Effect of spray-drying and extrusion on physicochemical characteristics of sweet potato starch

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Abstract This study aimed to understand the physicochemical characteristics of sweet potato starch following spray-drying and extrusion processes for desirable applications. Spray-dried starch showed formation of agglomerates and decreased in average granular size from 16.5 μm of the native starch granules to 14.1 μm . Spray-drying reduced the percentage crystallinity from 25.3 to 22.6% and showed a slight decrease in the molecular weight of amylopectin from 3.1 to $2.6 \times 10^8 \text{ g mol}^{-1}$. In addition,

changes in the pasting and gelatinization properties, higher final viscosity (454.4 RVU), and less enthalpy change (8.73 J g^{-1}) were reported after spray-drying. Thus, spray-drying resulted in partially gelatinized starch, which can be selected for making more viscous products. Extruded sweet potato starch displayed an amorphous structure, showed total loss of crystallinity, and significant reduction in molecular weight of amylopectin to $0.41 \times 10^8 \text{ g mol}^{-1}$, reflecting complete gelatinization of starch granules during extrusion. Extruded starch showed significant changes in pasting properties, including a display cold viscosity (9.4 RVU). Therefore, extruded starch was suitable for products that require quick solubility and a low final viscosity. Thus, the spray-drying and extrusion processes produce sweet potato starches with particular characteristics that can be used for different and potential applications in industries.

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Introduction

Starch comprises two distinct groups of polysaccharides, amylose and amylopectin. Amylose essentially displays $\alpha(1,4)$ glycosidic bonds, and having a primarily linear structure, with few branches. Amylopectin has a highly branched structure, with $\alpha(1,4)$ glycosidic linked linear chain and 4–5% of $\alpha(1,6)$ glycosidic branch linkages.

Pregelatinized starch has significant industrial applications. Pregelatinization process may convert the native starch into its partially or fully gelatinized form to obtain a gel at room temperature. The pregelatinization process can cause chemical and physical changes on the granules of starch because of the rearrangement of intra- and inter-

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molecular hydrogen bonding between water and starch molecules, and the crystalline zones in the starch granules are partially or completely destroyed; these changes on granular starch depend on intensity of the process (Fu et al. 2012). As a consequence, this starch gives consistency to products without cooking or exposure to high temperatures and/or stability upon storage at low temperatures. Pregelatinized starch has been produced by extrusion, or by heating starch dispersion in a drum-dryer or in a jet-cooking followed by spray-drying (Majzoobi et al. 2011).

Spray-drying is a very quick drying process, which convert drops of liquid solutions or suspensions into amorphous or semi-crystalline particles. By eliminating or expressively decreasing the crystalline fraction of the starch, significant changes may be observed in the distribution of the average size of the granules, crystallinity, swelling power, gelatinization temperature, as well as other physicochemical characteristics of these starches (Liu et al. 2010). Starch dispersion requires heating at temperature above gelatinization temperature for a short time before processing by spray-drying.

Extrusion produces gelatinized starch and may cause loss of the integrity and crystallinity of starch granules, and depolymerization of starch components. The process may result in the breakdown of covalent bonds in the starch, and intense structural disruption and mixing resulting in the modification in the functional properties of starch.

In 2016, the world production of sweet potato (*Ipomoea batatas* (L.) Lam.) roots reached 106.6 million of tons (FaoStat 2017). The main component of sweet potato root is starch. Over the last decade, a tremendous increase has been observed in the amount of studies on the applications of sweet potato starch in food and non-food industries (Zhu and Wang 2014). The pasting behavior of sweet potato starch is characterized by a high peak viscosity; and it show low breakdown, indicating high resistant to high temperatures and shear—characteristics that are important for industrial processing. Sweet potato starch shows high final viscosity and a great tendency to retrogradation (setback).

With the industrial demand for starches with specific technological properties, it is necessary to study new sources of non-conventional starch, as well as new processes. Spray-dried and extruded starches are considered as a natural and safe ingredient, and these starches can be used to promote cold solubility and develop viscosity at low temperature. Many studies have described the use of different types of processed starch, as spray-drying of green banana starch and corn starch, extrusion of potato starch, and heat moisture treatments of sweet potato starch (Izidoro et al. 2011; Fu et al. 2012; Mościcki et al. 2012; Huang et al. 2015). However, the changes in the properties

of starch of tubers processed by spray-drying are unclear. In this line, we investigated the effects of spray-drying and extrusion on the amylose content, molecular mass of amylopectin, crystallinity, morphology, granule size, thermal and pasting properties, swelling power, and solubility of sweet potato starch.

Materials and methods

Materials

Sweet potato roots were collected in São Manuel Experimental Farm (CERAT/UNESP). All reagents were of ACS grade and enzymes were from Sigma Chemical Co. (St. Louis, MO, USA) or Megazyme (Wicklow, Ireland).

Starch isolation

Sweet potato starch was extracted from the Canadian cultivar with 150 days after planting, following the methodology described by dos Santos et al. (2018). The sweet potato starch was characterized as physicochemical analysis, and the results obtained were 11.78% of moisture, 0.45% of ash, 0.89% of lipids, 0.22% of protein, 86.60% of starch (wet basis), and pH 6.12.

Processes

The sweet potato starch was processed by spray-drying and extrusion. These processes were repeated four times for each process. Extrusion cooking was used as a pattern process, due to its widespread applications in food and non-food industry.

Spray-drying process

The spray-drying process was carried out following the methodology described by Fu et al. (2012). Dispersions of starch (8%, m/m, dry starch basis) were prepared with distilled water at 40 ± 1 °C, and were stirred in a thermostatic water bath at 57 °C for 10 min, and beakers contains the starch dispersions were covered with plastic film to minimize water evaporation. After this time, the hot starch dispersions were then spray-dried, using a spray-dryer with a double-atomizer nozzle, with 0.70 mm diameter of the nozzle hole (Spray-dryer, Model MSD 0.5, Labmaq, São Paulo, SP, Brazil). The inlet and outlet temperature were 130 and 105 °C, respectively, and the feed rate was 0.5 L h^{-1} . The flow of compressed air and hot air were 0.40 L min^{-1} and $3.8 \text{ m}^3 \text{ min}^{-1}$, respectively, and the air compressed was set at 6 bars.

Extrusion process

The extrusion process was carried out following the methodology described by dos Santos et al. (2018). The extrusion process parameters were: extrusion temperature in the 1st (20–25 °C) 2nd (40–45 °C) and 3rd (70–75 °C) zones; screw compression ratio (3:1); screw diameter (32.60 mm), die diameter (4.00 mm); feed rate (150 g min⁻¹), and cutting speed (90 rpm). The initial moisture of the starch was 20%.

Amylose content

The determination of amylose content was performed following the protocol of Kasemsuwan et al. (1995), as described by dos Santos et al. (2018). This analysis was carried out in triplicate.

Molecular weight (M_w) and gyration z-average radius (R_z) of amylopectin

The molecular weight (M_w) and gyration z-average radius (R_z) of amylopectin were assessed by high performance size exclusion chromatography with multi-angle laser light scattering and refractive index detectors (HPSEC-MALLS-RI), following the protocol of Yoo and Jane (2002), as described by dos Santos et al. (2018). This analysis was carried out in triplicate.

X-ray diffraction analysis

X-ray diffraction patterns and percentage crystallinity was performed as described by (Nara and Komiya 1983). This analysis was carried out in triplicate.

Morphology and granule size of starches

The granule morphology of native, spray-dried, and extruded starches was studied using scanning electron microscopy (SEM) (Tecnai Spirit, Fey Company, Hillsboro, Oregon, USA), as described by dos Santos et al. (2018).

The granule average sizes and size distribution of native and spray-dried starches were determined using laser light diffraction technique with a He–Ne laser (Mastersizer 2000, Malvern Instruments Ltd., Worcestershire, UK), as described by Mesquita et al. (2016). This analysis was carried out in triplicate.

Thermal properties

Starches samples (2.50 mg, dry starch basis) were studied using a differential scanning calorimeter (DSC) (Model

Pyris 1, Perkin Elmer, Norwalk Connecticut, USA), used with distilled water (7.50 µL). The scanning temperature range was 25–100 °C and the heating rates were 10 °C min⁻¹. This analysis was carried out in triplicate.

The degrees of gelatinization (DG) of spray-dried starch sample were calculated according to the procedure described by Di Paola et al. (2003) as following equation:

$$DG (\%) = [1 - (\Delta H \text{ of spray-dried starch} / \Delta H \text{ of native starch})] \times 100 \quad (1)$$

Pasting properties

Samples were analyzed using a rapid visco analyzer (RVA) (Model Super 4, Newport Scientific, Warriewood, New South Wales, AU), following the protocol of dos Santos et al. (2018). This analysis was carried out in triplicate.

Swelling power and solubility

Swelling power and solubility was performed following the protocol of Schoch (1964), as described by Mesquita et al. (2016).

Statistical analysis

Data were subjected to analysis of variance using the SAS statistical software. When appropriate, the difference amongst means was determined using Tukey's multiple comparisons at the 0.05 probability level ($p < 0.05$). All measurements were made in triplicate and data are presented as mean $\pm$ standard deviation. Statistical analysis was carried out considering, separately, the processes, due to the fact that the selected processes have contrasting properties.

Results and discussion

Starch components and relative crystallinity

The amylose content of sweet potato native starch was similar to those reported in previous studies (between 19.10 and 26.80%) (Hoover 2001; Peroni et al. 2006; Zhu et al. 2011; Abegunde et al. 2013). The processes applied in this study did not change the amylose content of sweet potato starch (Table 1). The molecular weight of amylopectin in native sweet potato starch was higher than that previously reported (5.02×10^7 and 1.77×10^8 g mol⁻¹) (Huang et al. 2015) (Table 1). The gyration radius is related to the theoretical possibility of finding a molecule at a certain distance from the center and depends on the volume

Table 1 Amylose content, molecular weight (M_w), and gyration radius (R_z) of amylopectin and percentage crystallinity from native, spray-dried, and extruded starches

Starch	Amylose content (%)	M_w of amylopectin (g mol^{-1})	R_z of amylopectin (nm)	Percentage crystallinity (%)
Native	$22.00 \pm 0.13\text{a}\delta\Delta$	$3.06 \times 10^8 \pm 0.02\text{a}\delta\Delta$	$291.50 \pm 6.4\text{a}\delta\Delta$	$25.34 \pm 1.29\text{a}\delta$
Spray-dried	$21.64 \pm 1.05\text{a}\delta$	$2.57 \times 10^8 \pm 0.03\text{b}\omega$	$283.70 \pm 1.5\text{a}\delta$	$22.55 \pm 0.49\text{b}\omega$
Extruded	$21.94 \pm 0.87\text{a}\Delta$	$0.41 \times 10^8 \pm 0.05\text{c}\Omega$	$145.00 \pm 0.3\text{b}\Omega$	$0.1 \pm 0.0\text{c}$

Values followed by the same letter do not differ statistically from each other and values followed by the same Greek letter for each treatment do not differ statistically from each other (Greek lower-case letter for spray-drying, and Greek uppercase letter to extrusion), Tukey's HSD test ($p < 0.05$)

occupied by the molecule in the solution (Millard et al. 1997; Zeng et al. 2016). The length of amylopectin branched chains and the diffraction pattern of starch may affect the gyration radius value in the solution (Yoo and Jane 2002).

Spray-drying of the starch slightly decreased the molecular weight of amylopectin, while maintaining gyration radius. As a consequence spray-drying caused changes in the structure of starch.

Extruded starch showed a decrease in molecular weight of amylopectin, and a significant reduction in the gyration radius. The high temperature and intense shear rate within the extruder may break the $\alpha(1,4)$ and $\alpha(1,6)$ glycosidic bonds, as well as the ordered structure of starch such as crystallites and double helices. Amylopectin may be degraded mainly in the outer branched chains (Liu et al. 2010).

The native sweet potato starch displayed an C_a -type diffraction pattern, with most intense peaks at 15, 17 e 23° 2 θ (Fig. 1), indicative of its greater similarity with the A-type polymorph. Previous studies have also observed C- or C_a -type diffraction pattern for sweet potato starch with a higher proportion of short-branched chains of amylopectin (Zhu and Wang 2014; Huang et al. 2015).

The results showed that there was no change in the X-ray diffraction pattern with the spray-drying modification (C_a -type), however, there was decrease in crystallinity (Table 1). Fu et al. (2012) reported a decreased in the percentage crystallinity of corn starch after spray-drying and observed an increase in its degradation with an increase in the heating temperature.

The extrusion process resulted in the complete disappearance of the diffraction peak, this indicate that the crystalline structure of the starch was converted to the amorphous structure (Fig. 1), due to shear forces and temperature (Mościcki et al. 2012). These changes may indicate the breakdown of the double helices of starch structure (Su et al. 2009; Yan and Zhengbiao 2010; Huang et al. 2015).

Morphology and granule size

Sweet potato native starch displayed granules with smooth surface and oval and polygonal shape; a few depressions were also observed (Fig. 1a), similar observations were related by other authors (Hoover 2001; Zhu et al. 2011; Zhu and Wang 2014).

The changes in the morphology of the spray-dried sweet potato starch are shown in Fig. 1b. No drastic changes were observed in the surface structure and the shape of the sweet potato starch granules. However, some granules showed wrinkles, a common observation for macromolecule particles produced by spray-drying (Tonon et al. 2011).

The size of sweet potato native starch granules showed great variability, and showed 65.50% of granules between 11 and 20 μm . The size distribution of spray-dried starch showed a small reduction in the average size of the granules (16.50–14.10 μm , Table 2). After cooling, the swollen granules displayed wrinkles and reduced in sizes. The starch granules processed by spray-drying often show hollow structure in their center while presenting a regular shape (Yan and Zhengbiao 2010), which may explain the reduction in the granule size.

The spray-drying process resulted in increased the proportion of granules with size between 6 and 15 μm and decrease on the granules with size between 16 and 60 μm (Table 2), suggesting that during the pregelatinization stage may occur beginning of the break of granules, due to heating and agitation. Theses small particles, may correspond to parts of the granules, or wrinkled/shrunk granules.

After extrusion (Fig. 1c), a molten mass of gelatinized starch without presence of granules was observed, due to during the process, starch granules are subjected to high shear and temperature. This transformation involves the absorption of water into granules, followed by their swelling and their eventual disintegration to form a homogeneous gel.

Fig. 1 Micrographs of SEM of sweet potato starch: Native **a**, spray-dried **b**, and extruded **c**; Distribution of granule size and diffractogram of starches **d**, **e**

Table 2 Distribution of granule size of native and spray-dried starches

Starch	Granule size distribution (%)						Mean diameter (μm)
	2–5 μm	6–10 μm	11–15 μm	16–20 μm	21–30 μm	31–60 μm	
Native	0.005b	20.470b	26.385b	39.085a	11.670a	2.400a	16.5 ± 0.3a
Spray-dried	0.083a	33.423a	30.248a	30.848b	5.105b	0.297b	14.1 ± 0.4b

Values followed by the same letter do not differ statistically from each other (lower case letter for spray-drying), Tukey's HSD test ($p < 0.05$)

Thermal properties

The onset, peak, conclusion gelatinization temperatures (T_{onset} , T_{peak} , and $T_{\text{conclusion}}$) were of 63.83, 68.94, and 73.50 °C, respectively, for the native sweet potato starch, and were similar to those reported in the literature, as well as the enthalpy change (ΔH), 12.46 J g⁻¹ (Peroni et al. 2006; Abegunde et al. 2013; Huang et al. 2015). Higher values of temperatures and enthalpy change (ΔH) reflect stronger or more ordered crystalline molecular structures (Rocha et al. 2011). The higher gelatinization temperatures of sweet potato starch are related to the presence of strong binding forces within the granule with a high degree of

crystallinity which provided structural stability and made the granule more resistant to gelatinization (Barichello et al. 1991; Singh et al. 2018).

The spray-drying process showed no statistical changes in peak and conclusion temperatures (68.72 and 73.26 °C, respectively) but led to the reduction in the gelatinization temperature range of 9.93 to 8.89 °C, due to the increase in the onset gelatinization temperature (64.37 °C), resulting in a more homogenous distribution of crystals and provided a less structural stability of starch. Temperature range represent initiation of starch gelatinization at which the granules start imbibing water and undergo transition from semi-crystalline to gel state (Singh et al. 2018).

The extent of crystallinity of starch decreased after spray-drying process; as a result, the enthalpy change values were lower than those observed for the native starch, 10.31 J g^{-1} . The low degree of gelatinization (17.27%) was expected and can be attributed to the partially gelatinized granules in SEM micrographs (Fig. 1b), small reduction in crystallinity and low enthalpy change. Extruded starch did not display a curve during the DSC analysis; hence, no values was reported for gelatinization temperatures and enthalpy change. This result confirmed complete loss of the crystalline structure of the granules after extrusion.

Paste properties, swelling power and solubility

Pasting properties are influenced by the distribution of the amylopectin branched chain lengths, amylose content, and diameter of starch granules (Peroni et al. 2006; Tester and Morrison 1990; Yoo and Jane 2002; Yoo et al. 2009). Amylopectin is primarily responsible for granule swelling, whereas amylose and lipid restrict granule swelling (Tester and Morrison 1990).

The native sweet potato starch displayed typical pasting properties of root starch, did not show viscosity at cold temperature. The spray-dried starch did not develop viscosity at cold temperature (Table 3). The peak viscosity indicates represents the point of maximum of swelling of the granules with hot water, which reflects the ability of the starch to hydrate and swell. These results indicate a better alignment of the molecules after this process, leading to an increase in the water-retention capacity of the granules. The spray-drying led to increases in setback. Higher amylose content leads to increased setback, indicating reassociation of amylose molecules leaching out of the swollen granules during heating in an aqueous medium forming a network during the cooling process (setback) (Charles et al. 2005; Yoo et al. 2009). The increase in the final viscosity of the spray-dried starch indicate the slight changes in the starch structure induced by this process.

Extruded starch showed an increase in cold viscosity, reflecting loss of the crystalline structure. Other parameters of pasting properties showed a significant reduction, which may be associated with the small number of intact granules that still exhibit swelling capacity (Zhang et al. 2010).

Swelling power and solubility of spray-dried starches showed 60.25 g g^{-1} and 57.99%, respectively, that were higher than that of native starch (38.47 g g^{-1} and 25.05%), which may be due to the weakening of hydrogen bonds, resulting in decreased interactions between the amylose and amylopectin molecules, as well as between the amylopectin chains. The weakening of bonds is accompanied by the formation of a less stable structure and increased leaching of amylose molecules, resulting an increase in the surface and water inflow in granules (Gomes et al. 2005).

The degradation of the granular and molecular structure of the starch caused by extrusion process resulted in a decrease in swelling power (12.79 g g^{-1}). This observation may be related to shear forces that completely dispersed the starch components and changed the molecular arrangement and reducing in molecular weight, leading to an increased in solubility (58.40%).

The swelling power indicates the ability of water to penetrate starch granules. The crystalline areas of the granules maintain the integrity while permitting swelling, however may not allow complete dispersion of the macromolecules. The amorphous areas rapidly absorb water, allowing swelling. Thus, the mobility of starch molecules increases with temperature, which weakens the binding forces. Thus, the leaching of the soluble components is increased and results in the improvement in the solubility of starch.

Spray-drying and extrusion process lead to partial and total converted of starch to an amorphous mass, respectively. These differences could be observed by less degradation on molecular and structural degradation, as well as, maintaining of crystalline fraction after spray-drying process. Extrusion process showed loss of crystalline fraction, total gelatinization, and increased viscosity at room temperature. These factors make the spray-dried

Table 3 Pasting properties of native, spray-dried, and extruded starches

Starch	Viscosity (RVU)				
	Cold	Peak	Breakdown	Final	Setback
Native	$1.21 \pm 0.04\text{b}\delta\Omega$	$309.71 \pm 0.21\text{b}\omega\Delta$	$92.08 \pm 1.34\text{b}\omega\Delta$	$366.67 \pm 5.91\text{b}\omega\Delta$	$149.04 \pm 7.04\text{b}\omega\Delta$
Spray-dried	$1.39 \pm 0.03\text{b}\delta$	$385.84 \pm 15.90\text{a}\delta$	$121.95 \pm 4.78\text{a}\delta$	$454.40 \pm 26.86\text{a}\delta$	$190.50 \pm 15.71\text{a}\delta$
Extruded	$9.42 \pm 1.62\text{a}\Delta$	$9.32 \pm 1.56\text{c}\Omega$	$5.10 \pm 1.23\text{c}\Omega$	$9.94 \pm 1.36\text{c}\Omega$	$5.72 \pm 0.95\text{c}\Omega$

Breakdown = Peak – Hold. Values followed by the same letter do not differ statistically from each other and values followed by the same Greek letter for each treatment do not differ statistically from each other (Greek lower-case letter for spray-drying, and Greek uppercase letter to extrusion), Tukey's HSD test ($p < 0.05$)

starch able to be used in products that require high viscosity during production, and in the final product. Properties of extruded starch are very important because they will provide the extruded starchy materials with new and relevant properties either for food or non-food applications. These products and process with sweet potato as source can be applied in food and pharmaceutical industries. Characteristics shown by the sweet potato starch may be a factor contributing to lower degradation during extrusion and spray-drying processes. Extrusion is applicable to production of matrix formulations for controlled drug delivery. Spray-drying is useful for drugs soluble, controlled release particles, microcapsules as flavorings and functional ingredients.

Conclusion

The spray-drying process resulted in a decrease in the molecular mass of amylopectin, but without change of the gyration radius. In addition, this process led to the decreased of the crystalline area and enthalpy change, causing greater swelling power, as well as higher final viscosity. Thus, this process allows the production of sweet potato starch with high viscosity, a characteristic desired for use in products that require thickness. Extruded starch showed a decrease in the molecular mass of amylopectin with a change of the gyration radius, indicating the breakage of glycosidic bonds, influencing cold viscosity and solubility. The sweet potato extrudate starch is less prone to retrogradation, forming more dispersed paste with low viscosity and can be applied in industrial products such as soups, creams and flans, which require rapid solubility. Besides these uses, starches with low final viscosity can be used in products with high solids content such as infant formulas and foods for special diets, and as a binder in pharmaceutical formulations. Thus, the spray-drying and extrusion processes can broaden the uses of sweet potato starch.

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