

Spray-Drying of Amylase Hydrolyzed Sweetpotato Puree and Physicochemical Properties of Powder

J. A. GRABOWSKI, V.-D. TRUONG, AND C. R. DAUBERT

ABSTRACT: Spray-drying, which has been used for commercial production of functional ingredients from several fruits and vegetables, has not yet been studied for sweetpotato processing. Thus, the objective was to determine the effects of viscosity reduction of sweetpotato puree with alpha-amylase, maltodextrin (MD) addition, and inlet air temperature on the physicochemical characteristics of spray-dried sweetpotato powder. A face-centered cube design was used to evaluate the effects of amylase level (0, 3.75, and 7.5 mL/kg puree), MD concentration (0%, 10%, and 20%), and inlet air temperature (150 °C, 190 °C, and 220 °C) on powder characteristics. Model-fitting using response surface methodology was performed to examine the effects of independent variables on the moisture content, color, water absorption, solubility, particle size, bulk density, and glass transition temperature. The data were fit to a full second order polynomial equation. However, only the linear and quadratic terms proved to be significant for most dried powder attributes. MD significantly increased powder solubility, altered the hue value, and raised the glass transition temperature of the powder. Pretreatment with alpha-amylase resulted in a lower glass transition temperature and a decrease in particle size. Overall, results show that good quality sweetpotato powders can be produced using this drying method, with potential applications in food and nutraceutical products.

Keywords: alpha-amylase, glass transition, maltodextrin, spray-drying, sweetpotatoes

Introduction

Sweetpotatoes are highly nutritious vegetables rich in calories and biologically active phytochemicals such as β -carotene, polyphenols, ascorbic acid, and dietary fiber (Woolfe 1992). However, sweetpotato consumption has been on the decline in industrialized nations like the United States (FAO 2005). Limited choices of sweetpotato products for consumers beyond the raw storage root, and difficulties in availability, storage, and handling for food processors, contribute to consumption decline (Kays 1985). An approach intended to increase consumption of sweetpotatoes is to convert sweetpotato puree into dried powder to be used as a functional ingredient in food systems. Dried fruit and vegetable powders have been used to enhance color, flavor, water-binding capacity, and nutritional benefits of various food products (Francis and Phelps 2003).

Fruit and vegetable powders such as tomato, blackcurrant, apricot, raspberry, and pineapple have been produced using spray-drying (Bhandari and others 1993; Abadio and others 2004; Goula and others 2004). However, this drying technique has not yet been applied to starchy materials such as sweetpotatoes. Sweetpotato puree has been dried commercially by drum-drying into dark brown,

compact flakes with poor solubility. Spray-drying offers several advantages for dried sweetpotatoes. The resulting powder is of higher quality, can be easily dispersed in water, and readily incorporated into food products (Masters 1991).

Puree viscosity was anticipated to be a challenge in the spray-drying of sweetpotato puree. Thick, sticky puree can be difficult to pump and atomize in a spray-drying operation. Previous authors have reported the reduction of sweetpotato puree viscosity by elevated temperature, the addition of water, and the action of alpha-amylase in order to aid process control and produce consistent sweetpotato puree independent from seasonal and storage variations (Szyperki and others 1986; Kyereme and others 1999). Thus, employing these techniques may increase workability of the puree in the spray-drying process. These viscosity reduction techniques would undoubtedly influence dried product characteristics such as bulk density and water solubility, as reported in drum-drying of sweetpotato puree subjected to various alpha-amylase treatments (Manlan and others 1985).

Furthermore, previous researchers have demonstrated that spray-drying foods high in sugar has low efficiency due to product sticking to the walls of the drying chamber (Bhandari and others 1997a; Vega and others 2005). Cooked sweetpotatoes contain greater than 22% sugar on a dry weight basis (Truong and others 1986). Food products containing substances with low molecular weights, such as sugars, have very low glass transition temperatures (T_g), so these components can depress the T_g of the entire system. If the temperature of the spray-dried particle is greater than 20 °C above the glass transition temperature of that product, the particle will exhibit sticky behavior (Bhandari and Howes 1999). One method to avoid stickiness is to spray-dry at temperatures lower than the T_g + 20 °C. However, this approach is usually not economically feasible. In order to spray-dry sticky products such as sweetpotato puree,

MS 20050653 Submitted 11/1/2005, Accepted 3/18/2006. Authors Grabowski and Daubert are with Dept. of Food Science, N.C. State Univ., Raleigh, NC 27695-7624. Author Truong is with U.S. Dept. of Agriculture, Agricultural Research Service, and Dept. of Food Science, Box 7624, N.C. State Univ., Raleigh, NC 27695-7624. Direct inquiries to author Truong (E-mail: vtruong@unity.ncsu.edu).

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carriers such as maltodextrin (MD) can be used to facilitate the drying process. The high molecular weight of MD increases the glass transition temperature of the product (Bhandari and Howes 1999; Vega and others 2005).

The material composition and conditions under which products are spray-dried will have an effect on the physical properties of the resulting powder. In addition to moisture content, some of the most important characteristics of spray-dried powders include particle size, bulk density, particle porosity, and rehydration capability. The nature of the feed, including viscosity, solids concentration, temperature, and flow rate; the inlet and outlet drying air temperature; atomization technique; and addition of MD have dramatic effects on characteristics of spray-dried powders (Nath and Satpathy 1998; Masters 1991).

Anticipating issues associated with high viscosity and high sugar content in sweetpotato puree, the objective of this study was to determine the effects of predrying treatments such as alpha-amylase application for puree viscosity reduction and MD as a drying aid, and drying temperature on the physicochemical characteristics of spray-dried sweetpotato powder using response surface methodology (RSM). The addition of MD and amylase was expected to facilitate drying performance and improve product functionality. RSM is a collection of mathematical and statistical techniques that have been successfully used for developing, improving, and optimizing processes (Myers and Montgomery 1995). Most recently, RSM has been used to optimize spray-drying conditions for the extract of a tropical plant (Loh and others 2005) and sugar-rich foods (Truong and others 2005).

Materials and Methods

Materials

Sweetpotato puree from the orange-fleshed Beauregard cultivar with 18% dry matter was manufactured by the Bright Harvest Sweetpotato Co. (Clarksville, Ark., U.S.A.). The frozen puree was shipped in 20-kg bag-in-box containers and stored frozen until use. All enzyme treatments used alpha-amylase from *Aspergillus oryzae* (Fungamyl 800 L, Novozymes, Bagsvaerd, Denmark) to hydrolyze starch molecules. This alpha-amylase has optimum activity at a pH around 5 and at temperatures between 50 °C and 60 °C. From Novozymes specifications, the enzyme activity of Fungamyl is quantified as 1 fungal amylase unit (FAU) per mL. Using the chromogenic starch method with amylopectin azure as a substrate (Walter and Purcell 1973), the enzyme activity was measured at 22.9 amylase units/mL. MD with a dextrose equivalent (DE) of 11 (MD 01960) was obtained from Cargill Inc. (Cedar Rapids, Iowa, U.S.A.).

Puree viscosity reduction

The effects of puree temperature, amount of alpha-amylase, and length of enzyme reaction time on puree viscosity were studied in a series of experiments. First, the effect of elevated temperature on puree viscosity was examined at 25 °C, 60 °C, and 90 °C. Puree was then treated with amylase levels of 3.75 and 7.5 mL/kg of puree for 30 min reaction times. For amylase addition, puree was continuously agitated using a mixer with a 3-blade impeller and heated using a water-jacketed container on a hot plate. When the mixture reached 60 °C, the enzymes were added. The puree was held between 50 °C and 65 °C while mixing to allow the enzymes to act for the desired length of time. Another water bath system was used to further raise the temperature of the puree to above 90 °C. Puree was held above 90 °C for 5 min to deactivate the enzymes, and samples were taken for viscosity measurements at 25 °C and 60 °C. Viscosity of the treated puree was measured using a stress-controlled rheome-

ter (StressTech, Reologica, Lund, Sweden) equipped with serrated bob and cup geometry. Samples were exposed to 30 s of preshearing at 20 s⁻¹. Apparent viscosity and shear stress were recorded as shear rates were ramped from 1 to 500 s⁻¹. The StressTech temperature controller was used to heat the puree sample to the desired measurement temperature. After heating, temperature was allowed to equilibrate for 2 min before measurements were taken.

Spray-drying of sweetpotato puree

After determining the appropriate predrying conditions, sweetpotato puree was spray-dried under various conditions.

Equipment and drying conditions. Spray-drying experiments were first conducted using a pilot scale dryer (Anhydro Laboratory Spray Dryer S-1, Anhydro Inc., Attleboro Falls, Mass., U.S.A.) equipped with a 2-fluid nozzle for atomization and a mixed-flow air-product pattern. Drying air for this system was heated using electrical coils before moving into the top of the drying chamber in a slightly rotating motion. Feed was metered into the dryer using a peristaltic pump at a flow rates of 2 kg/h and sprayed upward into the dryer. A cyclone air separator/powder recovery system was used, and the dried powder samples were captured from the base of the cyclone.

A slightly larger capacity pilot-scale dryer (Production Minor Spray Dryer, Niro Inc., Columbia, Md., U.S.A.) was then used for the bulk of experimentation. This dryer had a cocurrent flow regime and utilized a rotary atomizer set at 20000 rpm. Drying air was heated using natural gas combustion. Feed was moved into the dryer at the flow rates of 6.6 to 23 kg/h using a progressive cavity pump (Metering Pump, Moyno, Inc., Springfield, Ohio, U.S.A.). Outlet temperature was maintained by adjusting the feed rate of product into the dryer. The dried powder samples were collected by a similar powder recovery system as mentioned above.

During experimentation on both spray dryers, feed temperature (60 °C), solids content (approximately 18%), inlet air temperatures (150 °C, 190 °C, and 220 °C), and outlet temperature (100 °C) were held constant. A steam-jacketed mixer was used to elevate the temperature of the puree. This mixer was also used to blend the puree and the alpha-amylase for the samples requiring enzyme treatment. The amylase was allowed to act for 30 min at approximately 60 °C before the temperature of the puree was raised above 90 °C to deactivate the enzyme. MD as a drying aid was similarly mixed with the puree using this system. In order to maintain a constant feed of solids to the dryer, water was added to the puree-MD mixture. For samples tested with both amylase and MD, the puree was treated with amylase and then the enzymes were inactivated. MD was mixed with the treated puree just prior to spray-drying.

Experimental design and statistical analysis. RSM was used to investigate the optimum combination of predrying treatments and drying conditions on the physicochemical properties of the spray-dried powders. A central composite design with two variables, MD concentration (0%, 10%, and 20%) and inlet air temperature (150 °C, 190 °C, and 210 °C), was used for the experiments with the 2-fluid-nozzle pilot-scale spray dryer. For the rotary atomized spray dryer, a 3-factor, 3-level, face-centered cube design was employed which consisted of 17 experimental runs, including 3 replicates at the center point (Myers and Montgomery 1995). All 17 treatment combinations are shown in Table 1. The independent design variables were MD concentration, amylase level, and inlet drying air temperature, while the response variables included moisture content, solubility, water absorption, color, bulk density, particle size, and glass transition temperature. This design examined differences between the treatments and generated a model for the response surface to predict how the independent variables will influence powder

characteristics. Review of the response surface can also determine whether conditions exist for optimal product functionality.

The response surface regression procedure (RSREG) of the statistical analysis system SAS software release 8.02 (SAS Institute, Inc., Cary, N.C., U.S.A.) was used to analyze the experimental data and obtain regression coefficients. The data were fit to a full second order polynomial equation as follows:

$$\begin{aligned}
 Y = & \beta_0 + \beta_1(\text{MD}) + \beta_2(\text{amylase}) + \beta_3(\text{temp}) \\
 & + \beta_1^2(\text{MD})^2 + \beta_2^2(\text{amylase})^2 + \beta_3^2(\text{temp})^2 \\
 & + \beta_{12}(\text{MD} \times \text{amylase}) + \beta_{13}(\text{MD} \times \text{temp}) \\
 & + \beta_{23}(\text{amylase} \times \text{temp})
 \end{aligned}
 \tag{1}$$

where *Y* is response of each physicochemical property, MD is the level of MD concentration, amylase is the level of amylase treatment, temp is the inlet air temperature, β_0 is intercept; β_1 , β_2 , and β_3 are the regression coefficients for the linear terms; β_1^2 , β_2^2 , and β_3^2 are the quadratic terms; β_{12} , β_{13} , and β_{23} are the interaction terms.

Powder analysis

Color. The color of the puree, dry powder, and reconstituted solutions was measured using a tristimulus colorimeter (Model DP9000, HunterLab, Reston, VA, U.S.A.) and expressed as *L**, *a**, and *b** values. Reconstituted samples were prepared by rehydrating the powder to the same moisture content as the puree. Three measurements were taken on each sample and values for *L**, *a**, and *b** were averaged. Hue angle (*H**) and color change (ΔE) were calculated as $H^* = \arctan(b^*/a^*)$, $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$ (Hutchings 1994). The color change was calculated for the reconstituted samples as compared to the original puree.

Moisture content. Moisture content was determined by the Karl–Fischer titration method using a Karl Fischer 701 Titrino (Metrohm Ltd., Herisau, Switzerland). The Karl–Fischer unit was enclosed in a Plexiglas™ dry box with compressed air pumped in to maintain low humidity. Between 0.25 and 0.30 g of spray-dried powder were added to the system. The extraction time was set at 180 s to enable the sample to fully dissolve. Methanol was used as the solvent, and hydranal composite 5 was the reactant. Each powder replicate was tested in triplicate.

Water solubility and water absorption. Water solubility index (WSI) and water absorption index (WAI) were determined using the

method described by Anderson and others (1969). A small sample of dry powder (2.5 g) was added to 30 mL of water at 30 °C in a 50-mL centrifuge tube, stirred intermittently for 30 min, and then centrifuged for 10 min at 10000 rpm. The supernatant was carefully poured off into a petri dish and oven-dried overnight. The amount of solids in the dried supernatant as a percentage of the total dry solids in the original 2.5 g sample gave an indication of solubility index. WAI was calculated as the weight of the solid pellet remaining after centrifugation divided by the amount of dry sample.

Bulk density. Bulk density was determined by adding 20 g of sweetpotato powder to a 50-mL graduated cylinder and holding the cylinder on a vortex vibrator for 1 min. The bulk density was calculated by dividing mass of the powder by the volume occupied in the cylinder (Goula and Adamopoulos 2004).

Particle size. Particle sizes of the dry sweetpotato powder were quantified with a S3000 laser diffraction particle analyzer equipped with a vibratory sample feeder (Microtrac Inc., Montgomeryville, Pa., U.S.A.). Results of this analysis were examined in several ways, including volume percentage distribution and average particle size. Volume percentage is an indication of the percentage of the particle population at different particle sizes and is typically displayed graphically. The average particle size (μm) was reported as the mean diameter of the volume distribution (MV), mean diameter of the number distribution (MN), mean diameter of the area distribution (MA), and median diameter of the volume distribution (MedV), also known as the 50th percentile. These values were calculated using the following equations where V_i = volume of the individual particle and d_i = diameter of particle.

$$\text{MV} = \frac{\sum V_i d_i}{\sum V_i}
 \tag{2}$$

$$\text{MN} = \frac{\sum (V_i/d_i^2)}{\sum (V_i/d_i^3)}
 \tag{3}$$

$$\text{MA} = \frac{\sum V_i}{\sum (V_i/d_i)}
 \tag{4}$$

MV is the center of gravity of the distribution and is weighted strongly by coarse particles. MA also uses the volume distribution, but represents a particle surface measurement. This average is less weighted

Table 1 – Levels of independent variables in experimental design

Treatment	x_1	x_2	x_3	Maltodextrin concentration (%)	Amylase level (mL/kg)	Inlet temperature (°C)
	Coded levels					
1	1	1	-1	20	7.5	150
2	1	-1	-1	20	0	150
3	-1	1	-1	0	7.5	150
4	-1	-1	-1	0	0	150
5	0	0	-1	10	3.75	150
6	1	1	1	20	7.5	220
7	1	-1	1	20	0	220
8	-1	1	1	0	7.5	220
9	-1	-1	1	0	0	220
10	0	0	1	10	3.75	220
11	0	1	0	10	7.5	190
12	1	0	0	20	3.75	190
13	0	-1	0	10	0	190
14	-1	0	0	0	3.75	190
15	0	0	0	10	3.75	190
16	0	0	0	10	3.75	190
17	0	0	0	10	3.75	190

by coarse particles and, thus, shows a smaller particle size. MN is related to the population of the number particles using volume distribution data and is weighted to smaller particles (Anonymous 2005).

Particle morphology. Surface morphology of the powder was evaluated using scanning electron microscopy. The powder samples were glued on specimen stubs using carbon-conducting tape and coated with gold-palladium using a Hummer 6.2 (Anatech, LTD, Denver, N.C., U.S.A.). Scanning electron micrographs were obtained using a JEOL JSM 5900LV scanning electron microscope (JEOL USA, Peabody, Mass., U.S.A.) at an accelerating voltage of 15 kV and digital images were captured at various magnifications with JEOL Digital Scan Generator software (v 2.00).

Glass transition temperature. A differential scanning calorimeter (7 Series/Unix DSC 7, Perkin Elmer, Norwalk, Conn., U.S.A.) equipped with an intracooler II refrigeration unit and a dry box was used to determine the glass transition temperatures (T_g) of the spray-dried powders. Nitrogen gas was used at 20 mL/min to flush the sample chamber, while nitrogen at 172 kPa was used to flush the dry box. The instrument was calibrated using mercury (melting point, mp = -38.8°C , $\Delta H = 11.47\text{ J/g}$) and indium (mp = 156.6°C , $\Delta H = 28.45\text{ J/g}$). Approximately 5 to 10 mg of sample were prepared in aluminum sample pans. Sweetpotato powders were heated from -70°C to 120°C at a rate of $20^\circ\text{C}/\text{min}$. An empty pan was used as a reference. A baseline was constructed using an empty pan over the same temperature range and scanning rate. Glass transition was analyzed using a Differential Scanning Calometry DSC 7 equipped with the Pyris thermal analysis software version 3.0 (Perkin Elmer Co., Norwalk, Conn., U.S.A.). T_g was taken at the midpoint of the T_g range. Thermograms were examined for onset temperature, end point temperature, and the change of specific heat of the glass transition region. The glass-transition midpoint value was calculated as the average of the onset and end point values and reported as the glass transition temperature (Bhandari and others 1997a).

Table 2 – Sweetpotato puree viscosity at shear rates of 50, 100, and 500 s^{-1} after treatments with elevated temperature, and alpha-amylase

Temperature ($^\circ\text{C}$)	Amylase level (mL/kg puree)	Length of activation (h)	η_{50} (Pa-s)	η_{100} (Pa-s)	η_{500} (Pa-s)
25	—	—	5.10 ± 0.184	2.56 ± 0.042	0.980 ± 0.021
60	—	—	4.55 ± 0.042	2.13 ± 0.035	0.700 ± 0.039
90	—	—	2.59 ± 0.537	1.55 ± 0.276	0.402 ± 0.033
60	3.75	0.5	1.70 ± 0.120	1.03 ± 0.007	0.398 ± 0.018
60	7.5	0.5	1.54 ± 0.028	0.99 ± 0.006	0.334 ± 0.006

Table 3 – Color values of spray-dried sweetpotato powders and reconstituted powder solutions

Treatment	Dry powder				Reconstituted powder solutions				
	L^*	a^*	b^*	Hue ($^\circ$)	L^*	a^*	b^*	Hue ($^\circ$)	ΔE
1	89.60	4.02	22.59	79.92	49.40	7.88	37.76	78.50	7.49
2	89.86	3.66	24.32	81.43	50.14	9.02	37.16	76.20	6.49
3	80.17	8.25	36.62	77.30	50.67	11.88	39.91	73.34	6.49
4	80.93	9.68	38.17	75.77	50.37	15.38	39.45	68.75	7.86
5	86.48	4.76	26.13	79.67	49.82	10.65	38.32	74.48	8.88
6	89.55	4.12	22.46	79.60	49.52	6.96	36.39	79.07	8.10
7	88.79	3.80	24.15	81.07	49.79	7.33	36.42	78.50	8.35
8	77.32	9.68	37.94	75.68	50.10	11.24	37.79	73.34	7.63
9	81.17	9.18	37.57	76.26	49.37	14.50	38.50	69.33	9.49
10	86.67	4.89	27.25	79.82	49.83	8.40	37.92	77.35	11.03
11	86.16	5.79	27.72	78.21	49.99	11.22	38.04	73.34	6.23
12	88.66	3.77	24.39	81.22	49.07	9.34	39.62	76.78	6.26
13	87.94	4.95	28.69	80.22	49.32	11.71	39.84	73.34	4.87
14	81.24	8.00	35.93	77.44	19.76	12.16	38.86	72.77	5.42
15	86.23	5.42	28.17	79.12	49.37	11.49	39.31	73.91	7.93
16	86.86	5.21	27.51	79.28	49.12	11.81	38.72	72.77	7.97
17	87.00	5.03	27.36	79.58	49.59	10.61	38.58	74.48	8.88

Results and Discussion

Viscosity reduction of sweetpotato puree

It is desirable to keep the viscosity of a material to be spray-dried below 0.25 Pa-s at the dryer atomizer (Anonymous 2003). Thus, a combination of elevated temperature and alpha-amylase treatment was used to reduce sweetpotato puree viscosity prior to spray-drying. As expected, viscosity decreased as the temperature increased from 25°C to 90°C (Table 2) which are in agreement with the results of Kyereme and others (1999). However, at 60°C and a shear rate of 500 s^{-1} , the viscosity of the puree was $0.70 \pm 0.04\text{ Pa-s}$. Further, at 90°C (shear rate = 500 s^{-1}), puree viscosity was still above $0.40 \pm 0.03\text{ Pa-s}$.

Thus, additional means were required to reduce puree viscosity to a suitable level for spray-drying. Water addition is not an efficient means of viscosity reduction since extra energy would be required to remove the additional liquid. The viscosity of sweetpotato puree had previously been successfully reduced by the action of alpha-amylase (Ice and others 1980; Szyperki and others 1986). In this study, the puree was treated with 7.5 and 3.75 mL of amylase per kg of puree for 30 min. These treatments reduced the puree viscosity below $0.40 \pm 0.02\text{ Pa-s}$ at 500 s^{-1} (Table 2). Extending the reaction time beyond 30 min did not further reduce the puree viscosity (data not shown). Although this viscosity level was not as low as the initial goal, spray-drying experiments with the 2-fluid-nozzle spray dryer found this viscosity level to be sufficient for the operation. Thus, amylase levels of 3.75 and 7.5 mL/kg puree were tested in further spray-drying experimentation.

Spray-dried sweetpotato powder characteristics

The physicochemical characteristics of dried sweetpotato powder for each treatment are shown in Table 3 and 4. Estimated regression coefficients for physical characteristic responses with coefficient of determination (R^2) are shown in Table 5 and 6.

Color. Color was represented by L^* , a^* , and b^* , where L^* values range from black (0) to white (100), a^* values range from green (–) to red (+), and b^* values range from blue (–) to yellow (+). Hue is described as the color from the rainbow or spectrum of colors. MD had a highly significant effect ($p < 0.001$) on both measured and

calculated (hue, ΔE) color values of the spray-dried powders, while amylase and inlet temperature showed minimal effects on some color values (Table 5). Raw puree is orange in color, a mix of yellow and red. As the MD concentration was increased, the a^* and b^* values decreased, indicating a loss in “yellowness” and “redness.” For

Table 4 – Moisture content, water absorbance index, water solubility index, glass transition temperature, bulk density, and mean particle diameter of spray-dried sweetpotato powder

Treatment	Moisture content (%)	WAI	WSI	T_g (°C)	Bulk density (g/mL)	Mean particle diameter (μm) ^a			
						Volume (MV)	Number (MN)	Area (MA)	50th Percentile (MedV)
1	1.21	136.0	81.8	67.61	0.704	34.81	8.31	17.16	21.41
2	1.15	142.4	83.1	63.95	0.732	36.38	9.8	20.65	27.54
3	2.38	225.3	74.9	55.98	0.591	41.15	8.12	20.33	32.09
4	2.40	277.3	61.7	57.65	0.82	51.48	11.19	31.72	41.28
5	1.85	174.7	57.0	58.38	0.774	38.2	8.02	17.58	23.8
6	1.86	121.3	82.4	43.97	0.707	30.11	9.1	17.8	21.36
7	1.70	132.0	82.5	64.91	0.743	33.98	10	20.25	25.76
8	3.53	186.7	70.8	42.78	0.546	141.5	7.06	28.28	53.48
9	2.50	261.3	66.0	58.77	0.744	45.2	10.59	28.9	39.28
10	2.25	180.0	80.8	62.17	0.707	31.81	8.55	17.65	22.33
11	2.41	170.7	73.3	64.57	0.721	31.04	8.8	17.36	21.63
12	1.74	150.7	86.7	59.89	0.695	38.45	8.59	18.62	23.87
13	1.44	138.7	81.4	63.30	0.749	33.8	8.91	19.75	26.34
14	2.77	229.3	88.4	51.75	0.798	59.49	8.6	25.85	39.41
15	2.40	169.3	80.6	60.21	0.764	32.2	8.43	17.67	23.03
16	2.56	169.3	78.6	63.05	0.698				
17	2.45	193.3	79.2	63.99	0.74				

^aParticle size was only measured on one of the center point replications.

Table 5 – Regression coefficients of predicted models for the response of powder color variables

Coefficient	Dry powder				Reconstituted powder solutions			
	L^*	a^*	b^*	Hue	a^*	b^*	Hue	ΔE
β_0	72.347 ^c	7.8099	18.886	74.844 ^d	17.1311 ^d	41.8868 ^d	67.1401 ^d	5.9365 ^c
Linear								
β_1	0.84569 ^d	–0.45888 ^d	–1.2409 ^d	0.30645 ^c	–0.24630 ^d	–0.07160 ^b	0.31520 ^d	
β_2	–0.23868	–0.2554	–0.59312 ^b	0.21639	–0.23360 ^b	–0.03947	0.30587 ^b	
β_3	0.10475	0.00928	0.20772	0.02654	–0.01663	–0.01409	0.01628	
Quadratic								
β_{11}	–0.01947 ^c	0.01023 ^c	0.02789 ^d	–0.00493				–0.00059
β_{22}	0.01088	0.03615	0.05928 ^a	–0.04322				–0.00829
β_{33}	–0.00031	–0.00002	–0.00055	–0.00008				0.00005
R^2	0.96	0.97	0.99	0.91	0.83	0.42	0.82	0.15
CV	1.12	8.41	2.54	0.883	9.99	2.39	1.85	20.7

^aSignificant at 10%.

^bSignificant at 5%.

^cSignificant at 1%.

^dSignificant at 0.1%.

Table 6 – Regression coefficients of predicted models for the response of moisture, water absorption, glass transition temperature, bulk density, and particle size

Coefficient	Moisture content	WAI	T_g	Bulk density	Mean particle diameter (μm)		
					Number (MN)	Area (MA)	50 Percentile (MedV)
β_0	0.9615	283.13 ^d	69.53 ^d	0.84583 ^d	10.0001 ^d	27.849	42.906
Linear							
β_1	–0.0592 ^d	–4.975 ^d	0.4155 ^b	0.00082	0.00240	–1.3253 ^c	–2.5909 ^c
β_2	0.0587 ^b	–2.979	–0.7387	–0.01384 ^b	–0.24267 ^c	–1.0313	–0.8165
β_3	0.0083 ^c	–0.226	–0.0634	–0.00044	–0.00095	–0.0012	–0.0556
Quadratic							
β_{11}						0.04597 ^b	0.08674 ^b
β_{22}						0.06518	0.07249
β_{33}						0.00004	0.00027
R^2	0.83	0.81	0.45	0.42	0.53	0.88	0.81
CV	12.92	12.1	8.92	7.9	9.23	10.47	18.7

^aSignificant at 10%.

^bSignificant at 5%.

^cSignificant at 1%.

^dSignificant at 0.1%.

example, comparing treatments 3 and 4 without MD to treatments 1 and 2 that had 20% MD (Table 3), a^* values and b^* values were both less in the MD treatments.

The color change due to MD concentration can be best described by the change in hue value. Hue values are affected by MD in both the dry powder and the reconstituted solution (Table 5). Raw puree has a hue value of 67.0°. As MD concentration increases, the hue value of the reconstituted powder solution increases away from the puree value and becomes more “yellow” in overall color (Figure 1; Table 3). If the sweetpotato powder is desired to have color similar to the puree, a lower level of MD should be used. In a study of spray-dried pineapple juice, Abadio and others (2004) reported that the color of all the light yellow powders did not appear to vary at MD concentrations up to 15%. However, Chopda and others (2001) found the color of guava powders spray-dried with MD levels of greater than 40% to be bright white in appearance, irrespective of the color of the feed material. In a practical sense, the color of the sweetpotato powder will become whiter and less orange as the level of MD increases. This must be taken into consideration, depending on the final application of the product.

Moisture. All of the drying conditions reduced the moisture content of the powder to less than 3.5%, which is desirable for spray-dried powders (Table 4). Each independent variable had a treatment effect on moisture content of the finished powder ($0.001 < p < 0.05$; Table 6). Higher inlet drying air temperatures often resulted in decreased moisture content. This trend may be difficult to discern in this experimental design. However, experiments with the 2-fluid-nozzle spray dryer on sweetpotato puree showed a decrease in moisture content as drying temperature increased (data not shown). Generally, the greater the temperature difference between the particle and the drying air, the greater the heat transfer into the particle, and, thus, the greater the evaporation rate. Goula and others (2004) reported similar results for spray-dried tomato paste. Moisture content also decreased with increasing MD concentration (Table 4), similar to the results Abadio and others (2004) reported for spray-dried pineapple juice. Moisture content of the powders increased at higher levels of amylase treatment as water more readily interacts with dextrins created by alpha-amylase action.

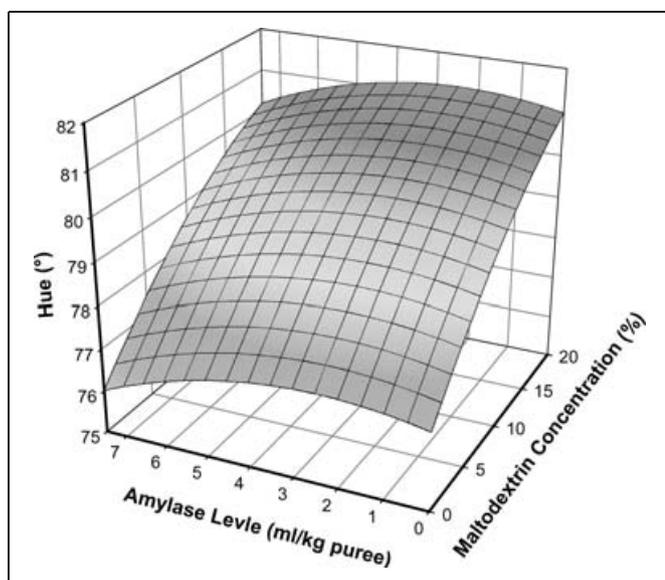


Figure 1 – Response surface of hue values of sweetpotato powder spray-dried at an inlet temperature of 190 °C as a function of maltodextrin concentration and alpha-amylase addition

Solubility and water absorption. The instant properties of a powder involve the ability of a powder to dissolve in water. Most powdered foods are intended for rehydration; therefore, the ideal powder would wet quickly and thoroughly, sink rather than float, and disperse/dissolve without lumps (Hogekamp and Schubert 2003). As discussed in the modeling section below, water solubility could not be modeled by the second order polynomial because none of the regression coefficients was significant (data not shown). However, the addition of MD during spray-drying of sweetpotatoes impacted the solubility of the sweetpotato powders. Solubility index increased by more than 20 units as a result of adding MD (Table 4). Conversely, adding MD reduced the water-holding capacity of the sweetpotato powders (Table 4). These effects of MD can be attributable to the inverse relationship between the MD concentration and the mean diameter of the particles (Table 6). MD can form outer layers on the drops and alter the surface stickiness of particles due to the transformation into glassy state (Adhikari and others 2003). The changes in surface stickiness reduce the particle–particle cohesion and particle–wall adhesion during spray-drying, resulting in less agglomerate formation and, therefore, lower water-holding capacity of the powders. Overall, however, all powders were only able to hold less than 3 times their weight in water, regardless of treatment.

Particle size, bulk density, and particle morphology. Pretreating the puree with alpha-amylase prior to spray-drying and the addition of MD both had significant effects on particle size and, thus, bulk density. According to the model (Table 6), particle size decreases with increasing amylase treatment. In the experiments on the 2-fluid-nozzle pilot-scale spray dryer, amylase treatment was compared to a control. In that experiment, mean particle diameter of the volume distribution decreased from 95.9 to 58.1 μm with amylase treatment. Similarly, comparing levels of enzyme-treated powders produced on the rotary atomizer dryer (Figure 2), particle size appeared to decrease as more amylase was added. Goula and Adamopoulos (2004) observed that the droplet size of the material increases as feed concentration and viscosity increase and energy for atomization decreases, resulting in larger dried particles. In this study, as alpha-amylase action decreased feed viscosity, and, with atomization energy remaining constant, the atomized droplet size may decrease, thus decreasing final particle size.

Particle size has a tremendous impact on bulk density. Typically, as particle size decreases the bulk density will increase, but this is not apparent in spray-dried sweetpotato powder. Of all the independent variables studied, alpha-amylase treatment had the most

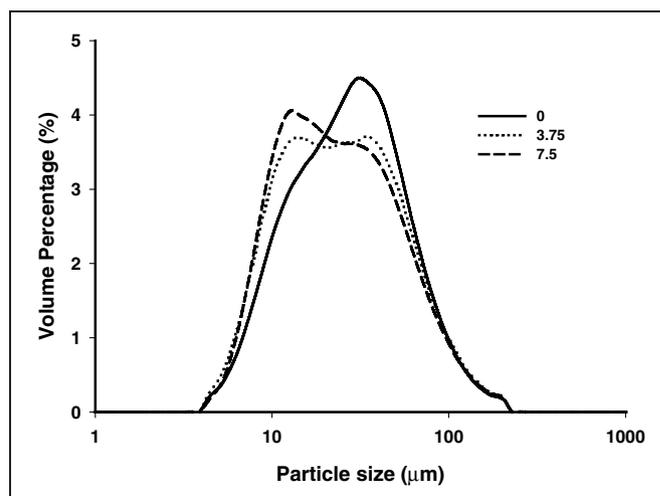


Figure 2 – Particle size distributions for different levels of amylase treatment

impact on bulk density (Table 6). Unfortunately, a low R^2 value does not allow an adequate prediction of bulk density based on the linear model. Bulk density was observed to decrease with amylase treatment (Table 4). This decrease in bulk density does not match the decreasing particle size, but an explanation exists for this behavior. If the dehydration conditions are such that the surface of the particle is not fully solidified or remains sticky and particles collide with each other, then the particles may agglomerate (Goula and Adamopoulos 2004). When observing particle morphology (Figure 3), some of the enzyme-treated powders appeared to aggregate as compared to samples without alpha-amylase addition. Although particles may be small when measured individually, these agglomerates take up a larger volume and, thus, would contribute to a smaller bulk density. These aggregated particles may also aid in the slightly increased water solubility of the powders treated with amylase (Table 4).

Since sweetpotato puree was spray-dried in both a 2-fluid nozzle and a rotary atomizer, comparing the particles produced on each dryer is worthwhile. Generally, products made on rotary atomizers have a smaller particle size than those made on dryers with nozzle atomizers, and such is the case for sweetpotato powders from these experimental dryers (Table 7; Figure 4). Powders from both dryers measure less than 150 μm and, thus, are considered of a fine particle

size. However, the particles from the rotary atomizer are less than half the size of the 2-fluid nozzle.

Glass transition temperature. MD, amylase, and inlet temperature all impacted the glass transition temperature of the sweetpotato powders. Generally, as inlet drying air temperature rose, the glass transition temperature of the powders also increased (Table 4).

Table 7—Summary of particle size analysis for sweetpotato powders spray-dried using a 2-fluid nozzle and a rotary atomizer. Each powder was produced at an inlet drying air temperature of 190 °C using 10% maltodextrin as a drying aid

Atomizer type	Average diameters (μm)			
	Volume ^a	Number ^b	Area ^c	50th percentile ^d
2-Fluid nozzle	102	14.14	69.93	93.58
Rotary atomizer	33.8	8.91	19.75	26.34

^aMean diameter of the volume distribution.

^bMean diameter of the number distribution.

^cMean diameter of the particle surface area distribution.

^dMedian diameter of the volume distribution.

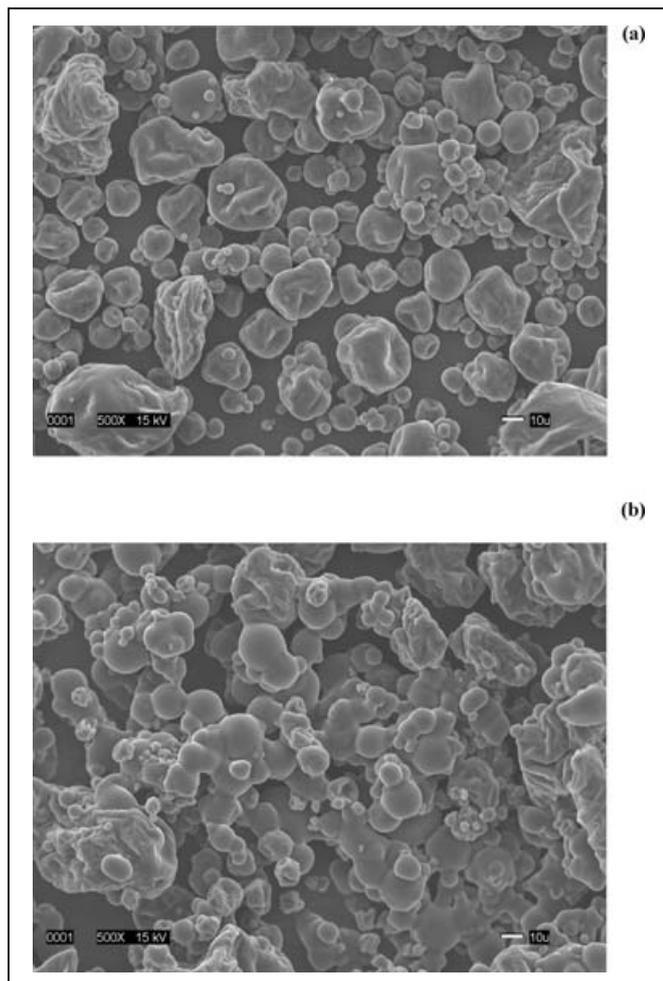


Figure 3—Scanning electron microscope image (500× magnification) of spray-dried sweetpotato powders with and without amylase treatment. (a) No amylase, 10% maltodextrin, 190 °C inlet drying air temperature; (b) 3.75 mL amylase/kg puree, 10% maltodextrin, 190 °C inlet drying air temperature.

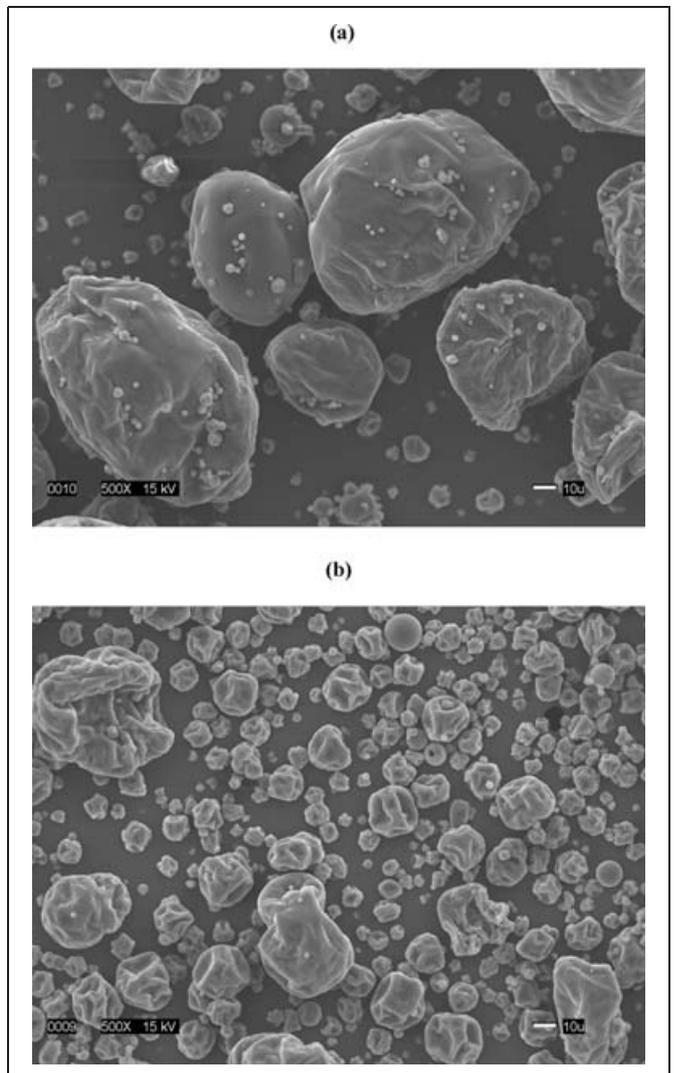


Figure 4—Scanning electron microscope image (500× magnification) of sweetpotato powders spray-dried using a two-fluid nozzle and a rotary atomizer. Each powder was produced at an inlet drying air temperature of 190 °C using 10% maltodextrin as a drying aid. (a) Two-fluid nozzle; (b) Rotary atomizer.

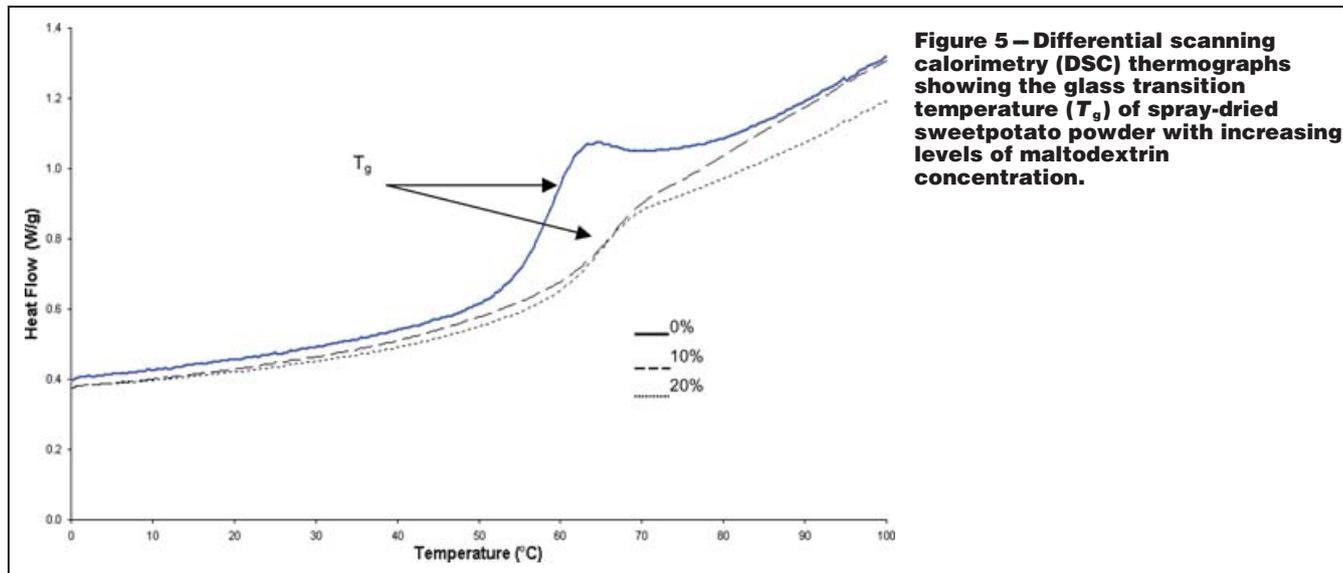


Figure 5 – Differential scanning calorimetry (DSC) thermographs showing the glass transition temperature (T_g) of spray-dried sweetpotato powder with increasing levels of maltodextrin concentration.

This behavior was also observed in the experiments with the 2-fluid-nozzle spray dryer (data not shown). Increases in drying temperature can result in lower residual moisture content, and a decrease in the amount of water in the system limits the ability of water to act as plasticizer and depress the T_g .

As expected, the glass transition temperature of the powders increased as MD was added (Figure 5, Table 4). The positive regression coefficient indicated a linear effect that may increase the response of T_g to MD (Table 6). The increase in T_g is likely caused by an increase in molecular weight of the components in the powder. Bhandari and others (1997b) also reported an increase in glass transition temperature and drying efficiency as the ratio of MD to pineapple solids was increased in the spray-drying of pineapple juice.

Conversely, the glass transition temperature of the powders was reduced as the amount of alpha-amylase allowed to act on the puree was increased (Figure 6). This reduction was expected as the amylase breaks down starch into lower molecular weight dextrans. Lower molecular weight molecules can depress the overall T_g of a food system. Additionally, the amylase-treated powders also had higher moisture content, and, thus, the additional water could also lower the glass transition temperature.

Glass transition temperature was best described by the linear model with MD having a significant impact; however, the R^2 was only 0.42, so T_g cannot be accurately predicted by this model alone.

Correlation models. The results of statistical analysis indicated that the linear and quadratic parameters were significant for many of the powder characteristics. On the other hand, the interaction terms did not produce a significant effect for any of the physicochemical attributes. Moisture content, WAI, a^* , b^* and hue of reconstituted puree, bulk density, MN, and T_g were best fit only using the linear components of the equation. L^* , a^* , b^* , hue, MA, and MedV had both linear and quadratic effects of the independent variables.

Based on these observations, the data were fit to the appropriate linear and/or quadratic model. Estimated regression coefficients for physical characteristics responses with the coefficient of determination (R^2) and coefficient of variation (CV) are shown in Table 5 and 6. None of these models fits the data at a significant level for the solubility index, L^* value of the reconstituted powder, and mean particle diameter of the volume distribution (MV). However, the R^2 values for L^* , a^* , b^* , hue, a^* and hue values of reconstituted puree, moisture, WAI, MA, and MedV were all above 0.80, indicating that the models are sufficiently accurate for prediction purposes. A high

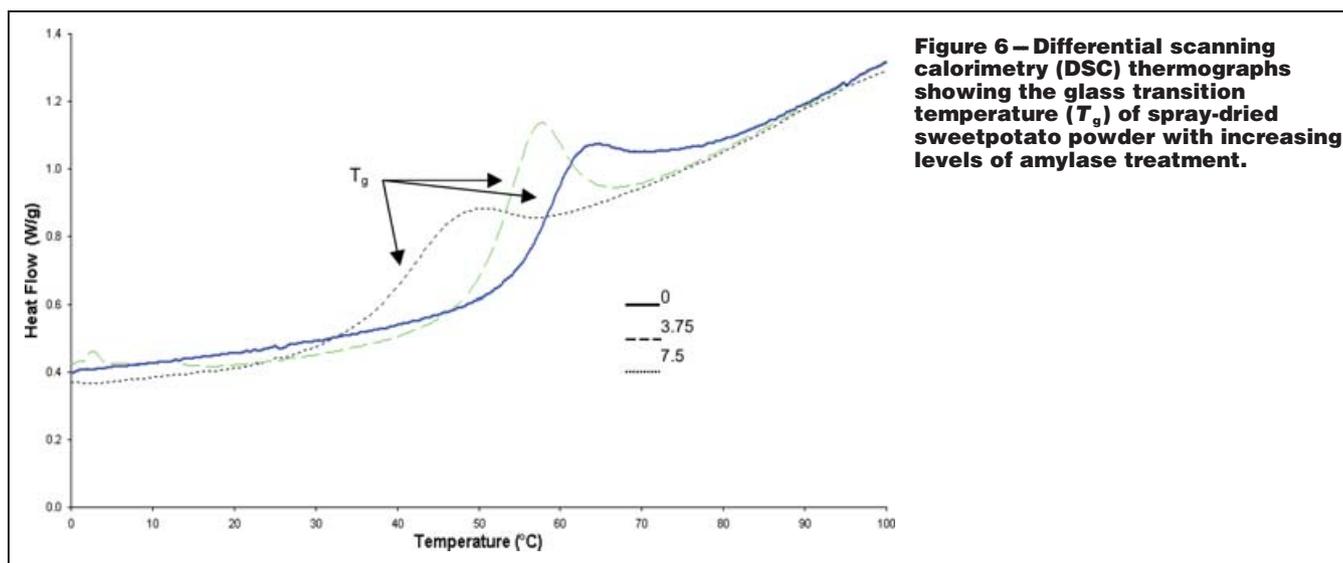


Figure 6 – Differential scanning calorimetry (DSC) thermographs showing the glass transition temperature (T_g) of spray-dried sweetpotato powder with increasing levels of amylase treatment.

proportion of the variability was explained by the mathematical models for these attributes, and, thus, the developed model could be considered accurate (Loh and others 2005). Conversely, T_g , bulk density, MN, reconstituted powder b^* value, and ΔE had some significant terms in the model but the overall model had very low R^2 values.

MD was the most important factor impacting moisture content, WAI, L^* value, a^* value, b^* value, and hue of the powder, and a^* value and hue of the reconstituted solution with high significance ($P < 0.0001$). The effect of MD on particle diameter (MA and MedV) was also highly significant ($P < 0.01$). T_g and b^* values of the reconstituted powder were also impacted ($P < 0.05$) by MD, but solubility index, ΔE , bulk density, and particle diameter (MV and MN) showed no significant effects. For MD, the correlation at the second order was highly significant for L^* value, a^* value ($P < 0.01$), and b^* value ($P < 0.001$) of the powder and significant for particle diameter (MA and MedV) ($P < 0.05$).

Amylase had a highly significant effect on powder hue ($P < 0.001$) and particle diameter (MN; $p < 0.01$). It also had a significant effect ($P < 0.05$) on moisture, powder b^* value, reconstituted a^* value, reconstituted hue value, and bulk density and no effects on the other powder characteristics. Correlation at the second order for amylase was significant ($P < 0.1$) for powder b^* value. Inlet temperature was highly significant for moisture content ($P < 0.01$).

Difficulty exists in determining the optimal finished product characteristics for dried sweetpotato powders since desirable attributes are dependent on the finished product application. For example, for a powdered drink product, high solubility may be important, but, in another product, orange color may be critical, while solubility may not be as important. In order to determine the optimal drying conditions, desired product characteristics would have to be established. Further experimentation and statistical analysis would have to be conducted to determine the best-fit models and the optimum drying conditions. The drying parameters and models used in this study result in a solid starting point for further study. This study also gives insight into the effect of the independent variables on the individual powder attributes and what possibilities are available for product optimization.

Conclusions

A combination of elevated temperature and alpha-amylase treatment was effective in reducing the viscosity of sweetpotato puree for spray-drying. For the amylase-treated puree, MD should also be used as a drying aid to increase the glass transition temperature of the material to reduce stickiness and aid product recovery. The predrying treatments and drying temperature impacted the final characteristics and functionality of the spray-dried sweetpotato powders. Sound RSM models were developed for several attributes such as color values, moisture, WAI, and particle size, while further study is required to accurately develop prediction models for other powder characteristics.

Changing the levels of the independent variables allows for alterations in the final powder characteristics. Optimal product attributes will be determined depending on the desired application of this ingredient. Further experimentation using RSM can be carried out to optimize the sweetpotato powders for a specific application. However, these results indicate that good quality powders can be produced by spray-drying sweetpotato puree

for potential use in dry mixes, soups, beverages, and other food and nutraceutical products. Adoption of this technology could, therefore, open up a new market opportunity for the sweetpotato industry.

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References

- Abadio FDB, Domingues AM, Borges SV, Oliveira VM. 2004. Physical properties of powdered pineapple (*Ananas comsus*) juice—effect of malt dextrin concentration and atomization speed. *J Food Eng* 64:285–7.
- Adhikari B, Howes T, Bhandari BR, Truong V. 2003. Characterization of the surface stickiness of fructose-maltodextrin solutions during drying. *Drying Technol* 21(1):17–20.
- Anderson RA, Conway HF, Pfeifer VF, Griffin Jr EL. 1969. Gelatinization of corn grits by roll and extrusion cooking. *Cer Sci Today* 14(1):4–7, 11–2.
- Anonymous. 2003. APV drying handbook. Tonawanda, NY: APV Solutions and Services.
- Anonymous. 2005. Microtrac S3000 data description. Montgomeryville, Pa: Microtrac, Inc.
- Bhandari BR, Howes T. 1999. Implication of glass transition for the drying and stability of dried foods. *J Food Eng* 40:71–9.
- Bhandari BR, Senoussi A, Dumoulin ED, Lebert A. 1993. Spray drying of concentrated fruit juices. *Drying Technol* 11(5):1081–92.
- Bhandari BR, Datta N, Howes T. 1997a. Problems associated with the spray drying of sugar-rich foods. *Drying Technol* 15(2):671–84.
- Bhandari BR, Datta N, Crooks R, Howes T, Rigby S. 1997b. A semi-empirical approach to optimize the quantity of drying aids required to spray dry sugar-rich foods. *Drying Technol* 15(10):2509–25.
- Chopda CA, Barrett DM. 2001. Optimization of guava juice and powder production. *J Food Proc Pres* 25:411–30.
- [FAO] Food and Agriculture Organization. 2005. FAOSTAT—FAO statistical database. FAO of the United Nations.
- Francis D, Phelps SK. 2003. Fruit and vegetable juice powders add value to cereal products. *Cer Foods World* 48(5):244–6.
- Goula AM, Adamopoulos KG. 2004. Spray drying of tomato pulp: effect of feed concentration. *Drying Technol* 22(10):2309–30.
- Goula AM, Adamopoulos KG, Kazakis NA. 2004. Influence of spray drying conditions on tomato powder properties. *Drying Technol* 22(5):1129–51.
- Hogekamp S, Schubert H. 2003. Rehydration of food powders. *Food Sci Tech Intern* 9(3):223–35.
- Hutchings JB. 1994. Food colouring and appearance. Glasgow, UK: Blackie Academic & Professional. p 199–237.
- Ice JR, Hamann DD, Purcell AE. 1980. Effects of pH, enzymes, and storage time on the rheology of sweetpotato puree. *J Food Sci* 45:1614–8.
- Kays SJ. 1985. Formulated sweetpotato products. In: Bouwkamp JC. Sweetpotato products: a natural resource for the tropics. Boca Raton, FL: CRC Press, Inc. p 205–18.
- Kyereme M, Hale SA, Farkas BE. 1999. Modeling the temperature effect on the flow behavior of sweetpotato puree. *J Food Proc Eng* 22:235–47.
- Loh SK, Che Man YB, Tan CP, Osman A, Hamid NSA. 2005. Process optimization of encapsulated pandan (*Pandanus amaryllifolius*) powder using spray-drying method. *J Sci Food Agric* 85(12):1999–2004.
- Manlan M, Matthews RF, Bates RP, O'Hair SK. 1985. Drum drying of tropical sweetpotatoes. *J Food Sci* 50: 764–8.
- Masters K. 1991. Spray drying handbook. New York: John Wiley & Sons, Inc.
- Myers RH, Montgomery DC. 1995. Response surface methodology: process and product optimization using designed experiments. New York: John Wiley & Sons, Inc.
- Nath S, Satpathy GR. 1998. A systematic approach for investigation of spray drying processes. *Drying Technol* 16(6):1173–93.
- Szyperki RJ, Hamann DD, Walter Jr WM. 1986. Controlled alpha amylase process for improved sweetpotato puree. *J Food Sci* 51(2):360–3, 377.
- Truong V, Bhandari BR, Howes T. 2005. Optimization of cocurrent spray drying process for sugar-rich foods. Part II—optimization of spray drying process based on glass transition concept. *J Food Eng* 71:66–72.
- Truong VD, Biermann CJ, Marlett JA. 1986. Simple sugars, oligosaccharides, and starch concentrations in raw and cooked sweetpotato. *J Agric Food Chem* 34:421–5.
- Vega C, Goff HD, Roos YH. 2005. Spray drying of high-sucrose dairy emulsions: feasibility and physicochemical properties. *J Food Sci* 70(3):244–51.
- Walter Jr WM, Purcell AE. 1973. Alpha-amylase in sweetpotatoes. A comparison between the amyloclastic and chromogenic starch methods of analysis. *J Food Sci* 38:548–9.
- Woolfe J. 1992. Sweetpotato: an untapped food resource. Cambridge, UK: Cambridge Univ. Press.