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Research paper

Material and tablet properties of pregelatinized (thermally modified) *Dioscorea* starches

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Abstract

The material and tablet formation properties of pregelatinized (thermally modified) forms of four *Dioscorea* starches have been investigated. *Dioscorea* starches were pregelatinized followed by either oven drying (PS) or freeze drying (FD) and used as excipient in direct compression. The physicochemical, morphological and material properties of the pregelatinized starches have been investigated. The tablet formation properties were assessed using the 3-D modeling parameters, the Heckel equation and the force–displacement profiles. The tablet properties were evaluated using the elastic recovery, compactibility plots and the disintegration test. The results indicate that pregelatinization improved the compressibility and flowability of the *Dioscorea* starches. The high bulk and tap densities of PS coupled with their good flowability offer a unique possibility of the starches being used as filler in capsule formulations. The modified starches generally showed differences in their time and pressure dependent deformation behaviour. PS exhibited higher elasticity during tableting. FD Chinese and FD Bitter showed higher plasticity and low fast elastic deformation than the PS forms of the starches indicating that the FD starches undergo the highest plastic deformation. However, FD starches generally showed higher compactibility compared to the PS forms of the *Dioscorea* starches. While FD White and FD Water showed fast disintegration time and high compactibility, FD Chinese and FD Bitter were non-disintegrating and showed high compactibility. The high compactibility observed with the FD starches appears to be as a result of material change occurring during tableting probably due to the effect of temperature or pressure or a combination of both factors. Thus, FD White and FD Water starches could be useful when high crushing force and fast disintegration are of concern while FD Chinese and FD Bitter, which were non-disintegrating, could find application as excipients for controlled drug delivery.

Keywords: Pregelatinized starch; Material properties; Tableting; *Dioscorea* starches; Yam

1. Introduction

Starch in general plays a prominent role in the production of pharmaceutical tablets. Tropical yam tubers are another potential starch source that could be used in the food and pharmaceutical industries that have not been explored commercially [1]. Previous studies on the material and tablet properties of starches from four different tropical *Dioscorea* (yam) species, namely White yam (*Dioscorea rotundata*), Bitter yam (*D. dumetorum*), Chinese yam (*D. oppositifolia*) and Water yam (*D. alata*), have shown that the four starches varied considerably in their physicochemical and material properties. The amylose content of the starches was 28.8%, 21.6%, 18.8% and 23.3% for White, Bitter, Chinese and Water starches, respectively. Furthermore, Chinese and Bitter yam starches were highly compressible and formed tablets of acceptable crushing force while White and Water yam starches showed poor compression properties and formed weak tablets even at very high compression pressures [2]. When comparing native starches in their applicability for tablet compression

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purposes, yam starches provided much better compaction properties [2]. Moreover, the high starch (70–80% dry basis) content of yam tubers and cheap cost makes them good candidates for the production of industrial starch. However, native *Dioscore*a starches generally exhibited poor flow properties characteristic of other native starches [3].

Physical and chemical modifications have been used to improve the compaction properties of some native starches [4–11]. Typical physical modifications include pregelatinization, particle size and moisture adjustment. Starches from corn, waxy maize, wheat and potato with different amylose/amylopectin ratios have been thermally modified by extrusion [6,7], drum drying [6–8] and controlled pregelatinization-spray drying techniques [7]. The process of gelatinization causes substantial changes in both the chemical and the physical nature of granular starch due to the rearrangement of intra- and intermolecular hydrogen bonding between the water and starch molecules resulting in the collapse or disruption of molecular orders within the starch granule [3]. This results in irreversible changes in the starch properties [12]. Evidence of the loss of an organized structure includes irreversible granule swelling, loss of bifringence and crystallinity [13,14].

To date, most studies on the production of modified starches have been limited to widely available starches such as corn, potato, wheat, tapioca and rice [6–11]. Modified starches from other botanical sources may yield starches with special properties and offer a wide range of functional properties permitting numerous applications. So far, no work has been done to evaluate the usefulness of thermally modified yam starches as directly compressible excipients in tablet formulation. Thus the aim of the present work is to investigate the compaction properties of four *Dioscorea* starches which were modified by pregelatinization followed by either oven drying or freeze drying. The tablet formation properties were assessed using the 3-D modeling parameters, the Heckel equation and the force–displacement profiles, while the properties of the starch tablets were evaluated using the elastic recovery, compactibility plots and disintegration test. The physicochemical, morphological and material properties of the pregelatinized starches were also investigated.

2. Materials

Tubers of four different *Dioscorea* species namely White yam – *Dioscorea rotundata* L., Bitter yam – *D. dumetorum* Kunth, Chinese yam – *D. oppositifolia* L. and Water yam – *D. alata* L. DIAL2 were obtained from local farmers in Ibadan, Nigeria. The starches were extracted from the relevant tubers using established procedures [15]. In the following text only the abbreviations FD will be used for freeze-dried pregelatinized starch and PS will be used for pregelatinized oven dried starch while White, Bitter, Chinese and Water will be used for the starches, respectively. All materials and tablets were equilibrated, produced and stored at 22 ± 1 °C and $45 \pm 2\%$ relative humidity (RH).

3. Methods

3.1. Preparation of pregelatinized starches

The pregelatinized forms of the *Dioscorea* starches were prepared using established methods [7]. 20% w/v aqueous starch slurry was heated at 80 °C with stirring for 15 min. For the oven dried pregelatinized starch (PS), the resultant paste was dried in a hot air oven at 40 °C for 24 h and then powdered using a laboratory mill. All the starches were passed through a 125 μm mesh sieve.

Freeze-dried pregelatinized starch (FD) was prepared by freeze drying the pregelatinized starch in a freeze dryer (Christ GmbH, Osterode, Germany) at −84 °C and pressure of −0.371 bar for 24 h.

3.2. Swelling power and solubility

The swelling and solubility properties of the starches in cold water (22 ± 1 °C) and hot water (85 ± 1 °C) were assessed using established methods [16,17]. Starch suspensions (1% w/w) were prepared in a flask and heated to appropriate temperature for 30 min with shaking every 5 min and left to cool at room temperature and centrifuged for 15 min at 3000*g*. The supernatant was decanted and dried in an oven for 2 h at 130 °C. The residue obtained after drying was weighed to obtain the swelling of the starch [16]. The solubility was calculated as *g* per 100 g of sample on dry weight basis.

3.3. X-ray powder diffraction

The X-ray diffraction pattern was recorded with Co-Kα_1 X-ray radiation (STOE STADI-MP diffractometer, STOE & Cie GmbH, Darmstadt, Germany). The starch powders tightly packed in a sample holder were exposed to X-ray beam at 40 kV and 30 mA. The scanning region of the diffraction angle (2θ) was from 3° to 50°. The total run time was 100 min.

3.4. Scanning electron microscopy

The starch powders and tablets (upper surface and breaking surface) were analysed using scanning electron microscopy (ESEM 30, Philips, Kassel Germany) at an accelerating voltage of 5 keV.

3.5. Water content

The water content was determined by thermogravimetric analysis using TGA 209 in triplicate (Netzsch Gerätebau GmbH, Selb, Germany). The powder was heated with 10 K min^{-1} from 20 to 150 °C.