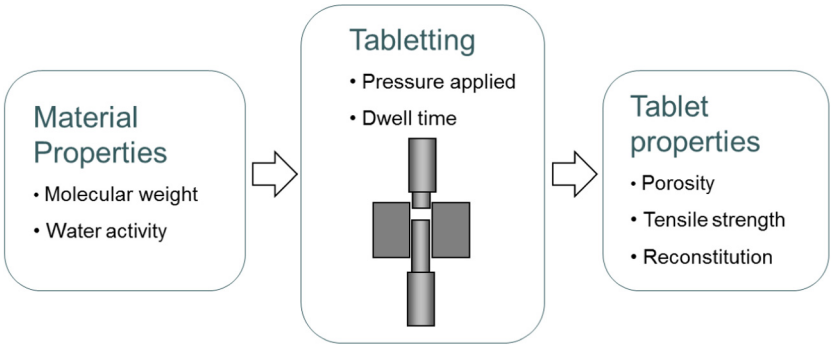
HIGHLIGHTS

- Amorphous powders compacted at varying molecular size, a_w, pressure, times.
- Temperature rise above T_g led to microsintering and strong bridge formation.
- Extent of sintering enhanced by increasing dwell times if $T - T_g > 10$ K.
- Dissolution regime of tablets determined by erosion vs. disintegration mechanisms.

GRAPHICAL ABSTRACT



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ABSTRACT

During pressure agglomeration of food powders, it is often difficult to control the final product properties due to their complex material behaviours. The current study aims to better elucidate how the quality of a compact is impacted by the material characteristics of the raw materials as well as the process conditions applied.

An amorphous powder was compacted under various conditions to investigate the influence of material properties, such as water activity and molecular weight, and process parameters (pressure and dwell time) on tablet porosity, tensile strength, and dissolution time.

With increasing pressure, the porosity decreased and the strength increased, due to the formation of bridges between particles at their contact points. If the glass transition temperature (T_g) was low (due to moisture-induced plasticisation or more extensive enzyme hydrolysis) and the compaction pressure high, then the temperature of the powder surpassed its T_g; resulting in local occurrences of temporary glass transition within the powder bed, allowing for enhanced deformation and microsintering between particles. This led to stronger interparticle bridges and overall tablet crushing strength – this strength increase was particularly strong for longer dwell times, as the extent of microsintering was augmented.

Abbreviations: a_w, water activity; DE, dextrose equivalents; DSC, differential scanning calorimetry; IT, "Instant" (agglomerated); MW, molecular weight; *RH*, relative humidity; SEM, scanning electron microscopy; *T*, temperature; T_g, glass transition temperature; XRT, X-ray tomography.

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Tablets dissolved more quickly at higher water temperatures, but more slowly for higher compaction pressures – this may be explained by a change of dissolution regimes (erosion vs. disintegration). A higher molecular weight resulted in slower dissolution due to slower liquid penetration due to wetting, viscosity-building, and pore collapsing effects.

The current work could be used to optimise the processing parameters, leading to improved product properties, particularly mechanical strength and reconstitution performance.

1. Introduction

Pressure agglomeration is a common process in the food, pharmaceutical, and chemical industries to compact powders into mechanically stable agglomerates without the use of a liquid binder (Palzer, 2007a, 2007b). Unit operations such as tableting can be used to apply high pressures to produce such agglomerates; during tabletting, a pre-dosed quantity of powder is compressed uniaxially to form a tablet with a defined size, shape and density. For consumer acceptability, tablets must have a strong enough structure to withstand fracture and friability during transport, but must also have acceptable dissolution properties for their intended purpose. However, food materials can undergo complex transitions when exposed to heat and pressure, influencing the way they deform under an applied stress, thereby making processes difficult to optimise. The aim of the current study is to better understand the interplay of the material properties of amorphous food powders and the processing conditions applied during tabletting, and how these affect the final product at a pilot scale.

1.1. Amorphous materials

The supra- molecular structure of a solid material has strong influence on its physical behaviour (Palzer, 2010). Molecules can be arranged in either a highly ordered structure (crystalline materials) or in a statistical non-equilibrium arrangement (amorphous materials). Unlike crystalline materials, amorphous water-soluble materials gradually absorb water with increasing relative humidity, and at equilibrium the water activity (a_w value) is equal to the relative humidity (*RH*), for a given temperature.

Amorphous solids can either be found in their brittle, "glassy" state (viscosity ca. 10^{11}–10^{12} Pa·s) or a less viscous, "rubbery" or "sticky" state (ca. 10^{8}–10^{9} Pa·s). By elevating the temperature, a glassy amorphous material will transform to the rubbery state during a phenomenon called glass transition, which has thermodynamic characteristics similar to a second-order transition (Roos, 2007). The temperature, at which this occurs, is the glass transition temperature (T_g). The T_g of a material can be determined, for instance, by a change in heat capacity using DSC (Differential Scanning Calorimetry).

Glass transition can be described as clusters of molecules obtaining enough kinetic energy to slip past one another, resulting in the observed viscosity decrease. Thus, if the molecules become plasticised or reduced in size, they would be able to slip past each other more easily; as a result, both an increase in water content (plasticisation) and a decrease in molecular weight will depress the T_g of a material. As glass transition does not occur at a single temperature, but rather over a range of temperatures, in the current report the *onset* of glass transition is assumed whenever T_g is mentioned.

1.2. Adhesion mechanisms during pressure agglomeration

As explained by Palzer (2007b), a number of phenomena occur inside the powder during tabletting, including the escaping of air from within the powder bed, rearrangement of particles, mechanical interlocking, as well as particle breakage and deformation, which will occur to varying degrees depending on the deformation behaviour of the materials (e.g. brittleness). All of these lead to increased contact points/surfaces between particles, where inter-particle bridges can be generated. Depending on the material properties and compaction conditions, these bridges may form due to enhanced van der Waals attraction between particles upon plastic or viscoelastic deformation, capillary condensation, or material sintering. Capillary condensation occurs when moisture from the air partially dissolves on the material surface, which for crystalline materials may lead to the creation of liquid bridges that when dried can leave behind a solid bridge, or for amorphous materials may soften the material matrix. Material sintering is the mass flow of a plasticized amorphous material between particles for form a bridge. For powders containing fats, these may melt during compression due to an increase in temperature during compaction, forming liquid bridges between particles that re-solidify after tablet cooling, leaving behind a solid fat bridge. This temperature rise can also lead to the amorphization of some crystalline components (Willart and Descamps, 2008).

The cause for this increase in temperature is due to energy dissipation resulting from the friction between particles, and between particles and the wall surface (Krok et al., 2016). Some authors have measured the temperature increase during compression, e.g. using a thermistor connected to a die by epoxy (DeCrosta et al., 2001) or by infrared measurement (Zavaliangos et al., 2008). The temperature rise can also be heterogeneous within the tablet, and the extent of this heterogeneity is affected by the process conditions, such as the amount of lubricant used (can decrease the friction between the powder and wall surface), and speed of compression (Krok et al., 2016). According to Picker (2003), the temperature of the tablet can increase above 90 °C, even if only briefly, as the tablets will cool considerably after ejection (Travers and Merriman, 1970), and if the press runs for longer periods of time, the entire mass may rise by as much as 20 °C (Nürnberg and Hopp, 1981).

The associated temperature rise observed during compression can have significant effects on the mechanical properties of the material being compressed (Dopfer et al., 2013; Haider et al., 2011, 2012). Haider et al. studied the stress-relaxation behaviour of maltodextrin, an amorphous food powder, and observed a significantly lower relaxation time for a $T\text{-}T_g > 10$ K. This enhanced deformation under an applied stress would thus lead to additional contact points for material bridges to form.

In the current study, the effects of material properties of maltodextrin were studied, incl. molecular mass, water activity, and particle microstructure. These maltodextrins were tableted at varying combinations of pressures and compaction times, and the resulting tablets were evaluated to determine their porosities, tensile strengths, and dissolution times at different water temperatures.