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Compression and mechanical properties of directly compressible pregelatinized sago starches

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ABSTRACT

This study investigates the compression and mechanical properties of directly compressible pregelatinized sago starches in comparison with Spress® B820 and Avicel® PH 101. The sago starch is pregelatinized at 65 °C with different pregelatinization times of 15, 30, 45, and 60 min, creating samples PS1, PS2, PS3, and PS4, respectively. Compressibility of the powders is analyzed by Heckel and Kawakita equations. The compressibility of sago starch is found to be lower than that of its pregelatinized forms, and the compressibility increases with an increase in the pregelatinization time. Avicel® PH 101 is the most compressible among the powders evaluated, followed by PS4, Spress® B820, PS3, PS2, PS1, and sago starch. As for mechanical properties, Avicel® PH 101 is found to have the highest radial tensile strength and the hardest compacts, indicating that it has the highest compactibility, followed by Spress® B820, PS4, PS3, PS2, PS1, and sago starch.

1. Introduction

Starch is widely used as a pharmaceutical excipient, primarily in tablet formulations, functioning as a diluent, binder, and disintegrant [1,2]. Worldwide, corn starch is the most widely used starch in tablet formulations owing to its availability [3]. Because of compression problems, native starches are not suitable for use as excipients in direct compression formulations [4,5]. Pregelatinization is a proven method that renders starches directly compressible [6,7]. As an example, corn starch has been successfully pregelatinized and is commonly used as a directly compressible excipient with the commercial name Spress® B820 [8].

Direct compression is a technique involving compaction of a bulk material whose ingredients are composited to form tablets [9]. Mixing and compressing are the only steps involved in direct compression for the production of tablets, making it preferable in tablet production.

Malaysia is one of the leading sago starch-producing countries in the world [10], mainly for use in food products [11]. Literature reviews show no report as yet on the application of a local sago starch for a directly compressible material in tableting. This study investigates the compression and mechanical properties of pregelatinized sago starch as a directly compressible excipient, and compares it with Spress® B820, a similar existing product; and Avicel® PH 101, a purified-partially depolymerized cellulose with extremely good binding properties in direct compression.

2. Experimental

2.1. Materials

This study used a local sago starch (food grade; Nee Seng Ngeng & Sons, Sago Industries Sdn Bhd) and pregelatinized sago starches. In addition, we used a commercially available pregelatinized corn starch called Spress® B820 (lot S0615476; GPC, Muscatine, IA, USA), as well as the product Avicel® PH 101 (lot 11363; Fluka, Cork, Ireland).

2.2. Preparation of pregelatinized sago starches

Pregelatinized sago starches (PS) were prepared according to Odeku et al. [12] and Adedokun and Itiola [13] with modifications. An aqueous slurry of 20% (w/v) sago starch was heated in a water bath (Grant SUB 36, Royston, England) at 65 °C with stirring at 700 rpm (WiseStir™ HD-30D; Daihan Scientific Co., Seoul, Korea) for 15 min (sample labeled PS1). The resulting paste of sago starch was dried in an oven (WTB Binder, Geprcifte Sicherheit, Germany) at 40 °C for 48 h. The dried mass was then powdered in a laboratory cutter mill (MX-895M, Selangor, Malaysia) to produce course powders. All of the starches were passed through a sieve (180-μm aperture) and stored in a tightly sealed white container before use. Three more batches were prepared with different heating times of 30 (PS2), 45 (PS3), and 60 min (PS4).

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2.3. Scanning electron microscopy

Powder samples were mounted on a stub and coated with gold. Three-dimensional images of the powder samples were taken with a scanning electron microscope (FEI Quanta 200 FESEM, Eindhoven, Holland) with the accelerating voltage ranging from 10 to 12.5 kV.

2.4. Particle size determination

Particle size was determined with a light microscope (Nikon Eclipse 80i; Nikon Instruments Inc., Kanagawa, Japan). Diameter of 300 particles projected on the computer screen was measured and the mean projected diameter was calculated automatically by NIS Element D2.30 computer software.

2.5. Moisture content

A sample of the powder (1 g) was dried in an oven at a temperature of 100–105 °C for 4 h. The percentage loss in weight was calculated as the moisture content.

2.6. Densities

The bulk density (ρ_0) and tapped density (ρ_t) of the powders were determined using a tapped density tester (JV2000; Dr. Schleuninger Pharmaton AG, Solothurn, Germany). A 250-mL glass cylinder was filled with 100 g of powder sample and placed on top of the tapped density tester and the bulk volume was recorded. The cylinder was then tapped 1000 times to a constant volume and the tap volume was recorded. Bulk and tapped densities were calculated based on the ratio of weight to volume. The procedure was done in triplicate. The mean and standard deviations were measured in a process modified from previous studies [14,15]. The true density (ρ_T) of the materials was determined by a helium pycnometer (AccuPyc 1330; Micromeritics, Norcross, USA), wherein the powder sample was weighed and loaded into the sample cell, and the true volume was obtained by calculating the difference in helium pressure before and after loading the sample. Each sample was tested in triplicate [16].

2.7. Total powder porosity

The total porosity (ε) of a powder was calculated from the equation [17,18]

$$\varepsilon(\text{total}) = \left(1 - \frac{\rho_0}{\rho_T}\right) \times 100\%, \tag{1}$$

where ρ_T is the true density and ρ_0 is the bulk density of the powder. The value for ε was calculated three times using the triplicate values obtained for ρ_T and ρ_0, and an average for ε was then obtained from these three calculated values.

2.8. Preparation of compacts

An Enerpac GA3 single punch machine (Globe Pharma, New Brunswick, NJ) equipped with a set of round flat-faced stainless steel toolings with a diameter of 8.00 mm was used in the preparation of compacts. Prior to the compression, a suspension of magnesium stearate in alcohol 95% was used to lubricate the punch faces and the die wall. Ten different compression pressures (from 20 to 200 MPa) were used to prepare the compacts from each material. The powder sample (300 ± 3 mg) was loaded manually into the die and the determined compression pressure was applied for 3 s [1]. The compact weight, hardness, and the dimensions of diameter and thickness were measured after being in storage for 24 h [19]. These data were used to calculate relative density (D), porosity (ε), and degree of volume reduction (C) of the compact at pressure P using the formulae

$$D = \rho_A / \rho_T, \tag{2}$$

$$\varepsilon = 1 - D, \tag{3}$$

$$C = 1 - \rho_0 / \rho_A, \tag{4}$$

where ρ_A is the apparent density of the compact at pressure P.

2.9. Analysis of compression properties

Compression properties of the powder compacts were analyzed according to the Heckel and the Kawakita equations [8],

$$\ln\left(\frac{1}{[1 - D]}\right) = kP + A, \tag{5}$$

$$P/C = P/a + 1/ab, \tag{6}$$

where k, A, a, and b are all constants. Heckel plots of $\ln(1 / [1 - D])$ versus P were established. The slope of the linear plot indicated the value of the constant k, whereas the intercept of the plot gave the value of the constant A. The constant k reflects the deformation of the particle under compression, and the reciprocal of constant k is known as the mean yield pressure (P_y) of the powder. The constant A is related to the particle rearrangement and die filling before deformation and the bonding of the discrete particles, and was used to calculate the relative density (D_a) using the formula

$$D_a = 1 - e^{-A}. \tag{7}$$

The relative density at zero compression pressure, D_0, is related to the initial rearrangement phase as a result of die filling; while the relative density, D_b, describes the rearrangement phase at low pressure, and can be calculated from the formula

$$D_b = D_a - D_0. \tag{8}$$

The Kawakita equation (Eq. (6)) was used to study the relationship between volume reduction of a powder and the pressure applied. The constants a and b can be determined by constructing a plot of P/C versus P. In this plot, the constant a is indicative of the total volume reduction of the powder bed, while the constant b is indicative of the plasticity of the powder, and its reciprocal value (P_k) is related to the yield strength of the particles.

2.10. Analysis of mechanical properties

The mechanical properties of the powder compacts were evaluated by calculating the tensile strength, T using the formula

$$T = 2F / \pi dt, \tag{9}$$

where F is the force required to fracture the compact determined by a tablet hardness tester (Model 6D, Dr. Schleuniger Pharmatron, New Hampshire, USA), d is the diameter of the compact, and t is thickness of the compact. The use of tensile strength in the evaluations was to allow the compact dimensions to be taken into consideration. All measurements were taken in triplicate.