

ABSTRACT

GRABOWSKI, JULIE ANN. Development and characterization of spray dried sweetpotatoes. (Under the direction of Dr. Van-Den Truong.)

Sweetpotatoes are a nutritious vegetable rich in calories, vitamins, minerals, and biologically active phytochemicals. However, the consumption of sweetpotatoes has been declining. Very few sweetpotato product choices exist for consumers beyond the raw root. Furthermore, special storage and handling requirements for sweetpotatoes have proven to be a challenge to incorporating this healthy vegetable into processed food products.

Therefore, a need exists to develop alternative processing methods to produce a functional and stable form of sweetpotatoes that is readily available for the food processing industry. One strategy is to convert sweetpotato puree into a dried powder to be used as a functional ingredient in different food systems. Spray drying, which has been used for commercial production of several fruit and vegetable powders, has not yet been applied to sweetpotato processing.

Several methods of producing a functional powder in an efficient manner were investigated since spray drying has never been applied to sweetpotato puree. The high viscosity of sweetpotato puree was anticipated to be a challenge in pumping and atomization during the spray drying process. Moreover, the sticky behavior due to high sugar content in sweetpotato puree may affect spray drying efficiency. Many spray dried products make use of a drying aid such as maltodextrin

in order to increase the glass transition temperature and reduce stickiness thus facilitating product recovery. These pre-drying treatments of viscosity reduction and maltodextrin addition, as well as drying temperature, were expected to have effects on the physicochemical characteristics of the final spray dried powder. Thus, model-fitting using response surface methodology was performed to examine the effects of the pre-drying treatments and spray drying conditions on the moisture content, color, water absorption, solubility, particle size, bulk density, and glass transition temperature of the spray dried powder.

Elevated temperature and the action of amylase proved to be effective in reducing puree viscosity. Furthermore, pre-treatment with alpha-amylase resulted in dried powders with a lower glass transition temperature and smaller particle size. The addition of maltodextrin significantly increased powder solubility, altered the hue value, and raised the glass transition temperature of the powder. Sound models were developed for some attributes while further study is needed to accurately develop prediction models for other powder characteristics. Difficulty existed in determining the optimal drying conditions for sweetpotatoes as the best finished product characteristics for dried sweetpotato powders are dependent on the finished product application. However, spray drying sweetpotatoes with a combination of amylase treatment and maltodextrin created a quality powder balancing all functional characteristics.

To better determine potential applications of powders produced with various levels of amylase and maltodextrin, nutrient composition and rheological properties of the powders in solution were examined and compared to sweetpotato puree.

Many of the characteristic sweetpotato nutrients such as beta-carotene and ascorbic acid were severely reduced during the spray drying of sweetpotato puree. Further isomerization of beta-carotene also occurred during dehydration. In addition to thermal degradation of components, the addition of maltodextrin as a drying aid diluted the amount of nutrients in the resulting powder. Rheological characterization of the powders found the viscosity of the reconstituted solutions was much lower than that of the puree at the same solid concentration. However, the reconstituted sweetpotato slurries did behave similarly to pregelatinized starch solutions.

Spray dried sweetpotato powders have been developed to become a functional ingredient in foods by enhancing natural color, flavor, and functionality. Overall, results show that good quality sweetpotato powders can be produced using this drying method. Despite the need for further increased nutrient retention, these powders have the potential to be a good source of beta-carotene and minerals as well as used in food systems as a thickener. As product developers research ways to improve food products, sweetpotato powders could potentially be used in dry mixes, soups, beverages, and other food and nutraceutical products. Furthermore, additional research could optimize the physicochemical characteristics for a specific product application.

**DEVELOPMENT AND CHARACTERIZATION OF
SPRAY DRIED SWEETPOTATOES**

by

JULIE ANN GRABOWSKI

A Thesis submitted to the Graduate Faculty of
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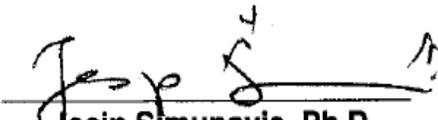
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DEDICATION

For Grandma Dee, who read books to me as a baby.

Your love and laughter will always be with me

BIOGRAPHY

Julie Ann Grabowski, the first daughter of John and Cindy Grabowski was born on July 22, 1976. She grew up in South Holland, IL, a southeast suburb of Chicago with her younger brothers, John and Joey. As a child she spent her days playing with her brothers and the neighborhood kids, swimming in the pool, playing basketball in the driveway, and attending day camp at the local park. Family lore has it that Julie was also known on many occasions to rally the cousins together to put on some kind of performance at holiday gatherings. She enjoyed a dance career in her early life until she discovered sports as her passion. She played basketball and volleyball for the St. Jude Flying J's and 16-inch softball in the summers. After graduating from St. Jude, her love of sports and desire for a fun and different academic experience lead her to Seton Academy, an all-girls Catholic high school. During her high school tenure, she continued to play a sport each season, was involved in student government and the yearbook, and built some great friendships.

After graduating as valedictorian of her high school class in May of 1994, Julie matriculated to the University of Illinois in Urbana-Champaign to pursue a degree in food and bioprocess engineering. At Illinois, in addition to cheering on the Fighting Illini teams, she was very active in the residence halls as hall president, desk clerk, and resident advisor. She also had interesting summer jobs stocking the shelves at Target, packing lunchmeat at Carl Buddig, working for Dr. Litchfield on aseptic processing, and exploring the world of meat casings at Viskase. Leaving Champaign behind for a semester, Julie studied abroad at Curtin University in Perth,

Western Australia where she became friends with people from all over the world and had many great adventures such as scuba diving the great barrier reef, surfing on the Gold Coast, swimming in waterfalls, testing her skills at cricket and “footy”, driving on the other side of the road, and learning to speak Australian.

Agricultural Engineering degree in hand, in 1999 Julie went to work as an operations resource for a Frito-Lay manufacturing facility in Beloit, WI making all kinds of snack chips. Julie grew up quickly during her Frito-Lay experience. Her time in the land of cheese gave her much insight into practical food science, how to manage through disaster with a smile on your face, and the beauty of Illinois-Wisconsin sports rivalries.

Desiring a career change to research and development, and to leave the flatness of the Midwest for a time, Julie decided to pursue a master’s degree in Food Science at the North Carolina State University. She was a founding member of Den Truong’s “Sweetpotato Club” and completed a research project on spray dried sweetpotatoes. During her time at North Carolina State, she also participated in a summer internship program with Kraft Foods, had many escapades and learning experiences as a member and president of the Food Science Club, explored the southeast with trips to the beaches and mountains, and completed her first half marathon. After graduation, she will return to the Chicagoland area to work for Kraft Foods as a product development scientist.

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CHAPTER 1

INTRODUCTION

1.1 - INTRODUCTION

Sweetpotatoes are nutritious vegetables rich in calories and biologically active phytochemicals. These root crops contain approximately 20-30 % dry matter of which the majority is comprised of starch. Sugar and dietary fiber also make up a considerable portion of the dry matter while protein and lipids are present in a lesser quantity. Orange fleshed sweetpotatoes are often recognized for containing a significant amount of beta-carotene, a pre-cursor to vitamin A. Sweetpotatoes are also a substantial source of vitamin C and several minerals (Woolfe 1992).

However, the consumption of sweetpotatoes has declined in the past several decades. This decline is attributed to difficulties in availability, storage, and handling for food processors as well as limited choices of sweetpotato products for consumers beyond the raw root (Collins & Walter 1992). During this same time period however, consumption of the white potato has increased. Part of this increase is due to the market success of processed products such as chips, fries, and frozen snacks (Kays 1985). Processed sweetpotato products have not met the same success as their white potato counterparts. The recent interest in healthier food choices provides an opportunity to increase consumption of sweetpotatoes. However, special storage and handling requirements for sweetpotatoes continue to be impediments to the use of sweetpotatoes in many processed food products. Therefore, new and alternative products and processing strategies must be developed in order to produce a functional and stable form of sweetpotatoes that is attractive to consumers and product developers. One approach intended to increase sweetpotato consumption is to convert sweetpotato puree into a dried

powder to be used as functional ingredient in different food systems. If a substantial market can be established for this ingredient form, the amount of sweetpotato roots left in the field due to improper size or shape may be reduced thus aiding sweetpotato farmers.

Sweetpotatoes have been drum dried for many years. However, these sweetpotato flakes have an undesirable brown color and limited functionality (Collins & Walter 1992). Many fruits and vegetables such as tomatoes, blackcurrant, apricot, raspberry, and pineapple, have been successfully spray dried; however, this drying technology has not yet been applied to sweetpotatoes. (Goula et al. 2004, Bhandari et al. 1993). These dried fruit and vegetable powders have been shown to contribute several nutritional and functional benefits to food products such as color, flavor, water binding, and additional nutrients (Francis & Phelps 2003).

Spray drying has never been applied to sweetpotato puree, thus all methods of producing a functional powder in an efficient manner should be investigated. The high viscosity of sweetpotato puree is anticipated to be a challenge in pumping and atomization during the spray drying process. Moreover, the sticky behavior due to high sugar content in sweetpotato puree may affect spray drying efficiency. Therefore, pre-drying treatments such as puree viscosity reduction and modification of the glass transition temperature using suitable drying aids need to be investigated to demonstrate the feasibility of this processing technology for sweetpotatoes.

The viscosity reduction method, use of drying aid, and drying conditions can all influence finished product characteristics. In order for product development scientists and consumers to fully utilize this new form of dried sweetpotatoes in a

variety of applications, nutritional and rheological information about the spray dried powders is needed in addition to the physicochemical characteristics. The chemical composition of sweetpotatoes can be altered during thermal processing. For example, the action of amylases converts starch to sugars and dextrans (Walter et al. 1975). Additionally, heat and oxidation can degrade beta-carotene and ascorbic acid (Chandler & Schwartz 1988, Bradbury & Singh 1986). One of the applications of other dried fruit and vegetable powders is to supplement the nutritional profile and create a healthful image so it must be determined if sweetpotato powders can also provide these benefits.

Furthermore, many dried powder ingredients are reconstituted in water in the food system. Thus, knowledge of the rheological performance of reconstituted powders as compared to the original puree is also beneficial in order to understand product functionality and find applications of the spray dried sweet potato powder as a functional ingredient in products such as dry mixes, soups, and beverages. Thus, the objectives of this study were:

1. Determine suitable pre-drying treatments to reduce sweetpotato puree viscosity to a level appropriate for spray drying.
2. Determine the effects of pre-drying treatments and drying conditions on the physicochemical properties of spray dried sweetpotato powder.
3. Characterize the nutrient composition and rheological properties of sweetpotato powders spray dried under different conditions as compared to sweetpotato puree.

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CHAPTER 2

LITERATURE REVIEW

2.1 - SWEETPOTATOES

2.1.1 – Production

Sweetpotatoes are a highly nutritious vegetable produced worldwide. The sweetpotato (*Ipomoea batatas*), considered a native of tropical America, is a perennial plant with trailing vines, prostrate or slender stems, leaves of variable size and shape, and blossoms that resemble those of the morning glory. Each plant develops 4 to 10 fleshy storage roots where excess energy is stored in the form of carbohydrates. This storage organ is a true root and not a tuber, or modified stem, as it often mistakenly referred (Kotecha & Kadam 1998, Rubatzky 1997, Woolfe 1992). Flesh root colors range from white to light yellow, dark orange, red, or purple. Various types of cultivars are popular in different regions of the world and breeding programs are currently working to select for certain desirable flesh root and yield traits in those regions (Kotecha & Kadam 1998).

Growth conditions have a tremendous impact on the flesh root development. Sweetpotatoes thrive in the warm, humid weather of the tropics but can also be grown in temperate climates during the frost free periods. Optimum growing temperature is between 20 and 30°C as sweetpotatoes are susceptible to chill injury below 10°C. Sandy or sandy loam soils are best for root shape and harvesting ease as well as balancing vegetative and flesh root growth. Sweetpotatoes require moderate rainfall which ideally occurs during the early stages of growth. In addition to temperature, soil, and rainfall, exposure to sunlight also impacts flesh root development. Long days will increase vine growth and decrease root yield, whereas

short days promote flesh root growth. The total growth period ranges from 90 to 150 days in temperate climates while in tropical areas sweetpotatoes are grown continuously until adequate root size is obtained. (Kotecha & Kadam 1998, Rubatzky 1997).

According to the Food and Agriculture Organization (FAO) Statistical Database over 127, 532,000 metric tons (MT) of sweetpotatoes were produced worldwide in 2004 (FAO 2005). Despite the American origin of the sweetpotato, today the majority of sweetpotatoes are grown in Asia and Africa. China is the leading producer worldwide with 106,197,100 MT in 2004. From 2002 to 2004, U.S. production estimates ranged from 580,000 to 720,000 MT (NASS 2004, FAO 2005). Sweetpotatoes are primarily grown in the southeastern United States where North Carolina is the largest producer. North Carolina generated approximately 40% of the US crop with 218,000 MT in 2002 (NASS 2004). However, overall production and consumption of sweetpotatoes in industrialized nations has been on the decline for the past 30 years. This decline is attributed to limited choices of sweetpotato products for consumers beyond the raw root as well as difficulties in availability, storage, and handling for food processors (Collins & Walter 1992).

2.1.2 – Chemical Composition of Roots

Dry Matter

The chemical composition of sweetpotato roots varies widely depending on the cultivar, growing conditions, maturity, and storage duration. Overall, sweetpotatoes have a high moisture level with an average dry matter content range

of 25 to 30%. Woolfe (1992) summarized dry matter content to be 13.6% to 35.1% in sweetpotatoes from Taiwan and 22.9 to 48.2% in Brazilian sweetpotatoes. Purcell et al. (1989) referred to studies on North Carolina sweetpotatoes where the solids content ranged from 17.9 to 49.3%. The composition of the dry matter is given in Table 1.

Carbohydrates

Most of the dry matter in sweetpotatoes consists of carbohydrates, primarily starch and sugars and to a lesser extent pectins, cellulose, and hemicellulose. Starch comprises 60 to 70% of the total dry matter, but the values vary for different types of cultivars. As with all starches, sweetpotato starch granules are made up of amylose and amylopectin. Amylose is a linear, unbranched molecule comprised of glucose units joined by alpha- 1, 4 linkages. Amylopectin is a larger, highly branched molecule made of alpha- 1, 4 linked glucose units and branched at the alpha- 1, 6 linkage (Woolfe 1992). Sweetpotato starch contains 79 to 83% amylopectin (Purcell et al. 1989).

Sugars are the next category of carbohydrate found in sweetpotatoes and much variability exists between sweetpotato samples. Using HPLC analysis, Truong et al. (1986) found total sugars to vary from 5.6% in a Filipino cultivar to 38% in a Louisiana cultivar on a dry weight basis (db). Sucrose, glucose, and fructose made up the majority of the total sugars while maltose was present to a lesser extent. Woolfe (1992) reported values between 2.9 to 5.5% on a fresh weight basis (fwb) for American cultivars and 0.38 to 5.64% for cultivars from the South Pacific.

Dietary Fiber

Dietary fiber is considered an important nutritional aspect of sweetpotatoes. Components of dietary fiber include soluble, non-starch polysaccharides and insoluble plant cell wall materials such as pectin, cellulose, hemicellulose, and lignin. Comparing values for dietary fiber is difficult as the amount of fiber is often defined by the method of measurement as well as the components that are included in the analysis (BeMiller & Whistler 1996). This difficulty also extends to sweetpotato research. Some researchers have included lignin – a non-carbohydrate polymer – and others have not. Research completed on South Pacific sweetpotato cultivars found dietary fiber to range from 0.49 to 4.71% with a mean of 1.64% on a fresh weight basis. Researchers in Tonga found their sweetpotatoes to contain 12.6% total dietary fiber and 1.4% lignin on a dry weight basis or 4.0% and 0.4% on a fresh weight basis. Raw sweetpotatoes from Papua New Guinea and the Solomon Islands were reported to have a total dietary fiber of 1.20 to 2.62% (fwb) while cooked American cultivars had mean fiber content of 3.6% (fwb) (Woolfe 1992). A review by Purcell et al. (1989) reported dietary fiber ranges from 2.5 to 5% (db). Most recently, a study of 18 Hawaiian cultivars of varying flesh colors listed total fiber values from 2.01 to 3.87% (fwb) (Huang et al. 1999).

Carotenoids

Carotenoid pigments are recognized in sweetpotatoes for both color and nutrition. These pigments impart cream, yellow, orange, and deep orange color in sweetpotato roots (Woolfe 1992). Kays (1992) reported the total carotene content to range from 0.5 to 44.6 mg/100g (db) while Lessin et al. (1997) reported an average

of 25.6mg/100g (db) of carotene in sweetpotatoes. Orange-fleshed sweetpotatoes such as the Centennial and Goldrush cultivars had a total carotene content of 13mg/100g (fwb) or 45mg/100g (db) (Purcell & Walter 1968). Further, total carotene values of 60 to 72mg/100g (db) were reported for various cultivars with specific values of 5.6 to 32mg/100g (db) for the Julian variety (Purcell et al. 1989).

Although beta-carotene is the most dominant, several different carotenoids are found in sweetpotatoes. Of the total carotene content, beta-carotene was responsible for 89.9% in the Goldrush variety and 86.4% in the Centennial variety. Goldrush contained 0.7% gamma-carotene and Centennial had 0.9% alpha-carotene and 0.8% gamma-carotene among other carotenenes (Purcell et al. 1989). For orange flesh sweetpotatoes in Hawaii, beta-carotene content ranged between 6.7 to 13.1 mg/100g (db) and alpha-carotene content was between 0.45 and 6.76 mg/100g (db) (Huang et al. 1999). Simonne et al. (1993) reported beta-carotene values between 0.1 and 19 mg/100g (db).

Carotenoids have nutritional implications because of their provitamin A activity. Carotenoids are produced by plants and transformed into vitamin A in the human body. The basic structure of carotenoids is comprised of a backbone of isoprene units covalently linked head to tail or tail to tail creating a symmetrical molecule. Different carotenoids are derived from this 40 carbon backbone (von Elbe & Schwartz 1996). To be used for vitamin A activity, the carotenoid must possess one unsubstituted beta-ionone ring with a polyene side chain of at least 11 carbons. The structure of the other end of the molecule may vary. Beta-carotene has a beta-ionone group on each end of the molecule and thus the beta- form has the most

vitamin A activity. Alpha, gamma, and beta-zeacarotene also biologically active carotenoids but only provide half as much activity as beta-carotene (Woolfe 1992).

Vitamins & Minerals

In addition to the provitamin A carotenoids, sweetpotatoes also contain other vitamins such as thiamin (B1), riboflavin (B2), niacin (B6), pantothenic acid (B5), folic acid and some vitamin E. Sweetpotatoes also contain a substantial amount of ascorbic acid or vitamin C. Woolfe (1992) summarized the work of several researchers and several cultivar types and reported the ascorbic acid content in the range of 17 to 35 mg/100g (fwb). Purcell et al. (1989) reported 23.5 and 33.3 mg/100g (fwb) of vitamin C in two different North American cultivars. Ascorbic acid (AA), a carbohydrate-like compound, is a polar, water soluble vitamin. Oxidation converts ascorbic acid to dehydroascorbic acid (DHAA) (Gregory 1996). Both AA and DHAA have vitamin C activity. However, if DHAA is further hydrolyzed to 2,3-diketogulonic acid, then vitamin C activity is lost (Woolfe 1992). Bradbury and Singh (1986) reported values between 9.5 and 25.0 mg/100g (fwb) for AA and 7.3 to 13.6 mg/100g (fwb) for DHA resulting in a total vitamin C activity range of 17.3 to 34.5 mg/100g for the sweetpotato.

The total ash content of sweetpotatoes is approximately 3% of the dry weight or between 0.3% and 1.0% of the fresh weight. This non-volatile inorganic residue contains a variety of minerals and trace elements. Mineral content is affected by growing conditions and the distribution of minerals is not uniform throughout the root. In several studies summarized by Woolfe (1992), potassium was the element with the greatest concentration in sweetpotatoes with an average of 396 mg/100g (fwb).

Phosphorous, calcium, magnesium, and sodium were also present in significant amounts. Other elements present in sweetpotatoes include iron, copper, manganese, zinc, sulfur, and chloride (Woolfe 1992).

Protein

Although mostly recognized for their carbohydrate content, sweetpotatoes also contain protein. Referencing worldwide reports, Purcell et al. (1989) listed values for protein content between 1.73 to 11.8% (db). In a study of a North Carolina root collection, the protein content ranged from 1.73 to 9.14% (db) (Purcell et al. 1972). Sweetpotatoes also contain non-protein nitrogen (NPN). NPN is defined as peptides too small to be coagulated by the reagents that react with true protein. These nitrogen sources may impact protein values determined through Kjeldahl analysis. Additionally, non-protein nitrogen may provide usable nitrogen to the body but little in the form of essential amino acids (Purcell et al. 1989, Woolfe 1992). Sweetpotato protein overall, however, is of good quality and the levels of essential amino acids compare significantly to the FAO reference protein (Purcell et al. 1972, Walter et. al 1984).

Lipids

The lipid content of sweet potatoes is low and slightly variable. Different researchers reported values between 0.1 and 0.8% (fwb) while a survey of American cultivars reported lipid values between 1.21 and 2.55% (db) (Woolfe 1992). These low quantities of lipids make them nutritionally trivial. However, lipids become a component of interest due to their role in off-flavor formation through oxidation in dehydrated sweetpotato flakes. Walter et al. (1971) identified and quantified the

lipids in the Centennial variety. The total lipid content was 2.7% (db) and the lipid fraction was categorized into phospholipids (27.1%), glycolipids (30.8%), and neutral lipids (42.1%).

Enzymes

Sweetpotatoes contain many enzymes but the amylases, alpha and beta, are considered the most significant. Overall the amylolytic enzyme system of sweetpotatoes has been shown to consist mainly of beta-amylase and a smaller amount of alpha-amylase. Ikemiya and Deobald (1966) reported alpha-amylase was found distributed throughout the entire root with a small concentration in the innermost root while the highest concentration of beta-amylase was in the inner tissues. However, Hagenimana et al. (1992) reported different results. In their study on a variety of cultivars, the highest level of alpha-amylase was in the outer root tissue and beta-amylase activity was well distributed throughout the root tissue. Beta-amylase activity remained dominant compared to alpha-amylase in the Hagenimana study.

Alpha-amylase randomly cleaves alpha- 1, 4 linkages in both the amylose and amylopectin molecules of starch to form dextrans. The dextrans from amylose can further be hydrolyzed to maltose. With amylopectin, alpha-amylase action results in the formation of maltose and 'limit dextrans' due to its limited ability to cleave alpha- 1, 6 linkages. From the non-reducing end, beta-amylase cleaves the starch molecule alternately at the alpha-1, 4 linkage resulting in the formation of maltose. The inability of beta-amylase to hydrolyze 1,6 linkages also results in the formation of limit dextrans (Woolfe 1992, BeMiller & Whistler 1996).

During the slow cooking of sweetpotatoes from 60 to 100°C, starch is converted to maltose and dextrans. This conversion is attributed to the amylolytic enzymes present in sweetpotatoes. Beta-amylase was the first amylolytic enzyme isolated in sweetpotatoes and was primarily attributed with maltose production during cooking (Balls 1948). Later, Ikemiya and Deobald (1966) characterized the activity of alpha-amylase in sweetpotatoes. The alpha-amylase found in sweetpotatoes has several unique characteristics such as high optimal activity temperature (70-75°C), low activity at ordinary temperatures (30°C), and significant thermal stability. Additionally, alpha-amylase activity increases as length of storage increases. Sweetpotato alpha-amylase is stable through a wide range of pH at low temperatures (30-45°C) but, when temperature is increased, the optimum pH range decreases. This enzyme can be inactivated by increasing the temperature on either side of the pH optimal range or by increasing the length of heating time (Ikemiya & Deobald 1966).

2.1.3 – Sweetpotato Processing

As previously mentioned, the sweetpotato is a highly nutritious vegetable, yet sweetpotato consumption has progressively declined. Part of this decline may be attributed to the lack of uses for sweetpotatoes for consumers beyond the raw roots. Conversely, the white potato has seen a great increase in production and a large portion of this increase is due to processed products such as frozen products and chips. Thus, one way to expand sweetpotato production is to develop appealing processed products (Kays 1985, Collins & Walter 1992). The National Sweetpotato

Collaborators Group has been researching new and improved sweetpotato products for over 50 years. This group has developed canned products, frozen patties, purees, dehydrated flakes, chips, French fries, a ready-to-eat product, and a fruit-leather. Unfortunately, many of these products have not been successfully marketed (Collins & Walter 1992). Despite these obstacles, research continues to be conducted to further improve and market these products and create new ones to expand sweetpotato consumption. Puree and dehydrated flakes are two products with unique uses, characteristics, and processing techniques and have been significantly investigated by various researchers and marketed as processed sweetpotato products.

Puree

Sweetpotato puree can be used in products such as baby foods and pie fillings and as the basis for frozen patties and dehydrated flakes. Pureeing sweetpotatoes has several advantages. The major advantage to pureeing is that roots of all sizes and shapes can be processed to make acceptable puree and therefore, the entire crop is utilized. Once processed aseptically, puree can be stored at ambient temperatures in bulk containers, thus requiring minimal energy for storage as compared to raw roots. Other advantages include reduced storage space requirements and a year-round supply (Kays 1985). A challenge in puree processing is difficulty in adjusting the process to account for differences in cultivar types; root handling, curing, and storage; and other parameters in order to create a consistent product every time.

Over the years, techniques have developed for puree processing in order to meet this challenge. The first sweetpotato purees were simply made by cooking the roots and then pureeing; however, the aforementioned challenges became an issue. The second technique added alpha- and beta-amylase after cooking and pureeing to obtain the desired amount of starch conversion – this, however, introduced a food additive to the process. The third method employs the “enzyme activation technique” using the native amylolytic enzymes and is now widely used in the food industry as described below (Kays 1985).

For puree production, roots are first washed using a drum washer. Next, roots are peeled using high pressure steam or hot lye solution. Roots may be rewashed after peeling and then inspected and trimmed to remove defects. The roots are pureed by using blades to force the puree through a screen. Next, steam injection is used to increase the temperature to between 74 and 85°C which gelatinizes the starch and activates the amylases. Starch hydrolysis is the most important step in the puree process. The alpha and beta amylases hydrolyze the starch producing maltose and dextrans. The majority of maltose production is completed in the first 10 minutes of the conversion process. However, further decreases in the molecular size of starch and dextrans occur for up to 60 minutes. In order to control the process to produce a consistent product, the length of conversion time is varied from 2 to 60 minutes depending on the root. After starch conversion, the temperature of the puree is raised to between 88 and 100°C in a heat exchanger to inactivate the enzymes. The puree is then packaged in sterilized containers and stored frozen (Kays 1985, Deobald et al. 1968).

Other methods of puree production have been investigated in order to gain additional process control and consistent product quality. Szyperski et al. (1986) developed a pureeing technique which included thermal gelatinization of starch, enzyme hydrolysis of a portion of the puree, and the subsequent blending of the enzyme treated portion with the untreated part. Walter and Schwartz (1993) addressed the issue of batch processing of sweetpotato puree by developing a pre-cook/finish cook method. This method, which could be converted to a continuous process to improve efficiency, produced puree acceptable to consumers.

Flakes

Sweetpotato puree is used as the basis for dehydrated sweetpotato flakes. After the pureeing process, the puree is fed into a double drum dryer. The revolving drums are heated with steam and the puree forms a thin film coating on the drum surface. The film thickness is determined by the space between the drums, the drum velocity, and puree viscosity. The residence time on the drum is short since heat and mass transfer occur quite rapidly. Moisture content is subsequently reduced to 2 to 3 %. After drying, the film sheet is scraped from the dryer and ground into flakes. Sweetpotato flakes are then stored in containers with good moisture and oxygen barrier properties that are purged with gas to remove oxygen before closing (Kays 1985, Heldman & Hartel 1997).

Sweetpotato flakes can be used for mashed potatoes, pies, and other products. Dehydrated flakes present several advantages such as a decrease in the weight of the product, the ability to be stored at ambient temperature, and a convenient product able to be reconstituted and prepared. However, the flakes do

have some limitations such as an undesirable dark brown color (Kays 1985). Dehydrated sweetpotato flakes also undergo rapid oxidative degradation which leads to the development of off flavors and loss of provitamin A activity. Deterioration can only be prevented by storing in a nitrogen atmosphere (Walter & Purcell 1974).

2.1.4 – Changes in sweetpotato components due to thermal treatments

Carbohydrates

Heat processing greatly affects the composition of sweetpotatoes. Perhaps the greatest change due to thermal treatments is the amyolytic conversion of starch to maltose and dextrans. The sequence of events that occurs during baking and other cooking methods involves starch gelatinization, enzymatic hydrolysis, and inactivation of amylases. Specifically, sweetpotato starch gelatinizes between 70 and 75°C. At about 77°C, alpha-amylase breaks down the starch molecules and beta-amylase further hydrolyzes those fragments to maltose. As the temperature continues to rise, beta-amylase will be inactivated. Alpha-amylase will continue to degrade the starch into dextrans until the temperature is above 95°C and alpha-amylase is inactivated. The exact changes that take place during heating depend on cultivar, cooking method, and quantity of amylases (Woolfe 1992).

Several studies have reported an increase in maltose concentration as a result of baking and cooking. Hoover and Harmon (1967) found maltose is the only sugar produced and that the majority of maltose was produced in the first 10 minutes of cooking at temperatures of 70 to 80°C. Deobald et al. (1969) examined the levels

of sugars at several stages during puree production and cooking. No appreciable change existed in glucose and sucrose contents throughout the process while maltose levels increased from 9.6% to 40.3% (db). Walter et al. (1975) examined the effects of the amylolytic enzymes on the carbohydrate changes in baked sweetpotatoes and found 14.6% (fwb) of maltose was formed in freshly harvested roots of the Centennial variety and 14.2% for the Jewel variety during baking. In a study on sugars and starch in sweetpotatoes from the Philippines and the United States, Truong et al. (1986) reported that the maltose content increased during steam cooking for 30 minutes while the amount of starch decreased. Levels of maltose in the uncooked cultivars ranged from 1.6 to 8.1 mg/g (db) and increased to 21.6 to 118 mg/g (db) after heating. Further, starch levels decreased from a range of 331-636 mg/g to 317-550 mg/g (db).

Carotenoids

In addition to carbohydrates, other components of the sweetpotato are also affected by heat processing. Carotenoids can transform and degrade to various products during processing and handling due to heat and oxidation. Carotenoids are easily oxidized due to their large number of conjugated double bonds. Oxidation reactions are complex and result in the formation of various epoxides, carbonyls, and alcohols. This degradation also translates into loss of color and provitamin A activity. Carotenoids are easily isomerized by heat, acid, or light. In general, carotenoids exist in an all *trans* configuration. When exposed to heat, the molecule may transform to a *cis* configuration typically at the 9, 13, and 15 carbon positions. The *cis* form reduces provitamin activity but color remains mostly unaffected.

Thermal processing such as canning will lead to isomerization. Extremely high temperature processing will cause fragmentation products and volatile release. Also, dehydration processes may contribute to oxidative degradation due to exposure to oxygen and an increased surface to mass ratio (von Elbe & Schwartz 1996).

For sweetpotatoes, Chandler and Schwartz (1988) studied the changes in beta-carotene and its isomerization products as a result of blanching, canning, dehydrating, and cooking. In general, the *all-trans*-beta-carotene was more prone to isomerization than to degradation during heat treatments. The length and severity of the heat treatment increased beta-carotene loss and isomerization. Blanching, lye peeling, and pureeing actually showed an increase in beta-carotene content but this increase was attributed to enhanced extraction efficiency due to the heat treatment. However, other common sweet potato processing treatments showed significant reductions in beta-carotene content: steam injection – 8.0% loss, canning 19.7% loss, dehydration by drum drying – 20.5% loss, microwaving - 22.7% loss, and baking - 31.4% loss (Chandler & Schwartz 1988). Lessin et al. (1997) quantified beta-carotene isomers after canning sweetpotatoes. The total beta-carotene content increased by 22% from 256.5mg/g (db) in the fresh root to 312.3 mg/g (db) in the canned product which was attributed to increased extraction efficiency. The beta-carotene in the fresh sweet potatoes existed in the *all-trans* form. After canning however, some of the *trans* form was transformed to *9-cis* (25.3 mg/g), *13-cis* (76.6mg/g), and *15-cis* (19.4mg/g) forms while 191mg/g (db) remained in the *trans* configuration.

Ascorbic Acid

Thermal treatments will also reduce the amount of vitamin C in sweetpotatoes. Purcell et al. (1989) reported roots with 24.5 mg/100g (fwb) of vitamin C before canning to contain 7mg/100g after processing and storage. In addition to reduction by the thermal treatment, some vitamin C from these roots may have leached out into the syrup. When sweetpotatoes were baked immediately after harvest, the amount of vitamin C was reduced by 20% while baking after 6 months of storage reduced the amount by 40% (Purcell et al. 1989). Bradbury and Singh (1986) reported baking sweetpotatoes for 30 minutes reduced AA and DHA by 45 to 55%. Boiling for less than 20 minutes decreased levels of AA while DHA initially increased. After 20 minutes, DHA concentration also began to decrease. For dehydrated flakes, Arthur and McLemore (1955) reported a 50 to 70% decrease in vitamin C for flakes drum dried at high temperatures.

Lipids

Lipids contribute to the formation of off flavors in dehydrated sweet potato flakes. The lipids in sweet potatoes consist of highly unsaturated fatty acids and carotenoid pigments making them readily available for autoxidation. Walter and Purcell (1974) reported that the autoxidation of the lipids in dehydrated flakes occurs via a dual mechanism. Some lipids in sweetpotato flakes are bound in a carbohydrate matrix while others are free on the flake surface. Surface and bound lipids both participate in autoxidation but surface lipids are oxidized at a much faster rate and thus are primarily responsible for off-flavor development.

2.2 – SPRAY DRYING

Spray drying is a unique drying process that involves both particle formation and dehydration. Spray drying transforms feed from a fluid state to a dried powder by spraying the fluid into hot air in a chamber. The process can be designed and controlled to produce powders with varying characteristics (Masters 1991). Many food products are commonly produced by spray drying including milk powders, instant coffee, powdered egg products, yeast extracts, cheese powders, and enzymes (Heldman & Hartel 1997).

Spray drying offers the following advantages (Masters 1991).

- When drying conditions are held constant, powder quality and specifications will remain constant.
- Spray drying is a continuous and easy drying operation and is adaptable to full automatic control.
- A wide range of dryers are available to meet final product specifications.
- Any pumpable material can be spray dried and spray drying is especially applicable to heat-sensitive and heat-resistant materials.

The main disadvantages of spray dryers include installation costs, thermal efficiency, waste heat, and exhaust air handling (Barbosa-Canovas & Vega-Mercado 1996).

2.2.1 – Spray Drying Process

Spray drying involves the atomization of the feed into a drying medium which results in moisture evaporation. This process consists of 4 main stages:

- Atomization of feed into spray
- Spray – air contact
- Drying of spray or moisture evaporation
- Separation of dried product from the air

In general, the material is introduced into the drying chamber through an atomizer to increase the surface area of the material (Figure 1). At the same time that the product is introduced, heated air also comes into the dryer. The material in the hot air dries rapidly as it falls through the drying chamber. The powder and the air exit the dryer and are separated in a cyclone. The air is exhausted and the dried powder collected (Masters 1991).

The first stage of spray drying is atomization of the liquid into small droplets between 10 and 500 μ m in size. This spray greatly increases the surface area of the material which allows for rapid drying. The different atomization techniques are based on the type of energy used to create the droplets and include rotary atomizers, pressure nozzles, and two-fluid nozzles. For rotary atomizers, fluid feed is pumped onto a wheel or spinning disk. Centrifugal forces cause the feed to flow outward over the surface and then expel from the end of the disk in a spray of droplets. Droplet sizes from a centrifugal atomizer depend on throughput rate, rotational speed, and diameter of the atomizer disk. With high-pressure nozzles, the feed is pumped at high pressure through a small orifice into the spray dryer.

Pressure energy is converted to kinetic energy and the feed emits from nozzle as a film that disintegrates into a spray. Droplet size is controlled by the pressure in the nozzle. Generally, pressure nozzles produce larger, coarser particles than rotary atomizers. The last type of atomizer is the two-fluid nozzle which utilizes kinetic energy for atomization. Here, the feed and the air travel separately before coming into contact in the nozzle head. High air velocities create frictional forces which cause the liquid to break down into droplets. Two-fluid nozzles can produce both coarse and fine sprays depending on feed viscosity (Masters 1991, Heldman & Hartel 1997, Barbosa-Canovas & Vega-Mercado 1996).

The next stage in spray drying process is the material spray and air interaction. The way in which the feed interacts with the air is an important consideration in the spray drying system as this interaction will determine droplet behavior during drying and final product characteristics (Masters 1991). In general, ambient air is heated by passing through steam or electric heated coils or a natural gas combustion chamber to temperatures between 150 and 300°C. When the air comes into contact with the droplets, the air picks up moisture and loses heat. Outlet air exhausts from the drying chamber between 50 and 100°C (Heldman & Hartel 1997).

Air-spray interaction is determined by the position of the air and the spray entering the drying chamber. Co-current flow is described as the product and the air moving through the dryer in the same direction i.e. both enter from the top and travel downward. This type of system is especially good for heat sensitive products because evaporation occurs quickly. Wet product comes in contact with the hottest

air so product temperature remains low when most of the drying occurs. The air at the bottom of the dryer is much cooler, thus the temperature of the product remains low as drying is completed. Alternately, the spray can come in contact with the air in a counter-current fashion where the air and spray enter at opposite ends of the dryer. Although in this configuration the driest particles end up passing through the hottest air, this system is able to produce good powder for non-heat sensitive products. The last type of dryer design incorporates both co- and counter-current flow in a mixed flow design. This dryer uses a fountain-type flow allowing for coarse powders to be made in small dryer chambers. Similar to counter-current flow however, particles can be subjected to higher temperatures. All of these configurations have advantages and disadvantages depending on the final desired product (Masters 1991).

After the droplets come in contact with the air, evaporation begins to take place. Drying can be described in two stages – the constant rate and falling rate periods. First, a saturated vapor film will form on the droplet surface. Moisture within the droplet is abundant and thus, the moisture content at the surface of the droplet is considered constant. Evaporation will take place at a constant rate as long as there is enough moisture diffusing to the surface. The main mode of heat transfer is convection between the drying air and the droplet. The temperature at the surface of the droplet remains constant and equal to the wet bulb temperature of the air because evaporative cooling offsets heat transfer from the air to the droplet. This allows for rapid drying at relatively low temperatures (Masters 1991).

When the moisture content is too low to maintain saturated conditions at the surface, the critical point is reached and the falling rate period begins. At the critical point, a shell starts to form on the surface of the droplet. Moisture diffusion through this shell now dictates the rate of evaporation. The shell thickness increases over time. At this point, evaporative cooling is insufficient to maintain the wet bulb temperature at the surface and the temperature of the particle gradually increases. At the same time, the drying air will cool. Eventually, the particle temperature will be the same as the drying air temperature and drying will cease. Final product moisture content is 5-10% or lower – reduced from an initial moisture content of 60-80% (Masters 1991, Heldman & Hartel 1997).

After the product is dried, the air and powder are separated. Gravity helps to remove the heavier particles and cyclones are used to separate the air and the finer particles. Product enters the cyclone tangentially. Centrifugal forces separate the particles, which settle to the bottom, from the air, which is exhausted from the top. The product is removed for further processing or packaging. After the cyclone separator, further filtering or scrubbing of the air may take place (Heldman & Hartel 1997). In an open loop system, the air is then exhausted to the atmosphere. This configuration wastes the heat present in the exhausted air. In a closed loop system, the air is reused in the drying operation (Barbosa-Canovas & Vega-Mercado 1996).

2.2.2 – Spray Dried Powder Characteristics

The powders that are formed as a result of spray drying are unique due to both material composition and drying conditions. In addition to moisture content,

some of the most important characteristics of spray dried powders include particle size, bulk density, and rehydration capability. Processing conditions including feed viscosity, solids concentration, temperature, and rate; the inlet and outlet drying air temperature; atomization technique, and flow pattern in the dryer have a dramatic effect on powder characteristics.

Bulk Density and Particle Size

As solids concentration in the feed increases, and consequently the feed viscosity increases, the particle size of the material will increase. Goula and Adamopoulos (2004) attributed an increase in particle size to an increased droplet size as feed viscosity increased for spray dried tomato paste. With increased feed concentration, the bulk density has been reported to both increase and decrease depending on product and drying operation (Masters 1991). Goula and Adamopoulos (2004) found a correlation between an increase in particle size and a decrease in bulk density for spray dried tomato paste. However, also in a study of tomatoes, Banat et al. (2002) found bulk density to increase with increased feed concentration due to the formation of heavy, solid spheres with high density.

Temperature of the feed has a variable effect on bulk density. If an increased feed temperature influences improved atomization to form spherical droplets, the bulk density will increase. However, if the feed is already easily atomized, increased feed temperature can lower bulk density. This variable is much dependent on the characteristics of the product (Masters 1991).

If a feed rate increase results in higher residual moisture content, the powder bulk density will also increase (Masters 1991). Conversely, Banat et al. (2002)

found that particle size could increase and bulk density decrease with increasing feed rate as a result of an increased potential for droplets to collide and coalesce.

Inlet drying air temperature must be carefully chosen for the product being spray dried. Many heat sensitive products such as eggs and milk cannot withstand temperatures greater than 100°C without degrading, but other products, such as coffee, can withstand much higher temperatures (Heldman & Hartel 1997). Masters (1991) reports increased inlet temperature decreases the bulk density for many products. In spray drying of tomato paste, higher air temperatures were associated with lower bulk densities. Higher temperatures generally cause an increase particle size. A greater tendency exists for particles to be hollow when produced at high inlet temperatures (Goula & Adamopoulos 2004). In addition to inlet air temperature, most spray drying systems are controlled by maintaining a set outlet air temperature, which in turn controls the moisture left in the particles. As the residual moisture increases, the bulk density will also increase (Masters 1991).

Rotary atomizers and nozzles also influence final product characteristics. In general, as the amount of energy available for atomization increases (i.e. increased rotational speed, higher pressure, increased air flow rate), smaller droplets will result. If the amount of energy for atomization is held constant but the feed rate or viscosity is increased, larger droplets will result. Rotary atomizers can be controlled to create a range of particle sizes. Typically, however, these atomizers produce fine to medium-coarse particles in the range of 20 to 150µm. As the wheel speed or diameter increases, the particle size will also increase usually resulting in a lower bulk density (Masters 1991, Nath & Satpathy 1998). In a study of the spray drying of

pineapple juice, as the rotational speed of the atomizer increased, the bulk density decreased (Abadio et al. 2004). Nozzle atomizers usually produce coarser particles from 150 to 300 μ m in diameter (Maters 1991).

The particle size, particle form, and temperature to which the product can be subjected are important factors in designing the air-product flow pattern and selecting an atomizer for a spray dryer. For example, for a finely particulated product that requires a low temperature, a co-current flow dryer equipped with an rotary atomizer is appropriate. A mixed flow-nozzle design would be suitable for a non-heat sensitive product that requires coarse particles when the drying chamber size is limited (Masters 1991).

Rehydration Characteristics

In addition to particle size and bulk density, the rehydration properties of dried powders are important. Most powdered foods are intended for rehydration and the ideal powder would wet quickly and thoroughly, sink rather than float, and disperse/dissolve without lumps. These instant properties of a powder involve the ability of a powder to dissolve in water. Rehydration can be divided into 4 steps: 1.) wetting, 2.) sinking, 3.) dispersing, and 4.) dissolving (Hogekamp & Schubert 2003, Barbosa-Canovas & Vega-Mercado 1996). Wettability is the ability of a particle to adsorb water on its surface and is dependent on the amount of available, wettable surface. Sinkability is affected by particle density and is the ability of the powder to sink into the water after wetting. Dispersability is the capacity of the powder to distribute throughout the water without lumping. Solubility is the dissolving rate

which is affected by the rapid swelling of particles or absence of flecks (Barbosa-Canovas & Vega-Mercado 1996).

Commonly, the spray drying process produces powders that are comprised of relatively small particles or unfavorable structures which negatively affect their instant properties. To improve rehydration characteristics, the surface area for wetting should be increased and the particle should have a large porous structure (Hogekamp & Schubert 2003). One technique often used to improve the instant properties of powder is agglomeration of the powder immediately following the spray dryer. During this instantizing process, the surface of the particles are slightly wetted and then fluidized in air in order for the particles to make contact. The transformation of single particles into porous agglomerates changes the physical characteristics of the powder so that rehydration occurs more quickly (Heldman & Hartel 1997).

Particle Morphology

The effects of processing conditions and material composition on particle morphology are also important considerations in examining spray dried materials. In a study of the morphology of several spray dried products including foods, particles were categorized into 3 main morphologies: crystalline, skin forming, and agglomerate. These structures were found to be material specific and dependent on drying conditions. Most of the food products fell into the skin forming category. These particles formed a non-liquid continuous layer, polymeric in appearance. For example, dairy products formed a skin of denatured proteins. Depending on drying conditions, different features such as particle inflation, collapse, shriveling,

exploding, cratering, cracking, vacuolation, hollowing, and solidifying become apparent in the particles (Walton 2000).

Particle skin begins to form during the falling drying rate period. During this time, the structure of the crust that forms begins to play a role in final particle morphology. Depending on the material, the crust can be defined as porous and rigid, less porous and rigid, and non-porous pliable. During the falling rate period, mass transfer becomes less than heat transfer and the droplet begins to heat up. Vaporization may occur within the particle. Particle formation depends on the nature of the solid crust and if the spray drying temperature is above or below the boiling point of the liquid inside in the droplet. If the temperature is above the boiling point, vaporization will occur in the particle increasing pressure. If the crust is porous, vapors will be released. However, if the crust is non-porous the droplet may rupture. If the crust is non-porous and pliable, the potential exists for the particle to inflate when these vapors are produced. When drying at a lower temperature, a thick, compact and irregular crust is more likely to form which subsequently could shrink and collapse. When a droplet with a rigid crust does not reach the boiling point, little change in particle morphology occurs as drying completes. However, particles with pliable crusts have more potential to collapse and shrivel under these conditions. For example, in spray drying of maltodextrin, particles tend to inflate, form crust, and break at higher temperatures. At lower temperatures, water diffusion is slower, allowing more time for structures to deform, shrink, and collapse (Masters 1991, Alamilla-Beltran et al. 2004).

2.2.3 – Stickiness and Glass Transition Temperature in Spray Drying

Spray drying is a dehydration technique applied to many types of food products. These products can be generally categorized into sticky and non-sticky products. For non-sticky products such as skim milk, gums, and proteins, a simple dryer design can be used and the resulting powder is non-hygroscopic and free flowing. On the other hand, sticky products are difficult to spray dry because these materials stick to the walls of the drying chamber and may remain in a syrup form after the drying process. These material properties lead to operational issues, low yields, and caking during storage (Bhandari et al. 1997a). The main food constituents which cause stickiness issues are sugars, organic acids, and fats as seen in products such as fruit juice, vegetables, honey, and amorphous lactose. High hygroscopicity, high solubility, low glass transition temperatures, and low melting point contribute to stickiness (Bhandari et al 2001).

Recently glass transition temperature (T_g) has been used to define stickiness because T_g encompasses the overall behavior of all constituents present in the food. Glass transition occurs in amorphous solids. Solid materials can be divided into crystalline and amorphous solids. Crystalline solids consist of an orderly array of molecules that will melt upon heating. Conversely, amorphous solids are comprised of disorderly, tangled molecules leaving an open and porous structure with more sites available for external interaction. Amorphous solids are in a glass state with a viscosity of 10^{12} to 10^{14} Pa-s. Glass and glass transition only apply to amorphous materials. As the temperature of a glassy solid is increased, structural changes occur. Above the critical temperature, or glass transition temperature, the structure

changes from a glassy state to a rubbery state and the viscosity is reduced to 10^6 - 10^8 Pa-s. In this state the material displays sticky behavior (Bhandari 2001). As the material temperature rises above the glass transition, the physical properties of the material including the heat capacity and free molecular volume will change. Glass transition can be measured using differential scanning calorimetry (DSC) and differential thermal analysis (DTA), which measure changes in heat capacity, and thermal mechanical analysis (TMA) which determines a change in the elastic modulus (Bhandari & Howes 1999). Glass transition is a second order transition occurring over a range of temperature and is reversible. On the other hand, heat causes crystalline solids to melt. Melting is a first order transition involving the latent heat of melting (Bhandari & Howes 1999, Bhandari 2001).

There are various ways to obtain an amorphous structure. For example, if a crystalline solid is melted and the melt is rapidly cooled, an amorphous solid will form because molecules do not have enough time to realign as a crystal. Also, the rapid cooling of liquid solutions or high moisture solids forms an amorphous solid. Finally, during the drying of foods, when moisture is removed rapidly, an amorphous solid results because the rapid process does not allow molecules to align and crystallize. Amorphous solids are not at thermodynamic equilibrium and thus not as stable as the crystalline form (Bhandari 2001, Bhandari & Howes 1999).

The glass transition temperature of amorphous solids is influenced by the molecular weight of the polymers in the material. Low molecular weight polymers (i.e. sucrose) and monomers (i.e. fructose, glucose) have low glass transition temperatures whereas longer chain molecules have higher glass transition

temperatures. For example, the glass transition temperature of fructose is 5°C whereas the glass transition temperature of maltodextrin with a dextrose equivalent of 5 (indicating a slightly hydrolyzed starch) is 188°C. Several studies have validated that glass transition temperature increases with increasing molecular weight. However, T_g can vary within compounds of the same molecular weight due to differences in chemical structure (Bhandari & Howes 1999).

Food systems are composed of many constituents. Carbohydrates will have a great influence on the glass transition temperature. Low molecular weight sugars with low glass transition temperatures will depress the overall T_g of a food. However, water will have the most significant effect in decreasing the glass transition temperature of a food system. The glass transition temperature of water is -135°C (Bhandari & Howes 1999). Thus, water can act as a plasticizer in the food system contributing to increased free volume, decreased viscosity, and glass transition temperature depression (Fennema 1996). Models have been developed to predict the glass transition temperature of multi-component systems so that the effects of carbohydrates and water can be taken into account for different systems (Bhandari & Howes 1999).

As stated earlier, the stickiness of sugar-rich spray dried foods is related to the glass transition temperature. The sticky behavior of an amorphous product is observed at temperatures about 10 to 20°C greater than the glass transition temperature. At these temperatures, a critical viscosity of about 10^7 Pa-s is reached indicating sticky behavior. Figure 2 represents the physical changes of a sugar-rich food material occurring within the spray dryer. The solution is atomized into fine

droplets. As water evaporates, the solutes become more concentrated. As the product passes through the dryer, the physical state changes from liquid solution to syrup to solid form. Depending on the product, the final amorphous product could be a syrup, a sticky powder, or a free-flowing powder.

As the amount of water is reduced during spray drying, the glass transition temperature increases. At the beginning of drying, the particle surface temperature is near the wet bulb temperature of the air. As the particle begins to heat up, the temperature of the surface of the particle will be much greater than the glass transition temperature and the viscosity of the droplets will be lower than the critical viscosity of 10^7 Pa-s. At the end of drying, the temperature of the particle is near the outlet air temperature (Bhandari et al. 1997a). In order to obtain free-flowing powder, the final particle temperature must be lower than 20°C above the glass transition temperature. But, if the product is high in sugar, the glass transition temperature will be low and the particle temperature may be much higher. Thus, the product may remain syrup even at a low moisture level with a viscosity lower than the critical viscosity. If the particle temperature is higher than the glass transition temperature but the material does not remain a syrup, the product may still have a tendency to show adhesion and stickiness (Bhandari et al 1997a, Bhandari & Howes 1999, Vega et al. 2005).

Vega et al. (2005) conducted a study on the spray drying of high-sucrose dairy emulsions or ice cream mixes. As expected, product recovery increased as the amount of sucrose in the mix decreased because the glass transition temperature was higher. The outlet temperature also affected recovery as this

temperature controls final product moisture which in turn affects the T_g . Also, at the end of the drying operation, particle temperature is near the outlet temperature and this temperature must be less than the $T_g + 20^\circ\text{C}$ or stickiness will be an issue. Ideally, then, stickiness could be avoided by spray drying at a temperature so that the particle temperature is not more than 20°C above the glass transition temperature (Bhandari et al. 1997a, Bhandari & Howes 1999, Vega et al. 2005). However, the glass transition temperature of sugar-rich foods is so low that spray drying them in the appropriate temperature range would not be economically feasible.

2.2.4 – Maltodextrin

The most common approach, therefore, to dry these products is to add high molecular weight additives. Typically, maltodextrins are added to help raise the glass transition temperature. Maltodextrin is a starch derivative produced by acid or enzyme hydrolysis. Maltodextrin is usually described by a dextrose equivalent (DE) which gives an indication of the degree of starch hydrolysis. The DE is a measure of the reducing power of the molecule compared to pure dextrose. Therefore, as the DE of a maltodextrin increases, the degree of hydrolysis is greater and the molecular weight is lower. Products with a DE less than 20 are called maltodextrins whereas hydrolysis products with a DE between 20 and 60 are called corn syrup solids. Products with a low DE are non-hydroscopic - hydroscopicity increases as the DE increases. Maltodextrin also has a bland flavor, good solubility, and is often used as a bulking agent (BeMiller & Whistler 1996).

The glass transition of maltodextrins range from 100 to 243°C (pure starch) depending on their dextrose equivalent and thus are often used to raise the glass transition temperature for drying sugar-rich foods. Using a lower DE maltodextrin will require less addition of the drying aid since this maltodextrin will elevate the glass transition temperature of the mixture higher than a maltodextrin with a higher DE (Bhandari et al. 1997a). Bhandari et al. (1997b) found that a correlation existed between the T_g and the spray drying behavior of fruit juices with maltodextrin addition. As the amount of maltodextrin increased, the glass transition temperature increased and thus, product recovery improved. Bhandari et al. (1997b) also developed an empirical approach to optimize the amount of additive required for sugar-rich foods and found this relationship to be suitable for fruit juices and honey.

Maltodextrin has been used as a carrier in the spray drying of fruit and vegetable juices and purees. Bhandari et al. (1993) tested various levels of maltodextrin with a range of dextrose equivalents in the spray drying of blackcurrant, apricot, and raspberry juices. The optimal fruit juice to maltodextrin ratio for favorable product recovery was 65/35 for blackcurrant, 60/40 for apricot, and 55/45 for raspberry. Differences are attributed to the type and nature of sugars in the individual juices. Maltodextrins were also used during the production of spray dried powder from guava juice. The use of maltodextrin prevented excessive “burn-on” during processing (Chopda & Barrett 2001). Abadio et al. (2004) studied the effects of increasing levels of maltodextrin (10, 12.5, 15%) on the physical properties of spray dried pineapple juice powder. Increases in maltodextrin concentration decreased the moisture content, bulk density, and solubility of the powder. In a

study on spray-dried tomato paste, Banat et al. (2002) added from 25 to 50% starch to act as a carrier. Starch addition improved overall drying and resulted in an increase in total solids. Bulk density decreased as starch percentage increased. Lower water content, starch being less dense than tomato powder, and the agglomerating properties of starch to create bigger particles, all contributed to a decrease in bulk density.

In addition to lowering the glass transition temperature, maltodextrin also facilitates drying by encapsulating the particles. Microencapsulation helps with dry handling and the conversion of liquids and sticky solids to free-flowing powders. This process can also protect sensitive food components from loss during storage. Spray drying is one way to produce microcapsules in a continuous processing operation. Flavors, colors, sweeteners, antioxidants, and enzymes are often encapsulated using spray drying. Two main types of microcapsule structure exist based on the way the core material is distributed in the system. In the first type of structure, the core material is concentrated in the center surrounded by a definite and continuous film of the coating material. In the second type, the core is uniformly dispersed through a matrix. Microcapsules produced by spray drying are usually of the matrix type. Before spray drying, a solutions of the matrix material i.e. maltodextrin and the core material are prepared and mixed together. The mixture then goes through the spray drying process and the resulting in the core material within a protective matrix (Re 1998).

Encapsulation with various dextrose equivalent hydrolyzed starches affected the stability of alpha- and beta-carotene in spray-dried carrot powders. Carrot

powder carotenes that were encapsulated in maltodextrin were stable for a longer period of time than those not encapsulated. Further, the greater the amount of maltodextrin used for encapsulation, the lower the rate of carotene degradation (Wagner & Warthesen 1995). The microencapsulation properties of maltodextrin also aided in the retention of ascorbic acid and soluble solids in guava juice powder. Maltodextrin coated the solids and thus provided thermal and oxidative protection (Chopda & Barrett 2001). Maltodextrin also acted as an encapsulating agent in the successful spray drying of anthocyanins from concord grapes, cranberries, and the tropical plant Roselle (Main et al 1978).

2.3 – RHEOLOGICAL CHARACTERIZATION

2.3.1 – Rheology concepts

Rheology is the science of the flow and deformation of matter, or more specifically, the study of how materials respond to stress and strain. Knowledge of the rheological characteristics of different food products is important in many areas of the industry such as the design of equipment and processes for manufacturing, ingredient functionality in product development, quality control, correlation with sensory data, and understanding of the relationship between microstructure and functionality (Steffe 1996).

The flow behavior of materials is often described by the relationship between shear stress (σ) and shear strain rate ($\dot{\gamma}$) otherwise known as viscosity (η). In

Newtonian fluids, the shear rate is directly proportional to the shear stress as described by the viscosity equation (Eq.1):

$$\text{Newtonian viscosity: } \eta = \frac{\sigma}{\dot{\gamma}} \quad (1)$$

However, in non-Newtonian fluids, the viscosity is a function of shear rate. Apparent viscosity (Eq. 2) is dependent on the shear rate at which it is measured (Steffe 1996).

$$\text{Apparent Viscosity: } \eta = \frac{\sigma}{\dot{\gamma}} = \frac{f(\dot{\gamma})}{\dot{\gamma}} \quad (2)$$

Newtonian foods include fruit juice, milk, and vegetable oil. However, most food products are considered to be non-Newtonian fluids and the flow behavior of these foods is often described by empirical models which relate shear stress and shear rate. Common models include the Bingham plastic (Eq. 3), power law (Eq. 4), and Herschel-Bulkley (Eq. 5) along with many others (Steffe 1996).

$$\text{Bingham Plastic} \quad \sigma = \sigma_o + \mu_{pl} \dot{\gamma} \quad (3)$$

$$\text{Power Law} \quad \sigma = K \dot{\gamma}^n \quad (4)$$

$$\text{Herschel Bulkley} \quad \sigma = \sigma_o + K \dot{\gamma}^n \quad (5)$$

The Bingham plastic model is similar to the Newtonian model with some exceptions. First, the coefficient (μ_{pl}) is constant value called the plastic viscosity. Another difference from the Newtonian model is that the Bingham plastic model involves a

yield stress. A yield stress is the threshold level of force needed to initiate flow. The flow behavior of ketchup, margarine, and mayonnaise can be described by the Bingham plastic model. The power law model is a more versatile model where n is the flow behavior index and K is the consistency coefficient. This model fits experimental results over a range of shear rates for a variety of materials. If a viscosity of a material decreases with increasing shear rate, the substance is known as a shear thinning or pseudoplastic material and the flow behavior index is between zero and one. Applesauce, banana puree, and orange juice concentrate are examples of shear thinning foods. If viscosity increases with increasing shear rate, the material is known as shear thickening or dilatent material and n is greater than 1. Corn starch is the best example of a dilatent material. The Herschel-Bulkley model is another general relationship to describe non-Newtonian fluids. The Newtonian, Bingham, and power law models are considered special cases of the Herschel-Bulkley model. The flow behavior of fish paste and raisin paste are best described by this model. Many other models exist to describe stress and strain relationships (Steffe 1996).

In non-Newtonian fluids, viscosity may also be a function of other factors such as temperature, pressure, time, physical/chemical properties, and voltage in addition to shear rate. For Newtonian fluids, the influence of temperature on viscosity is described by an Arrhenius relationship (Eq. 6) involving absolute temperature (T), universal gas constant (R), and activation energy (E_a).

$$\mu = f(T) = Ae^{\left(\frac{E_a}{RT}\right)} \quad (6)$$

If the material is not Newtonian, dependence on shear rate must also be considered. A different equation involving different empirical constants may be used to determine the effects of numerous factors – for example, shear rate ($\dot{\gamma}$), temperature (T) and concentration (C) (Eq. 7) (Steffe 1996).

$$\eta = f(T, \dot{\gamma}, C) = K_{T, \dot{\gamma}, C} \exp\left[\frac{E_a}{RT} + B(C)\right] \dot{\gamma}^{n-1} \quad (7)$$

Other terminology describes material characteristics which are dependent on time. If viscosity decreases over time at a fixed rate of shear, the material is described as thixotropic. If the material thickens over time at a constant shear rate, this substance is showing rheopectic, or anti-thixotropic behavior. After manufacturing, some foods such as baby food or yogurt will develop internal structures or gels. When exposed to constant shearing, the shear stress of these thixotropic materials will decrease over time (Steffe 1996).

2.3.2 – Rheological characteristics of fruit and vegetable puree

The rheological behavior of both fruit and vegetable purees has been studied by various researchers. Recently, Krokida et al. (2001) compiled data of several fruit and vegetable products and listed values for consistency coefficient and flow behavior index along with the corresponding ranges of temperature and concentration. Almost all fruit and vegetable purees display shear thinning behavior and most have a yield stress. Temperature and concentration are also shown to have an effect on the viscosity of fruit and vegetable purees. The shear thinning flow behavior of peach, papaya, and mango purees was best modeled by the

Herschel-Bulkley equation. Increasing the solids concentration in these purees increased the consistency coefficient while temperature and pH had little effect on the flow parameters (Guerrero & Alzamora 1998). Banana purees were also found to be pseudoplastic with appreciable yield stress best represented by the Herschel-Bulkley model (Guerrero & Alzamora 1997). As expected, the apparent viscosity of banana puree decreased with increasing temperatures. Further, differences in stress values for increasing and decreasing shear rate sweeps suggested a time dependency for banana puree (Ditchfield et al. 2004).

Often fruit purees are treated with enzymes to reduce their viscosity for processing. The apparent viscosity of papaya puree, a pseudoplastic material with a yield stress, was reduced by elevated temperatures and the action of amylase (Ahmed & Ramaswamy 2004). Likewise, mango pulp was treated with varying concentrations and activation lengths of pectinase at different temperatures. Viscosity of the pulp decreased with increasing enzyme concentration and time of treatment (Bhattacharya & Rastogi 1998).

Rheological characteristics of sweetpotato puree

Sweetpotatoes follow similar patterns to these fruit purees. In studying the relationship between rheological characteristics and mouthfeel of sweetpotato puree, Rao et al. (1975a) found sweetpotatoes to exhibit non-Newtonian, pseudoplastic behavior with a yield stress fitting the Herschel-Bulkley model. Yield stresses for the seven different cultivars studied ranged from 230 to 663 dyne/cm² (23 – 66.3 Pa) over a range of storage times. Consistency coefficient values ranged from 17.9 to 248.1 dyne-s/cm² (1.79-24.8 Pa-s) and flow behavior index values varied from 0.333

to 0.564. Viscosity values ranged from 534 to 2893 centipoise (0.534 – 2.89Pa-s) (Rao 1975b).

Like other fruit and vegetable purees, temperature has an effect on sweetpotato puree flow behavior. Kyereme et al. (1999) studied the effect of temperature on viscosity of sweetpotato puree. As expected, sweetpotato puree viscosity decreased as temperature increased. The flow behavior of the puree was well represented by the Herschel-Bulkley or Modified Casson models. Further, the Modified Casson model was used to develop a combination shear rate-temperature model for sweetpotato puree. As part of their studies, Ice et al. (1980) and Szyperski et al. (1986) examined the effect of added amylases on sweetpotato puree viscosity and found that the purees with unhydrolyzed starch had higher yield stresses and apparent viscosities than those purees exposed to enzyme treatments.

2.3.3 – Rheology of Reconstituted Powders

Many food products are made into dried powders to be used in a variety of applications. It is often desirable to reconstitute dehydrated powders back to their previous state and observe how the reconstituted powders compare to their original fluid state. Knowing the rheological characteristics of these powders in reconstituted solutions is important in determining powder functionality in the intended food application. Additionally, these characteristics give insight into product on a molecular level.

In a recent study, Abu-Jdayil et al. (2004) compared the rheological characteristics of tomato paste with solutions made from reconstituted tomato

powder. Specifically, the flow behavior and time dependency were examined at various solids concentrations and temperatures. The tomato paste best fit a Bingham plastic model. At low solids concentrations, the tomato reconstituted solution (TRCS), behaved as a Newtonian fluid. However, as powder concentration increased, the reconstituted solution exhibited Bingham plastic behavior. The yield stress and the viscosity of both products were similar and exhibited an increase with increasing solids concentration and a decrease with increasing temperature. However, at similar concentrations and temperatures, the stress values for the tomato paste were much higher than those of the TRCS. This difference is attributed to changes in pectins and other long chain carbohydrate polymers found in the tomato paste. These polymers are hydrolyzed by the heat of spray drying resulting in smaller molecules which affect the colloidal properties of the solution. Also, the yield stress and apparent viscosity of the tomato paste were much higher than that of the TRCS at similar concentrations and temperatures. Large differences in particle size between the dried powder and the paste, which has suspended particles in colloidal serum, accounted for differences in yield stress and viscosity. TRCS exhibited time independent behavior while tomato paste showed slight time dependency.

Little work has been done on the rheological characteristics of reconstituted solutions of drum dried sweetpotato flakes. Rao and Graham (1982) studied the effects of pre-drying treatments on sweetpotato puree on the rheological, chemical, and textural characteristics of dehydrated sweetpotato flakes. In this study, the reconstituted solution flow data was best fit to a Herschel-Bulkley model. Yield

stress values ranged from 8.95 to 16.82Pa. Values for K, the consistency coefficient, ranged from 1.76 to 11.18Pa-s while the flow behavior index went from 0.38 to 0.53. These values are comparable to the values obtained by Rao et al. (1975) for sweetpotato puree except that the yield stress values of the puree were much higher (23-66Pa). The range for the consistency coefficient of the puree also extended to about 25 Pa-s. These differences can be attributed to further fragmentation of starch and other polymers upon dehydration.

2.4 – FRUIT AND VEGETABLE POWDER APPLICATIONS

Fruit and vegetable juice powders have promising application in the food industry as value-added ingredients providing numerous functional and nutritional benefits. As an ingredient, these powders provide functionality in the food system as well as improving the health image of the product for consumers. Additionally, these powders offer the handling and storage benefits of dry ingredients along with the characteristics of the original juice (Francis & Phelps 2003).

Freeze dried fruit and vegetable powders retain the nutrients, colors, and flavors of the original fruits and vegetables. These ingredients thus can deliver texture properties, flavor, color, and nutraceutical benefits, or other functional or health attributes adding value to the food system. Fruits and vegetables are often used as natural colorants. If the anthocyanin, carotenoid, chlorophyll, and tannin pigments can be maintained throughout processing, then fruit and vegetable powders can be used to naturally color foods. Fruit juice powders can also be used

to provide familiar flavor notes to foods. Dried fruit and vegetable powders also have good water binding capabilities as these powders retain the humectant properties of the sugars and fibers originally present in their juices (Francis & Phelps 2003).

Fruit and vegetable powders can enhance the nutritional benefits of many products by contributing fiber, vitamins, minerals, or bioflavonoids. Their physico-chemical properties make them ideal for carrying fat and water soluble nutrients. Fruit and vegetable powders contain high levels of anthocyanidins, flavonols, and flavones and therefore these powders can contribute to the antioxidant capacity of the food products to which the powders are added (Francis & Phelps 2003). For example, freeze dried tomato powders were found to have properties that could help prevent oxidative degradation in foods and in vivo (Lavelli et al. 2001). Fruits and vegetables are also good sources of fiber and thus their powders can be added to foods to help support a fiber health claim. Finally, fruits and vegetables are significant sources of vitamins and minerals. If fruit and vegetable powders are able to retain vitamin levels during processing, these powders can provide products with enhanced levels of vitamins (Francis & Phelps 2003).

In a review of ingredient delivery systems, Pszczola (2003) emphasized both freeze dried and drum dried fruit and vegetable powders. Freeze dried powders add characteristic flavors and nutrients to foods and have been used to create ethnic flavor profiles in sauces, marinades, and seasonings. Drum dried fruit and vegetable ingredients are an economical way to impart color, flavor, and nutrients in a food. These powders are easy to incorporate into the food and are suitable for use

in soup and sauce mixes, baby foods, dips, extruded cereal products, fruit purees for confections, and fillings for frozen toaster snacks.

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Table 1. Proximate composition of dry matter in raw sweet potato root (Woolfe 1992).

| Component | % in Dry Matter | |
|---|------------------------|--------------|
| | Average | Range |
| Starch | 70 | 30 – 85 |
| Total Sugars | 10 | 5 – 38 |
| Total Protein (N x 6.25) | 5 | 1.2 – 10 |
| Lipid | 1 | 1 – 2.5 |
| Ash | 3 | 0.6 – 4.5 |
| Total Fiber (Non-starch polysaccharides & lignin) | 10 | |
| Vitamins, organic acids, and others | <1 | |

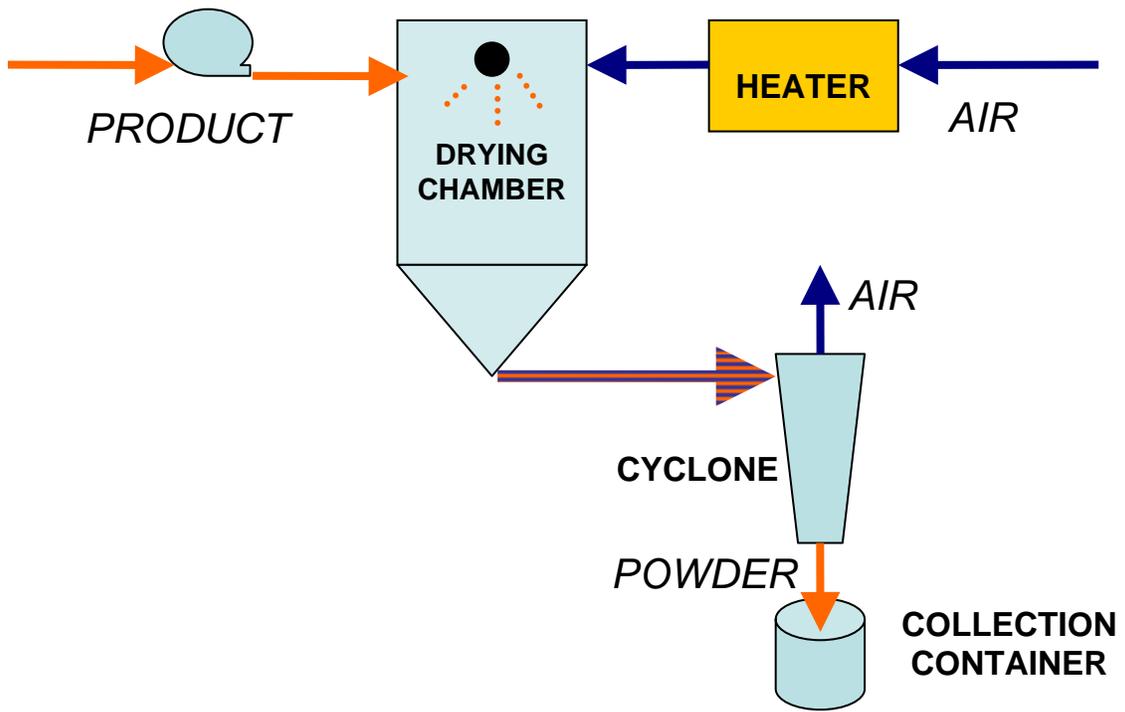


Figure 1. Spray drying process

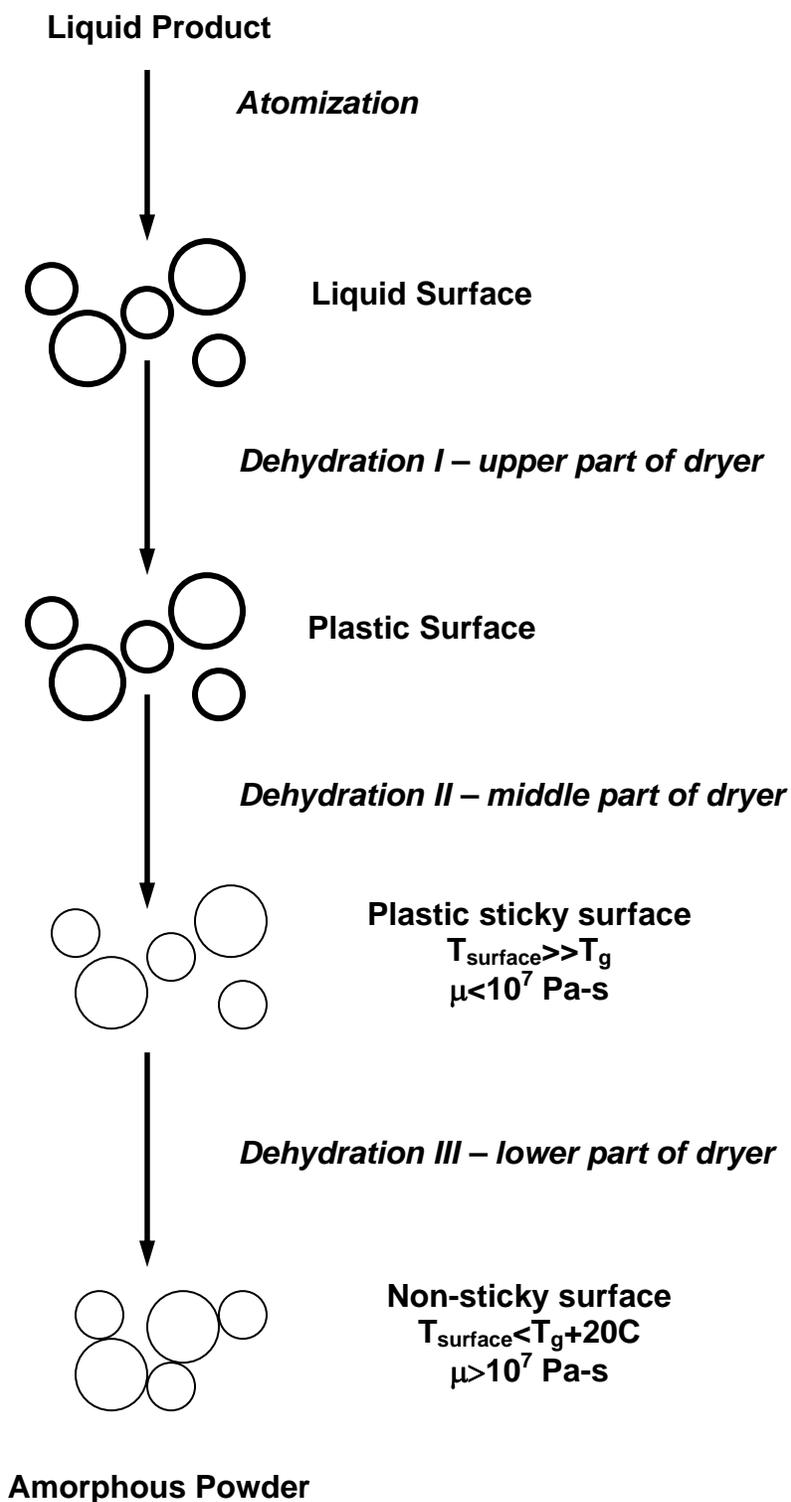


Figure 2. Schematic representation of physical changes on droplets during spray drying process. Dehydration I, II, III represent only arbitrary stages of dehydration, μ =viscosity, T_g =glass transition temperature, T_{surface} =surface temperature of drying particle (Bhandari et al 1997a).

CHAPTER 3

EFFECTS OF ALPHA-AMYLASE, MALTODEXTRIN, AND DRYING TEMPERATURE ON THE PHYSICOCHEMICAL PROPERTIES OF SPRAY DRIED SWEETPOTATOES

3.1 – ABSTRACT

An approach intended to increase the consumption of sweetpotatoes is to convert sweetpotato puree into a dried powder to be used as a functional ingredient in food systems. Spray drying, which has been used for commercial production of several fruit and vegetable powders, has not yet been studied for sweetpotato processing. Thus, the objective was to determine the effects of alpha-amylase, maltodextrin concentration, and inlet air temperature on the physicochemical characteristics of spray dried sweetpotato powder. Amylase and elevated temperature were employed to reduce puree viscosity before spray-drying on a pilot-scale dryer. A face center cube design was used to evaluate the effects of amylase level (0, 3.75, and 7.5ml/kg puree), inlet air temperature (150, 190 and 220°C) and maltodextrin concentration (0, 10 and 20%) on powder characteristics. Model-fitting using response surface methodology was performed to examine the effects of the pre-drying treatments and spray drying conditions on the moisture content, color, water absorption, solubility, particle size, bulk density, and glass transition temperature. The data was fit to a full second order polynomial equation; however, only the linear and quadratic and not the interaction terms proved to be significant for most dried powder attributes. Maltodextrin significantly increased powder solubility, altered the hue value, and raised the glass transition temperature of the powder. Pre-treatment of alpha-amylase resulted in a lowering of the glass transition temperature and a decrease in particle size. Although specific optimum conditions were not obtained within the examined region, a combination of amylase

treatment and maltodextrin created a powder balancing all functional characteristics. Overall, results show that good quality sweetpotato powders can be produced using this drying method with potential applications in dry mixes, soups, beverages, and other food and nutraceutical products.

3.2 – INTRODUCTION

Sweetpotatoes are highly nutritious vegetables rich in calories and biologically active phytochemicals. However, sweetpotato consumption has been on the decline in industrialized nations like the United States (Woolfe 1992, FAO 2005). Limited choices of sweetpotato products for consumers beyond the raw root as well as difficulties in availability, storage, and handling for food processors all contribute to the consumption decline (Kays 1985).

One approach intended to increase consumption of sweetpotatoes is to convert sweetpotato puree into a dried powder to be used as a functional ingredient in food systems. Dried fruit and vegetable powders can contribute color, flavor, water binding capacity, and nutritional benefits to various food products. These powders are easily incorporated into foods and are suitable for use in soup and sauce mixes, baby food, dips, and cereal products (Francis & Phelps 2003).

Several different fruit and vegetable powders have been produced using spray drying; however, this drying technique has not yet been applied to sweetpotatoes. Tomatoes, Roselle, blackcurrant, apricot, raspberry, guava, pineapple, and several other fruits and vegetables have all been successfully spray

dried into powders with varying characteristics (Goula et al. 2004a, Al-Kahtani & Hassan 1990, Bhandari et al. 1993, Chopda & Barrett 2001, Abadio et al. 2004).

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. This dehydration technique transforms the product from a fluid state to a dried powder by spraying fluid into a hot air drying chamber.

Atomization of the feed in the dryer greatly increases surface area which allows the product to dry rapidly without the deterioration in quality as seen in other drying methods. Dehydrated powders have high practical importance in terms of reduced packaging, handling, and transportation costs; decreased storage space; and increased shelf life. Additionally, the resulting powder is of higher quality, can be easily dispersed in water, and readily incorporated into some foods (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 improve puree quality and aid process control (Kyereme et al. 1999, Szyperski et al. 1986). Thus, employing these techniques may increase workability of the puree in the spray drying process.

Furthermore, previous researchers have demonstrated that spray drying foods high in sugar leads to losses in efficiency due to product sticking to the walls of the drying chamber (Bhandari et al. 1997a, Vega et al. 2005). Cooked sweetpotatoes contain greater than 22% sugar on a dry weight basis (Truong et al.

1986). Products with low molecular weights such as sugars have very low glass transition temperatures (T_g) and these components can depress the T_g of the entire food 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 & Howes 1999).

One method to avoid stickiness is to spray dry at temperatures lower than the $T_g+20^\circ\text{C}$. However, this approach is not economically feasible. In order to spray dry sticky products such as sweetpotatoes, carriers such as maltodextrin can be used to facilitate the drying process. The high molecular weight of maltodextrin increases the glass transition temperature of the product. Additionally, maltodextrin also partially encapsulate the particles, forming a protective layer, which consequently reduces stickiness and increases product recovery (Bhandari & Howes 1999, Vega et al. 2005). Maltodextrin also has other desirable properties such as low hygroscopicity, high solubility, and bland flavor (BeMiller & Whistler 1996).

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; and atomization technique have dramatic effects on powder characteristics (Nath & Sapathy 1998, Masters 1991). The addition of maltodextrin can also affect powder moisture content, bulk density, and particle size as well as color, solubility, and glass

transition temperature (Abadio et al. 2004, Chopda & Barrett 2001, Bhandari et al.1997b, Desobry et al. 1997, Bhandari et al. 1993).

One method used to study the effects of drying conditions on finished product characteristics is response surface methodology (RSM). RSM is a collection of mathematical and statistical techniques that has been successfully used for developing, improving, and optimizing processes. This method allows for the evaluation of the effects of several process variables on response variables (Myers & Montgomery 1995). This technique is often used to model and optimize food systems. Most recently, Loh et al. (2005) used RSM to optimize spray drying conditions for the extract of a tropical plant.

Anticipating issues associated with the thick sweetpotato puree, the first objective of this study was to determine applicable pre-drying treatments to reduce sweetpotato puree viscosity to a level suitable for spray drying. These viscosity reduction techniques would undoubtedly influence dried product characteristics. Furthermore, the addition of maltodextrin as a drying aid in the spray drying of sweetpotatoes could affect the resulting powder. Therefore, the second objective was to determine the effects of these pre-drying treatments and drying conditions on the physicochemical characteristics of spray dried sweet potato powder using response surface methodology. The addition of maltodextrin and amylase was expected to facilitate drying performance and improve product functionality.

3.3 – MATERIALS AND METHODS

3.3.1 – Materials

Sweetpotato puree from the orange fleshed Beauregard cultivar with approximately 18% dry matter was manufactured by Bright Harvest Sweet Potato Co. (Clarksville, AK). The frozen puree was shipped in 20kg 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 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 described by Walter and Purcell (1973), the enzyme activity was verified to be 22.9 APA Amylase Units per ml. Maltodextrin (MD) with a dextrose equivalent (DE) of 11 (MD 01960) was obtained from Cargill Inc. (Cedar Rapids, IA).

3.3.2 – Puree Viscosity Reduction

The effects of puree temperature, amount of alpha-amylase, and length of enzyme activation time on puree viscosity were studied in a series of experiments. First, the effect of elevated temperature on puree viscosity was examined and viscosity was subsequently measured at 25, 50, 60, 70, and 90°C. Next the level of amylase addition and length of enzyme activation were studied. Puree was treated with 7.5ml of amylase per kilogram of puree for 30 minutes and the viscosity

measured. Subsequent experimentation increased the length of time the enzyme was active in the sample to 1 hour and 2 hours. The amylase level was then reduced to 3.75 and 2.5ml/kg puree and samples for viscosity measurement were taken at 0, ½, 1, and 2 hours of activation. For all testing with alpha-amylase, viscosity measurements were taken at 25 and 60°C.

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 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 minutes to deactivate the enzymes.

Viscosity of the treated puree was measured using a stress-controlled rheometer (StressTech, Reologica, Lund, Sweden) equipped with serrated bob and cup geometry. Samples were exposed to 30 seconds of pre-shearing at 20s⁻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 minutes before measurements were taken. Flow behavior was described by the Herschel-Bulkley model (Eq.1)

$$\sigma = \sigma_o + K \dot{\gamma}^n \quad (1)$$

where σ =stress, σ_0 =yield stress, $\dot{\gamma}$ =shear rate, K=consistency coefficient, and n=flow behavior index (Steffe 1996).

3.3.3 – Spray Drying of Sweetpotato Puree

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

3.3.3.1 – Equipment and Drying Conditions

Preliminary spray drying experiments were conducted using a pilot scale dryer (Anhydro Laboratory Spray Dryer S-1 Anhydro Inc., Attleboro Falls, MA) equipped with a two-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 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) was then used for the bulk of experimentation. This dryer had a co-current flow regime and utilized a rotary atomizer set at 20,000rpm. Drying air was heated using natural gas combustion. Feed was moved into the dryer using a progressive cavity pump (Metering Pump, Moyno, Inc., Springfield, OH). 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 (~18%), 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 minutes at approximately 60°C before the temperature of the puree was raised above 90°C to deactivate the enzymes. Maltodextrin was similarly mixed with the puree using this system. In order to maintain the same amount of solids being fed to the dryer, water was added to the puree-maltodextrin mixture. For samples testing both amylase and maltodextrin, the puree was treated with amylase and then the enzymes were inactivated. Maltodextrin was mixed with the treated puree just prior to spray drying.

3.3.3.2 – Experimental Design and Statistical Analysis

Response surface methodology (RSM) was used to investigate the optimum combination of pre-drying treatments and drying conditions on the physicochemical properties of the spray dried powders. A three-factor and a three-level face-centered cube design was employed and consisted of seventeen experimental runs including three replicates at the center point (Myers & Montgomery 1995). All 17 treatment combinations can be found in Table 1. The independent design variables were maltodextrin concentration (x_1), amylase level (x_2), and inlet drying air temperature (x_3) while the response variables included moisture content, solubility,

water absorption, color, bulk density, particle size, and glass transition temperature. This design both examined differences between the treatments and generated a model for the response surface to predict how the amylase level, maltodextrin concentration, and inlet drying air temperature will influence powder characteristics. Review of the response surface can also determine if conditions exist for optimal product functionality.

The response surface regression procedure (RSREG) of the statistical analysis system (SAS Release 8.02, Cary, NC) was used to analyze the experimental data and obtain regression coefficients. The data was fit to a full second order polynomial equation as follows

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

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

3.3.3.3 – Powder Analysis

Color

The color of the puree, dry powder, and reconstituted solutions was measured using a tristimulus colorimeter (Model DP9000, HunterLab, Reston, VA) and expressed as L*, a*, 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 (Equations 3-4) (Hutchings 1994). The color change was calculated for the reconstituted samples as compared to the original puree.

$$H^* = \arctan(b^*/a^*) \quad (3)$$

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (4)$$

Moisture Content

Moisture content was determined by Karl-Fischer titration method using a Karl Fischer 701 Titrino (Metrohm Ltd., Herisau, Switzerland). Between 0.25 and 0.30g of spray dried powder were added to the system. Methanol was used as the solvent and Hydranal composite 5 was the reactant. The Karl Fischer unit was enclosed in a plexi-glass dry box with compressed air pumped in to maintain low humidity. The extraction time was set at 180s to enable the sample to fully dissolve. 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 et al. (1969). A small sample of dry powder (2.5g) was added to 30ml of water at 30°C in a 50 ml centrifuge tube, stirred intermittently for 30 minutes, and then centrifuged for 10 minutes at 10,000rpm. The supernatant was carefully poured off into a petri dish and oven dried overnight. The amount of solids in the dried supernatant as a percent of the

total dry solids in the original 2.5g sample gave an indication of solubility index.

Water absorption index is 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 20g of sweetpotato powder to a 50ml graduated cylinder and holding the cylinder on a vortex for 1 minute. The bulk density was calculated by dividing mass of the powder by the volume occupied in the cylinder.

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). Results of this analysis were examined in many 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 50 percentile. These values are calculated using the following equations where V_i =volume of the individual particle and d_i =diameter of particle.

$$MV = \frac{\sum V_i d_i}{\sum V_i} \quad (5)$$

$$MN = \frac{\sum (V_i / d_i^2)}{\sum (V_i / d_i^3)} \quad (6)$$

$$MA = \frac{\sum V_i}{\sum (V_i / d_i)} \quad (7)$$

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 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, NC). Scanning electron micrographs were obtained using a JEOL JSM 5900LV scanning electron microscope (JEOL U.S.A., Peabody, MA) at an accelerating voltage of 15 kV and digital images were captured at various magnifications with JEOL Digital Scan Generator software v2.00.

Glass Transition Temperature

A Perkin Elmer (Norwalk, CT) 7 Series/Unix DSC 7 Differential Scanning Calorimeter 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 5-10mg of sample was prepared in aluminum sample pans. Sweetpotato powders were heated from -70 to 120°C at a rate of 20°C per minute. An empty pan was used at a reference. A baseline was constructed using an empty pan over the same temperature range and scanning rate. Glass transition was analyzed using with Pyris thermal analysis software. 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 et al. 1997a).

3.4 – RESULTS AND DISCUSSION

3.4.1 – 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 (Table 2).

Puree viscosity was measured at temperatures between 25 and 90°C. As expected, shear stress and viscosity decreased as the temperature increased similar to the results of Kyereme et al. (1999). However, at 60°C and a shear rate of 500 s⁻¹,

the viscosity of the puree was 0.70 Pa-s. Further, at 90°C (shear rate = 500 s⁻¹), puree viscosity was still above 0.40 Pa-s (Table 2).

Thus, additional means were required to reduce puree viscosity to the desired range. Water addition is one simple method of reducing puree viscosity. Moreover, the viscosity of sweetpotato puree had previously been successfully reduced by the action of alpha-amylase (Ice et. al 1980, Szyperski et al. 1986). Therefore, the sweetpotato puree was treated with 13% water and 7.5ml of alpha-amylase per kilogram of puree for 30 minutes and viscosity measured at both 25 and 60°C. This treatment significantly reduced the puree viscosity to below .25Pa-s at 60°C (Table 2). Subsequently, longer lengths of enzyme activation time were studied using water addition and 7.5ml amylase/kg puree. One hour of enzyme treatment was not significantly different from 30 minutes of activity. With two hours of treatment, the viscosity actually increased due to moisture loss from extended time at elevated temperatures (Table 2). When the amylase level was cut in half, the puree viscosity was still sufficiently reduced to the desired range at 60°C. However, further reducing the amylase level did not give the required decrease in viscosity.

Water addition is not an efficient means of viscosity reduction for spray drying since extra energy would be required to remove the additional liquid. Thus, a viscosity reduction method that did not involve water addition was desired for reducing sweetpotato puree viscosity for spray drying. Puree was treated with 7.5 and 3.75ml of amylase per kg of puree for 30 minutes and the viscosity measured. These treatments reduced viscosity below 0.40Pa-s at 500s⁻¹ (Table 2). Although this viscosity level was not as low as the initial goal, preliminary spray drying

experiments found this viscosity level to be sufficient for the operation. Thus, amylase levels of 3.75 and 7.5ml/kg puree were tested in further spray drying experimentation.

The flow behavior of the sweetpotato puree studied was fit to the Herschel-Bulkley model. The model parameters also indicated the reduction in viscosity due to elevated temperature and amylase action (Table 3). The consistency coefficient, an indication of viscosity, was reduced with elevated temperature and amylase from 49.4 to 14.6 Pa-sⁿ. The flow behavior index of the puree with these treatments was between 0.39 and 0.32 indicating shear thinning behavior while yield stress values ranged between 18 and 37.5 Pa. Similarly, in a study of the relationship between rheological characteristics and mouthfeel of sweetpotato puree, Rao et al. (1975) found sweetpotato puree to exhibit non-Newtonian, pseudoplastic behavior with a yield stress fitting the Herschel-Bulkley model. Yield stresses for the seven different cultivars studied ranged from 23 – 66.3 Pa, consistency coefficient values ranged from 1.79-24.8 Pa-sⁿ, and flow behavior index values varied from 0.333 to 0.564. Additionally, in a study of elevated temperature effects on sweetpotato puree, Kyereme et al. (1999) reported Herschel-Bulkley parameters between 2.8 and 21.5Pa-sⁿ for the consistency coefficient and 0.33 to 0.20 for the flow behavior index with a yield stress of 10Pa.

3.4.2 – Spray Dried Sweetpotato Powder Characteristics

The physicochemical characteristics of dried sweetpotato powder for each treatment are shown in Tables 4 and 5. Estimated regression coefficients for physical characteristics responses with the coefficient of determination (R^2) are shown in Tables 6 - 9. Analysis of variance of the regression parameters of the predicted response surface quadratic models for all physicochemical attributes are shown in Appendix Tables A1 – A4. Analysis of variance for the response surface quadratic model for each physicochemical characteristic showing lack of fit are shown in Appendix Tables B1 – B3.

Color

Color was represented by L^* , a^* , b^* where L^* values range from black to white, 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. Maltodextrin had a highly significant effect ($p < .001$) on both measured and calculated (chroma, hue, ΔE) color values of the spray dried powders, while amylase and inlet temperature showed minimal effects on some color values (Tables 6 & 7). Raw puree is orange in color – a mix of yellow and red. As the maltodextrin concentration was increased, the a^* and b^* values decreased indicating a loss in “yellowness” and “redness”. For example, comparing treatments 3 and 4 without maltodextrin to treatments 1 and 2 that had 20% maltodextrin (Table 4), a^* values and b^* values were both less in the maltodextrin treatments.

But perhaps the color change due to maltodextrin concentration is best described by the change in hue value. Hue values are affected by maltodextrin in

both the dry powder and the reconstituted solution (Table 6 & 7). Raw puree has a hue value of 67.0°. As maltodextrin 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 4). If the sweetpotato powder is desired to have color similar to the puree, ideally then, a lower level of maltodextrin should be used. In study of spray dried pineapple juice, Abadio et al. (2004) reported that the color of all the light yellow powders did not appear to vary at maltodextrin concentrations up to 15%. However, Chopda et al. (2001) found the color of guava powders spray dried with maltodextrin to be bright white in appearance irrespective of the color of the feed material. However, maltodextrin levels of greater than 40% were used in the study. In a practical sense, the color of the sweetpotato powder will become whiter, and less orange, as the level of maltodextrin increases and 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 5). Each independent variable had a treatment effect on moisture content of the finished powder ($0.001 < p < 0.05$) (Table 8). Higher inlet drying air temperatures often resulted in decreased moisture content. This trend may be difficult to discern in this experimental design; however, preliminary experiments on sweetpotato puree showed a decrease in moisture content as drying temperature increased. 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 et al. (2004a) reported similar results for spray dried tomato paste. Moisture content also decreased with increasing maltodextrin concentration (Table 5), similar to the results Abadio et al. (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 dextrans created by alpha-amylase action.

Solubility and Absorbance

The instant properties of a powder involve the ability of a powder to dissolve in water. Most powdered foods are intended for rehydration and the ideal powder would wet quickly and thoroughly, sink rather than float, and disperse/dissolve without lumps (Hogekamp & Schubert 2003). As discussed in the modeling section below, water solubility could not be modeled by the second order polynomial as none of the regression coefficients were significant (Table A3). However, the addition of maltodextrin during the spray drying of sweetpotatoes impacted the solubility of the sweetpotato powders. Solubility index increased by more than 20 units as a result of adding maltodextrin (Table 5). Conversely, adding maltodextrin reduced the water holding capacity of the sweetpotato powders (Table 5). 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

Pre-treating the puree with alpha-amylase prior to spray drying and the addition of maltodextrin both had significant effects on particle size and thus, bulk density. According to the model (Table 9), particle size decreases with increasing

amylase treatment. In preliminary experiments on the two-fluid nozzle pilot scale dryer, amylase treatment was compared to a control. In that experiment, mean particle diameter of the volume distribution decreased from 95.9 μm to 58.1 μm with amylase treatment. Likewise, 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 (2004b) 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 impact on bulk density (Table 9). Unfortunately, a low R^2 value does not allow one to adequately predict bulk density based on the linear model. Bulk density was observed to decrease with amylase treatment (Table 5). 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 & Adamopoulos 2004b). 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 5).

Since sweetpotato puree was spray dried on both a two-fluid nozzle and 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 10, Figure 4). Powders for 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 two-fluid nozzle.

Glass Transition Temperature

Maltodextrin, 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 5). This behavior was also observed in preliminary drying experiments (data not shown). Increases in drying temperature can result in lower residual moisture content. This 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 maltodextrin was added (Figure 5, Table 5). The positive regression coefficient indicated linear effects that may increase the response of T_g to maltodextrin (Table

8). The increase in T_g is likely caused by an increase in molecular weight of the powder. Bhandari et al. (1997b) also reported an increase in glass transition temperature and drying efficiency as the ratio of maltodextrin 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 maltodextrin having a significant impact; however, the R^2 was only 0.42 so T_g cannot be accurately predicted by this model alone.

Predictive Models

The results indicate that the linear and quadratic parameters were significant for many of the powder characteristics (Tables A1 – A4). On the other hand, the interaction terms did not produce a significant effect for any of the physicochemical attributes. Moisture content, WAI, reconstituted a^* , b^* , and hue, 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. Thus, using the full second-order polynomial model was not needed to fit

the data because most of the powder characteristics could be described by an additive rather than multiplicative model.

Based on these observations, the data was 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 Tables 6 - 9. The lack of fit for these models was insignificant (Tables B1 – B3). None of these models fit 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, reconstituted a^* value, reconstituted hue value, moisture, WAI, MA, and MedV were all above 0.80 indicating that the models are sufficiently accurate for prediction purposes. A high proportion of the variability was explained by the mathematical models for these attributes and thus, the developed model could be considered accurate (Loh et al. 2005). Conversely, T_g , bulk density, MN, and reconstituted powder b^* value, and ΔE , had some significant terms in the model but the overall model had very low R^2 values.

Maltodextrin 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 maltodextrin on particle diameter (MA and MedV) was also highly significant ($p < 0.01$). T_g and b^* value of the reconstituted powder were also impacted ($p < 0.05$) by maltodextrin but solubility index, ΔE , bulk density, and particle diameter (MV and MN) showed no significant effects. For maltodextrin, 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 & MedV) ($p < 0.05$).

Amylase had a highly significant effect on powder hue ($p < 0.001$) and particle diameter (MN) ($p < 0.01$) and 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 most optimal finished product characteristics for dried sweetpotato powders as 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 instrumental and 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 use 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 an optimal product.

3.5 – CONCLUSIONS

Prior to spray drying, the viscosity of sweetpotato puree can be reduced by a combination of elevated temperature and the action of alpha-amylase. As seen by glass transition temperatures, if amylase is used as a puree viscosity reduction method, then maltodextrin should also be used as a drying aid to help increase the glass transition temperature of the system to avoid stickiness and aid product recovery.

Response surface methodology was used to examine effects of alpha-amylase treatment, maltodextrin concentration, and inlet temperature on spray dried sweetpotato characteristics. Sound models were developed for some attributes while further study is needed to accurately develop prediction models for other powder characteristics. However, using RSM did show that pre-drying treatments as well as drying conditions impacted the final powder characteristics of spray dried sweetpotatoes and that powder functionality in food systems will be affected by alpha-amylase and maltodextrin addition to sweetpotato puree.

Optimal product attributes will be determined by the desired application of this ingredient. Changing the levels of the independent variables allows for alterations in the final powder characteristics. Further experimentation using response surface methodology 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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Table 1. Levels of independent variables in experimental design

| Treatment | x_1 | x_2 | x_3 | Maltodextrin | Amylase | Inlet |
|--------------|-------|-------|-------|-------------------|---------------|------------------|
| | | | | Concentration (%) | Level (ml/kg) | Temperature (°C) |
| Coded Levels | | | | Actual 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 |

Table 2. Sweetpotato puree viscosity at shear rates of 50, 100, and 500s⁻¹ after treatments with elevated temperature, water addition, and alpha-amylase

| Temperature (°C) | Water Addition (%) | Amylase Level (ml/kg puree) | Length of Activation (hour) | 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 |
| 25 | 13 | 7.5 | 0.5 | 1.43 ± 0.205 | 0.749 ± 0.138 | 0.246 ± 0.006 |
| 60 | 13 | 7.5 | 0.5 | 0.806 ± 0.047 | 0.483 ± 0.027 | 0.165 ± 0.003 |
| 60 | 13 | 7.5 | 1 | 0.810 ± 0.014 | 0.559 ± 0.095 | 0.171 ± 0.006 |
| 60 | 13 | 7.5 | 2 | 1.58 ± 0.113 | 0.888 ± 0.057 | 0.261 ± 0.003 |
| 60 | 13 | 3.75 | 0.5 | 1.39 ± 0.113 | 0.809 ± 0.093 | 0.212 ± 0.006 |
| 60 | 13 | 3.75 | 1 | 1.22 ± 0.184 | 0.683 ± 0.124 | 0.206 ± 0.006 |
| 60 | 13 | 3.75 | 2 | 1.48 ± 0.290 | 0.781 ± 0.112 | 0.242 ± 0.007 |
| 60 | 13 | 2.5 | 0.5 | 2.12 ± 0.170 | 1.23 ± 0.134 | 0.395 ± 0.010 |
| 60 | 13 | 2.5 | 1 | 1.94 ± 0.049 | 1.22 ± 0.021 | 0.403 ± 0.013 |
| 60 | 13 | 2.5 | 2 | 2.35 ± 0.226 | 1.39 ± 0.106 | 0.443 ± 0.010 |
| 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 | 0.996 ± 0.006 | 0.334 ± 0.006 |

Table 3. Herschel-Bulkley flow model constants for sweetpotato puree at different temperatures and alpha-amylase treatments over a shear rate range of 1-500s⁻¹

| Temperature (°C) | Alpha-Amylase Addition (ml/kg puree) | Yield Stress, σ_0 (Pa) | Flow behavior index, n | Consistency coefficient, K (Pa·s ⁿ) | R ² |
|------------------|--------------------------------------|-------------------------------|------------------------|---|----------------|
| 25 | 0 | 35.0 ± 14.1 | 0.322 ± 0.043 | 49.4 ± 14.6 | 0.96 |
| 60 | 0 | 37.5 ± 3.5 | 0.371 ± 0.012 | 26.44 ± 2.2 | 0.98 |
| 60 | 3.75 | 18.0 ± 2.8 | 0.394 ± 0.010 | 14.64 ± 0.099 | 0.99 |

Table 4. Color values of spray dried sweetpotato powders and reconstituted powder solutions

| Treatment | Dry Powder | | | | Reconstituted Powder Solutions | | | | |
|-----------|------------|------|-------|---------|--------------------------------|-------|-------|---------|-------|
| | L* | a* | b* | Hue (°) | L* | a* | b* | Hue (°) | 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 |

Table 5. 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)* | | | |
|-----------|----------------------|-------|------|---------------------|---------------------|------------------------------|-------------|-----------|-------------------------|
| | | | | | | Volume -MV- | Number -MN- | Area -MA- | Fifty 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 | | | | |

* Particle size was only measured on one of the center point replications.

Table 6. Regression coefficients of predicted models for the response of powder color variables

| Coefficient | L* | a* | b* | Hue |
|--------------|-------------|--------------|-------------|------------|
| β_0 | 72.347*** | 7.8099 | 18.886 | 74.844**** |
| Linear | | | | |
| β_1 | 0.84569**** | -0.45888**** | -1.2409**** | 0.30645*** |
| β_2 | -0.23868 | -0.25540 | -0.59312** | 0.21639 |
| β_3 | 0.10475 | 0.009279 | 0.20772 | 0.02654 |
| Quadratic | | | | |
| β_{11} | -0.01947*** | 0.01023*** | 0.02789**** | -0.00493 |
| β_{22} | 0.01088 | 0.03615 | 0.05928* | -0.04322 |
| β_{33} | -0.00031 | -0.00002 | -0.00055 | -0.000085 |
| R^2 | 0.96 | 0.97 | 0.99 | 0.91 |
| CV | 1.12 | 8.41 | 2.54 | 0.883 |

*Significant at 10%

** Significant at 5%

***Significant at 1%

****Significant at 0.1%

Table 7. Regression coefficients of predicted models for the response of reconstituted powder solution color variables

| Coefficient | a* | b* | Hue | ΔE |
|--------------|--------------|-------------|-------------|------------|
| β_0 | 17.1311**** | 41.8868**** | 67.1401**** | 5.9365*** |
| Linear | | | | |
| β_1 | -0.24630**** | -0.07160** | 0.31520**** | |
| β_2 | -0.23360** | -0.03947 | 0.30587** | |
| β_3 | -0.01663 | -0.01409 | 0.01628 | |
| Quadratic | | | | |
| β_{11} | | | | -0.00059 |
| β_{22} | | | | -0.00829 |
| β_{33} | | | | 5.46E-05 |
| R^2 | 0.83 | 0.42 | 0.82 | 0.15 |
| CV | 9.99 | 2.39 | 1.85 | 20.70 |

*Significant at 10%

** Significant at 5%

***Significant at 1%

****Significant at 0.1%

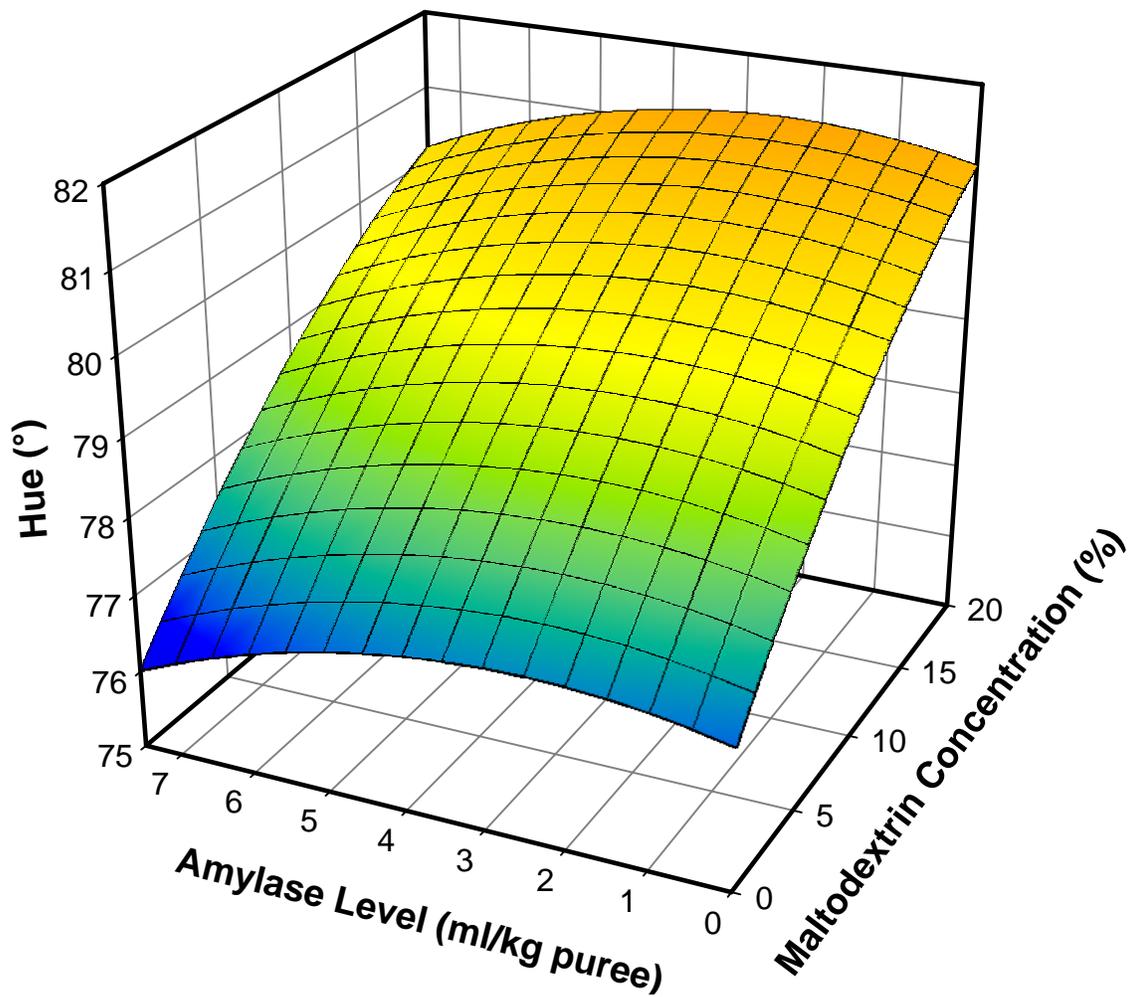


Figure 1. Response surface of hue values of sweetpotato powder spray dried at an inlet temperature of 190°C as a function of maltodextrin concentration and alpha-amylase addition. Model R^2 value is 0.91

Table 8. Regression coefficients of predicted models for the response of moisture, water absorption, and glass transition temperature

| Coefficient | MC | WAI | T _g |
|----------------|-------------|------------|----------------|
| β_0 | 0.9615* | 283.13**** | 69.53**** |
| Linear | | | |
| β_1 | -0.0592**** | -4.975**** | 0.4155** |
| β_2 | 0.0587** | -2.979 | -0.7387 |
| β_3 | 0.0083*** | -0.2262 | -0.0634 |
| R ² | 0.83 | 0.81 | 0.45 |
| CV | 12.92 | 12.1 | 8.92 |

*Significant at 10%

** Significant at 5%

***Significant at 1%

****Significant at 0.1%

Table 9. Regression coefficients of predicted models for the response of bulk density and particle size

| Coefficient | Mean Particle Diameter (μm) | | | |
|--------------|--|----------------|--------------|----------------------------|
| | Bulk Density | Number -MN- | Area -MA- | Fifty Percentile -MedV- |
| β_0 | 0.84583**** | 10.0001**** | 27.849 | 42.906 |
| Linear | | | | |
| β_1 | 0.00082 | 0.00240 | -1.3253*** | -2.5909*** |
| β_2 | -0.01384** | -0.24267*** | -1.0313 | -0.8165 |
| β_3 | -0.00044 | -0.00095 | -0.00122 | -0.05556 |
| Quadratic | | | | |
| β_{11} | | | 0.04597** | 0.08674** |
| β_{22} | | | 0.06518 | 0.07249 |
| β_{33} | | | 0.000045 | 0.00027 |
| R^2 | 0.42 | 0.53 | 0.88 | 0.81 |
| CV | 7.9 | 9.23 | 10.47 | 18.70 |

*Significant at 10%

** Significant at 5%

***Significant at 1%

****Significant at 0.1%

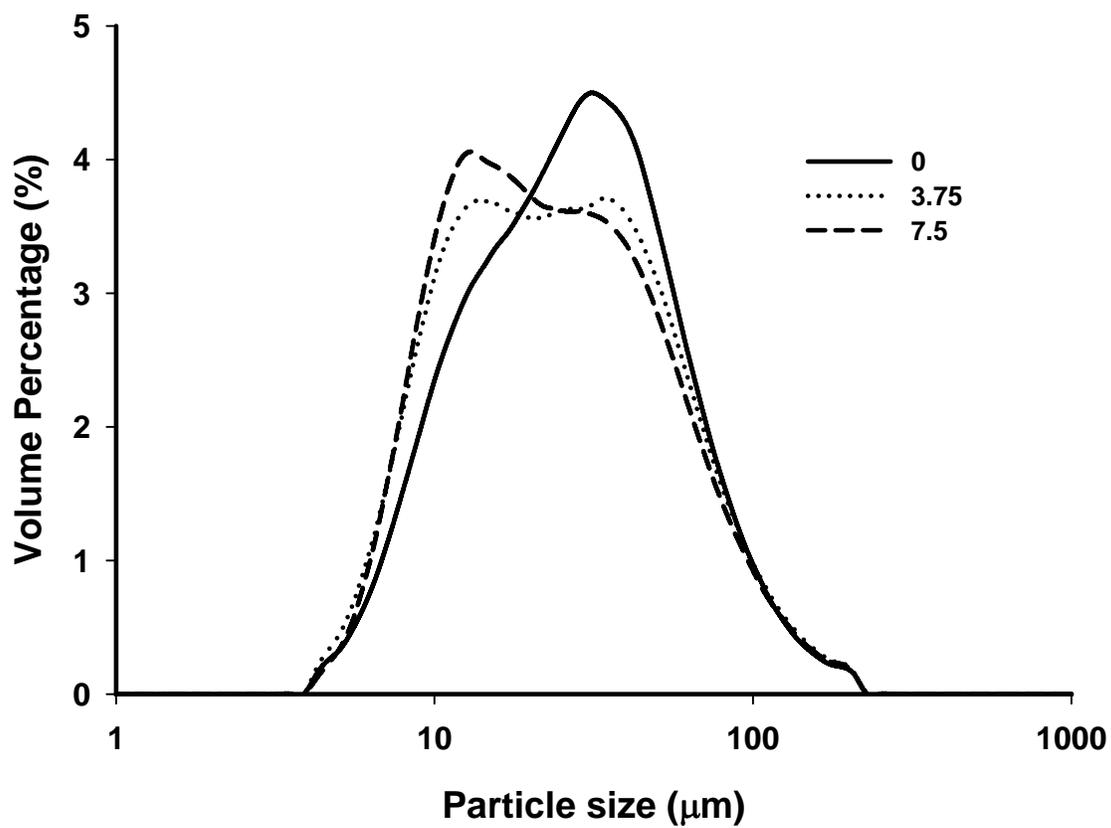
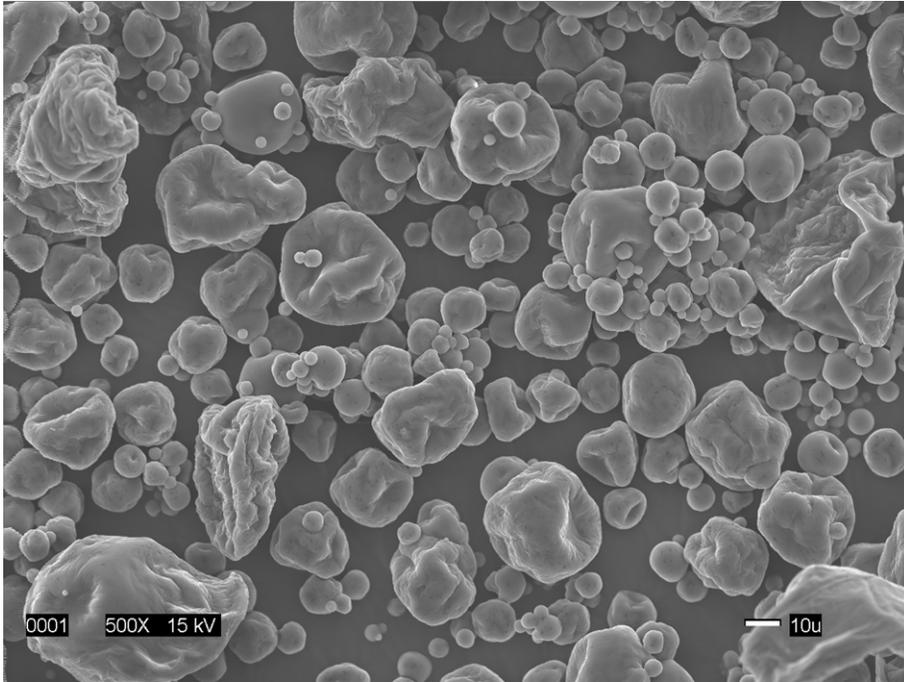
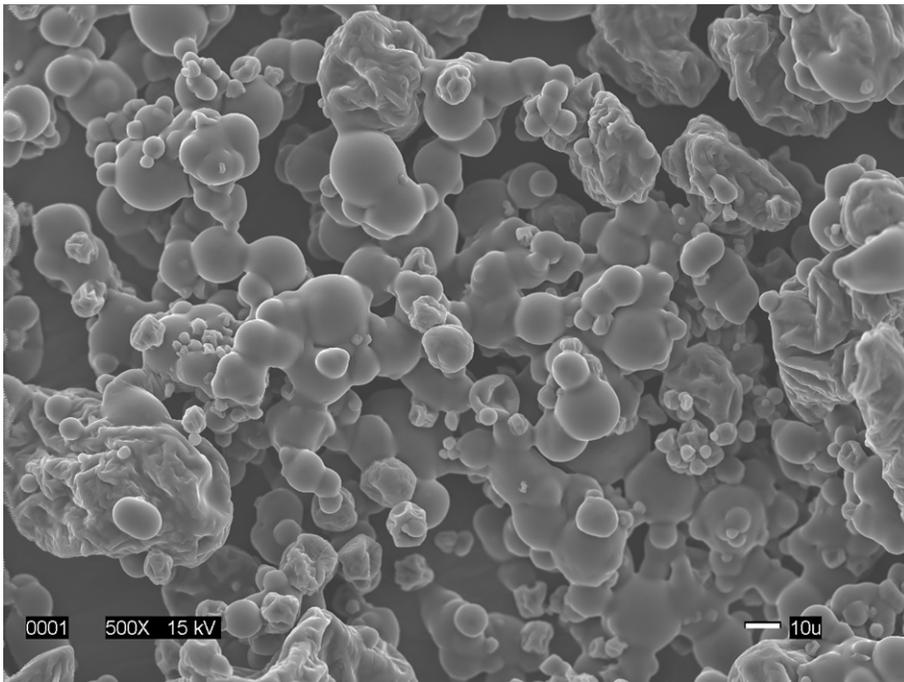


Figure 2. Particle size distributions for different levels of amylase treatment



(a) No amylase, 10% maltodextrin, 190°C inlet drying air temperature



(b) 3.75ml amylase/kg puree, 10% maltodextrin, 190°C inlet drying air temperature

Figure 3. Scanning electron microscope image (500x magnification) of spray dried sweetpotato powders with and without amylase treatment

Table 10. Summary of particle size analysis for 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

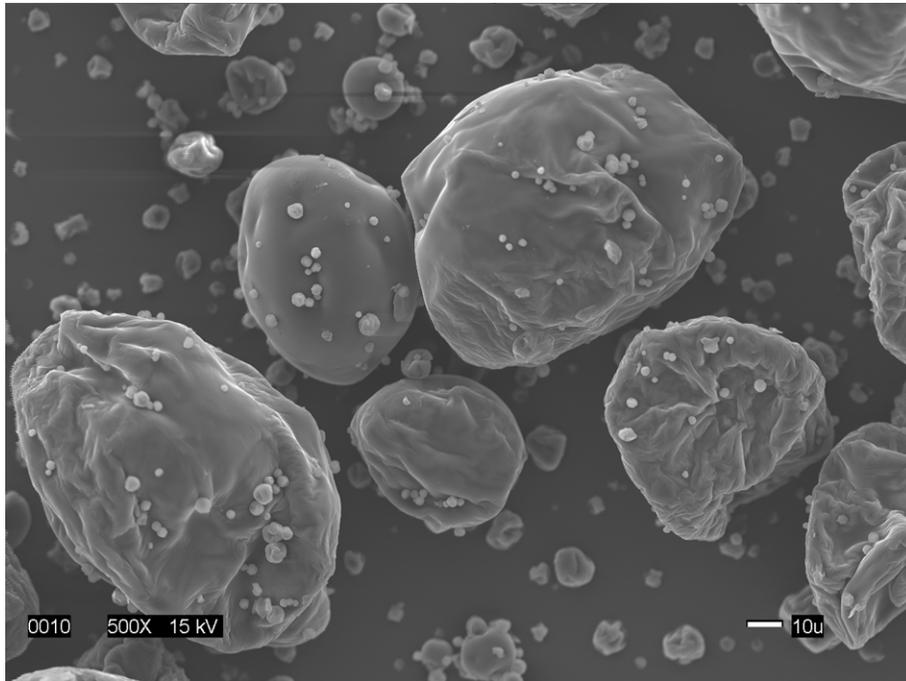
| Atomizer Type | Average diameters (μm) | | | |
|------------------|------------------------|---------------------|-------------------|-------------------------------|
| | Volume ^a | Number ^b | Area ^c | Fifty Percentile ^d |
| Two-fluid nozzle | 102 | 14.14 | 69.93 | 93.58 |
| Rotary atomizer | 33.8 | 8.91 | 19.75 | 26.34 |

^a Mean diameter of the volume distribution.

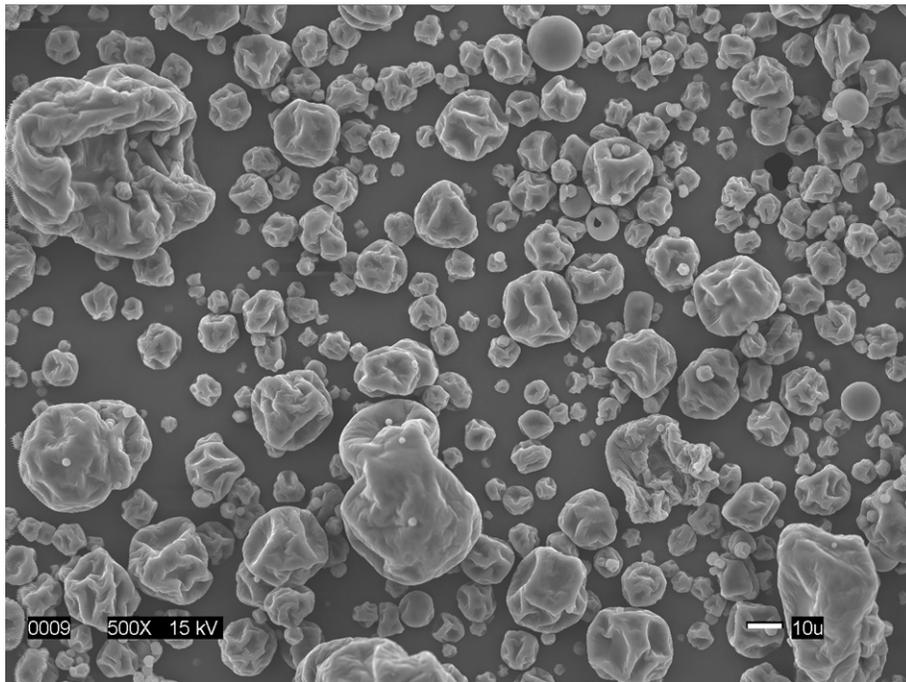
^b Mean diameter of the number distribution.

^c Mean diameter of the particle surface area distribution.

^d Median diameter of the volume distribution.



(a) Two-fluid nozzle



(b) Rotary atomizer

Figure 4. Scanning electron microscope image (500x 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.

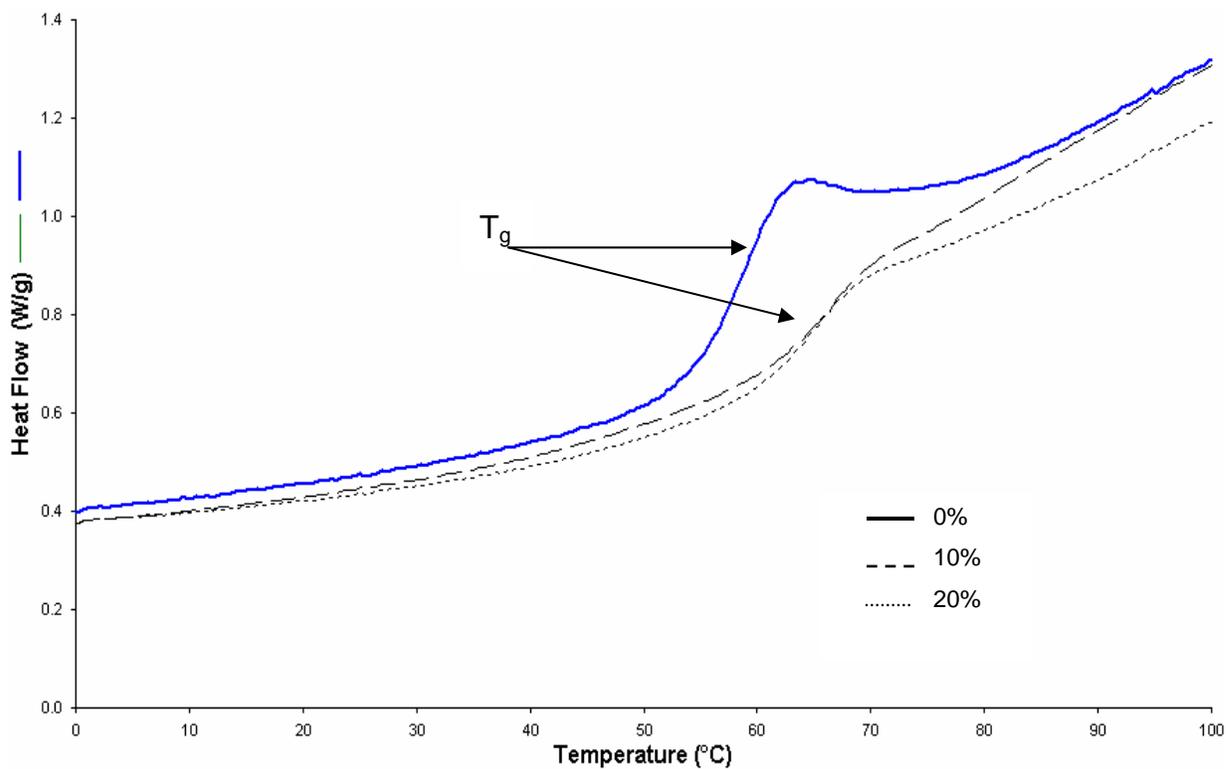


Figure 5. Differential scanning calorimetry (DSC) thermographs showing the glass transition temperature (T_g) of spray dried sweetpotato powder with increasing levels of maltodextrin concentration.

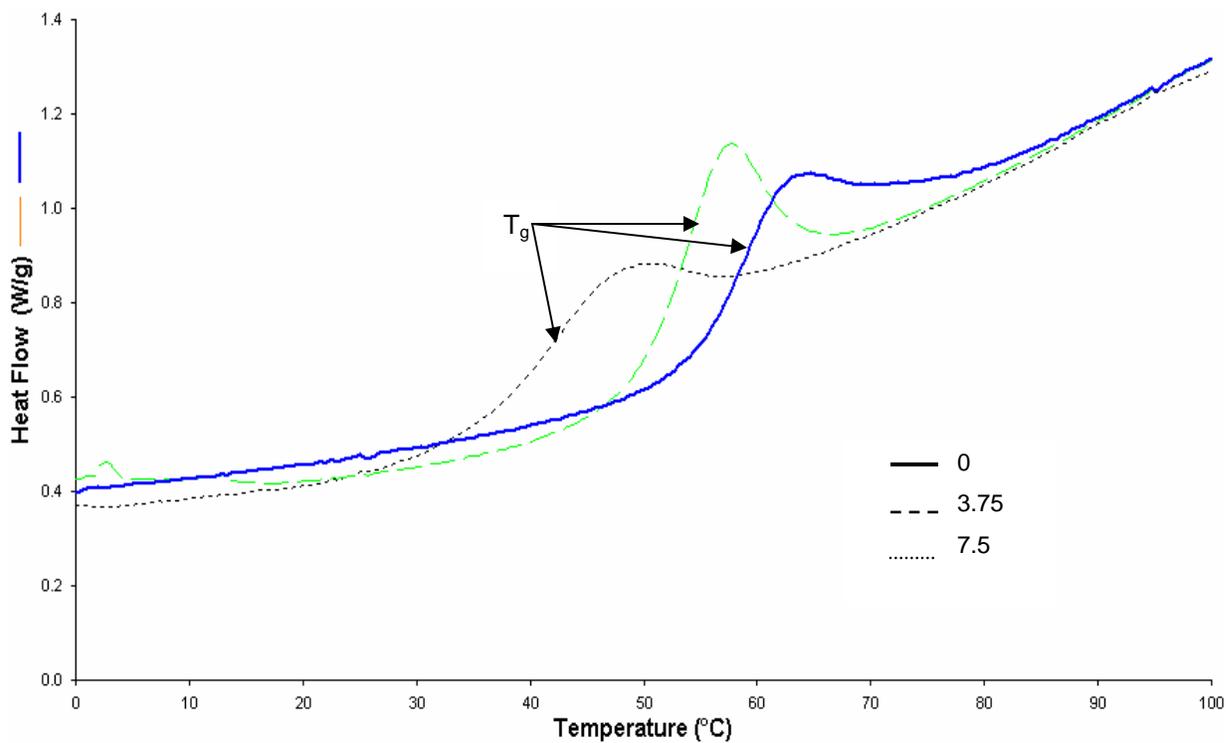


Figure 6. Differential scanning calorimetry (DSC) thermographs showing the glass transition temperature (T_g) of spray dried sweetpotato powder with increasing levels of amylase treatment.

3.7 – APPENDICES

APPENDIX A

Tables of the analysis of variance of the regression parameters of the predicted response surface quadratic models

Table A1. Analysis of variance of the regression parameters of the predicted response surface quadratic models for the color values of spray dried sweetpotato powder

| Regression | DF | Type 1 Sum of Squares | R- Square | F Value | p-value |
|---------------|----|-----------------------------|--------------|---------|----------|
| <i>L*</i> | | | | | |
| Linear | 3 | 212.46 | 0.89 | 96.72 | < 0.0001 |
| Quadratic | 3 | 17.79 | 0.07 | 8.10 | 0.01 |
| Crossproduct | 3 | 4.04 | 0.02 | 1.84 | 0.23 |
| Total Model | 9 | 234.28 | 0.98 | 35.55 | < 0.0001 |
| <i>a*</i> | | | | | |
| Linear | 3 | 64.72 | 0.87 | 94.16 | < 0.0001 |
| Quadratic | 3 | 7.44 | 0.10 | 10.83 | 0.005 |
| Crossproduct | 3 | 0.86 | 0.01 | 1.25 | 0.36 |
| Total Model | 9 | 73.03 | 0.98 | 35.41 | < 0.0001 |
| <i>b*</i> | | | | | |
| Linear | 3 | 469.94 | 0.92 | 253.66 | < 0.0001 |
| Quadratic | 3 | 36.35 | 0.07 | 19.62 | 0.0009 |
| Crossproduct | 3 | 1.18 | 0.002 | 0.64 | 0.61 |
| Total Model | 9 | 507.47 | 0.99 | 91.31 | < 0.0001 |
| <i>Hue</i> | | | | | |
| Linear | 3 | 45.07 | 0.83 | 46.14 | < 0.0001 |
| Quadratic | 3 | 4.28 | 0.08 | 4.38 | 0.05 |
| Crossproduct | 3 | 2.58 | 0.05 | 2.64 | 0.13 |
| Total Model | 9 | 51.93 | 0.96 | 17.72 | 0.0005 |
| <i>Chroma</i> | | | | | |
| Linear | 3 | 522.03 | 0.92 | 238.41 | < 0.0001 |
| Quadratic | 3 | 41.94 | 0.07 | 19.15 | 0.0009 |
| Crossproduct | 3 | 1.26 | 0.002 | 0.58 | 0.65 |
| Total Model | 9 | 565.23 | 0.99 | 86.05 | < 0.0001 |

Table A2. Analysis of variance of the regression parameters of the predicted response surface quadratic models for the color values of reconstituted powder solutions

| Regression | DF | Type 1 Sum of Squares | R-Square | F Value | p-value |
|---------------|----|-----------------------------|----------|---------|---------|
| <i>L*</i> | | | | | |
| Linear | 3 | 78.57 | 0.09 | 0.43 | 0.74 |
| Quadratic | 3 | 341.01 | 0.40 | 1.87 | 0.22 |
| Crossproduct | 3 | 3.43 | 0.004 | 0.02 | 0.99 |
| Total Model | 9 | 423.01 | 0.50 | 0.77 | 0.64 |
| <i>a*</i> | | | | | |
| Linear | 3 | 71.75 | 0.83 | 28.65 | 0.0003 |
| Quadratic | 3 | 5.27 | 0.06 | 2.10 | 0.19 |
| Crossproduct | 3 | 3.70 | 0.04 | 1.48 | 0.30 |
| Total Model | 9 | 80.72 | 0.93 | 10.74 | 0.003 |
| <i>b*</i> | | | | | |
| Linear | 3 | 7.79 | 0.42 | 3.62 | 0.07 |
| Quadratic | 3 | 5.16 | 0.28 | 2.40 | 0.15 |
| Crossproduct | 3 | 0.79 | 0.04 | 0.37 | 0.78 |
| Total Model | 9 | 13.75 | 0.73 | 2.13 | 0.17 |
| <i>Hue</i> | | | | | |
| Linear | 3 | 115.78 | 0.82 | 24.23 | 0.0005 |
| Quadratic | 3 | 8.22 | 0.06 | 1.72 | 0.25 |
| Crossproduct | 3 | 5.45 | 0.04 | 1.14 | 0.40 |
| Total Model | 9 | 129.45 | 0.92 | 9.03 | 0.004 |
| <i>Chroma</i> | | | | | |
| Linear | 3 | 24.58 | 0.64 | 9.78 | 0.007 |
| Quadratic | 3 | 6.29 | 0.16 | 2.50 | 0.14 |
| Crossproduct | 3 | 1.59 | 0.04 | 0.63 | 0.62 |
| Total Model | 9 | 32.46 | 0.85 | 4.30 | 0.03 |
| <i>ΔE</i> | | | | | |
| Linear | 3 | 4.42 | 0.12 | 1.73 | 0.25 |
| Quadratic | 3 | 25.46 | 0.67 | 9.98 | 0.006 |
| Crossproduct | 3 | 2.27 | 0.06 | 0.89 | 0.49 |
| Total Model | 9 | 32.14 | 0.84 | 4.20 | 0.04 |

Table A3. Analysis of variance of the regression parameters of the predicted response surface quadratic models for the moisture, water absorbance index, water solubility index, and glass transition temperature of spray dried sweetpotato powder

| Regression | DF | Type 1 Sum of Squares | R-Square | F Value | p-value |
|-----------------|----|-----------------------------|----------|---------|---------|
| <i>Moisture</i> | | | | | |
| Linear | 3 | 4.85 | 0.83 | 21.35 | 0.0007 |
| Quadratic | 3 | 0.21 | 0.04 | 0.93 | 0.48 |
| Crossproduct | 3 | 0.27 | 0.05 | 1.17 | 0.39 |
| Total Model | 9 | 5.32 | 0.91 | 7.81 | 0.006 |
| <i>WAI</i> | | | | | |
| Linear | 3 | 26630 | 0.81 | 21.22 | 0.0007 |
| Quadratic | 3 | 1553 | 0.05 | 1.24 | 0.37 |
| Crossproduct | 3 | 1676 | 0.05 | 1.34 | 0.34 |
| Total Model | 9 | 29860 | 0.91 | 7.93 | 0.006 |
| <i>WSI</i> | | | | | |
| Linear | 3 | 385.07 | 0.31 | 2.27 | 0.17 |
| Quadratic | 3 | 371.67 | 0.31 | 2.19 | 0.18 |
| Crossproduct | 3 | 56.75 | 0.05 | 0.33 | 0.80 |
| Total Model | 9 | 813.49 | 0.67 | 1.60 | 0.27 |
| <i>Tg</i> | | | | | |
| Linear | 3 | 299.04 | 0.45 | 7.44 | 0.01 |
| Quadratic | 3 | 122.79 | 0.19 | 3.06 | 0.10 |
| Crossproduct | 3 | 144.90 | 0.22 | 3.61 | 0.07 |
| Total Model | 9 | 566.74 | 0.86 | 4.70 | 0.03 |

Table A4. Analysis of variance of the regression parameters of the predicted response surface quadratic models for the bulk density and mean particle diameter of spray dried sweetpotato powder

| Regression | DF | Type 1 Sum of Squares | R- Square | F Value | p-value |
|---------------------------------|----|-----------------------------|--------------|---------|---------|
| <i>Bulk Density</i> | | | | | |
| Linear | 3 | 0.03 | 0.42 | 4.10 | 0.06 |
| Quadratic | 3 | 0.007 | 0.09 | 0.90 | 0.49 |
| Crossproduct | 3 | 0.02 | 0.25 | 2.51 | 0.14 |
| Total Model | 9 | 0.05 | 0.76 | 2.50 | 0.12 |
| <i>Particle Diameter</i> | | | | | |
| <i>Mean Diameter - Volume</i> | | | | | |
| Linear | 3 | 3905 | 0.36 | 2.93 | 0.14 |
| Quadratic | 3 | 1157 | 0.11 | 0.87 | 0.52 |
| Crossproduct | 3 | 3564 | 0.33 | 2.68 | 0.16 |
| Total Model | 9 | 8626 | 0.80 | 2.16 | 0.21 |
| <i>Mean Diameter - Number</i> | | | | | |
| Linear | 3 | 8.30 | 0.53 | 6.33 | 0.04 |
| Quadratic | 3 | 2.21 | 0.14 | 1.69 | 0.28 |
| Crossproduct | 3 | 3.10 | 0.20 | 2.37 | 0.19 |
| Total Model | 9 | 13.61 | 0.86 | 3.46 | 0.09 |
| <i>Mean Diameter - Area</i> | | | | | |
| Linear | 3 | 208.13 | 0.62 | 24.32 | 0.0002 |
| Quadratic | 3 | 85.16 | 0.26 | 9.95 | 0.01 |
| Crossproduct | 3 | 25.51 | 0.08 | 2.98 | 0.14 |
| Total Model | 9 | 318.80 | 0.96 | 12.42 | 0.006 |
| <i>Median Diameter - Volume</i> | | | | | |
| Linear | 3 | 763.21 | 0.59 | 15.42 | 0.006 |
| Quadratic | 3 | 286.34 | 0.22 | 5.79 | 0.04 |
| Crossproduct | 3 | 161.25 | 0.12 | 3.26 | 0.12 |
| Total Model | 9 | 1210.81 | 0.94 | 8.26 | 0.02 |

APPENDIX B

Tables of the analysis of variance for the response surface quadratic model for each physicochemical characteristic

Table B1. Analysis of variance for the response surface model for color values of spray dried sweetpotato powder

| Source | DF* | Sum of Squares | Mean Square | F-Value |
|-------------|-----|----------------|-------------|---------|
| <i>L*</i> | | | | |
| Lack of Fit | 11 | 8.83 | 0.75 | 6.56** |
| Pure Error | 2 | 0.34 | 0.17 | |
| Total Error | 13 | 9.16 | 0.92 | |
| <i>a*</i> | | | | |
| Lack of Fit | 8 | 2.39 | 0.21 | 7.83** |
| Pure Error | 2 | 0.08 | 0.04 | |
| Total Error | 10 | 2.46 | 0.25 | |
| <i>b*</i> | | | | |
| Lack of Fit | 8 | 5.13 | 0.36 | 3.45** |
| Pure Error | 2 | 0.37 | 0.19 | |
| Total Error | 10 | 5.50 | 0.55 | |
| <i>Hue</i> | | | | |
| Lack of Fit | 8 | 4.74 | 0.42 | 10.53** |
| Pure Error | 2 | 0.11 | 0.06 | |
| Total Error | 10 | 4.85 | 0.48 | |

*Degrees of Freedom

** Not significant at the 95% significance level

Table B2. Analysis of variance for the response surface model for color values of the reconstituted powder solutions

| Source | DF* | Sum of Squares | Mean Square | F-Value |
|----------------|-----|----------------|-------------|---------|
| <i>a*</i> | | | | |
| Lack of Fit | 11 | 14.04 | 0.75 | 3.31** |
| Pure Error | 2 | 0.77 | 0.39 | |
| Total Error | 13 | 14.81 | 1.14 | |
| <i>b*</i> | | | | |
| Lack of Fit | 11 | 10.68 | 0.69 | 6.47** |
| Pure Error | 2 | 0.30 | 0.15 | |
| Total Error | 13 | 10.98 | 0.84 | |
| <i>Hue</i> | | | | |
| Lack of Fit | 11 | 23.29 | 1.14 | 2.76** |
| Pure Error | 2 | 1.53 | 0.77 | |
| Total Error | 13 | 24.82 | 1.91 | |
| <i>Delta E</i> | | | | |
| Lack of Fit | 11 | 31.69 | 2.19 | 9.98** |
| Pure Error | 2 | 0.58 | 0.29 | |
| Total Error | 13 | 32.27 | 2.48 | |

*Degrees of Freedom

** Not significant at the 95% significance level

Table B3. Analysis of variance for the response surface model for moisture content, WAI, Tg, and bulk density of spray dried sweetpotato powder

| Source | DF [*] | Sum of Squares | Mean Square | F-Value |
|-------------------------|-----------------|----------------|-------------|---------------------|
| <i>Moisture Content</i> | | | | |
| Lack of Fit | 11 | 0.99 | 0.07 | 13.46 ^{**} |
| Pure Error | 2 | 0.01 | 0.01 | |
| Total Error | 13 | 1.01 | 0.08 | |
| <i>WAI</i> | | | | |
| Lack of Fit | 11 | 5774.09 | 281.70 | 2.73 ^{**} |
| Pure Error | 2 | 384.00 | 192.00 | |
| Total Error | 13 | 6158.09 | 473.70 | |
| <i>Tg</i> | | | | |
| Lack of Fit | 11 | 353.71 | 23.94 | 8.32 ^{**} |
| Pure Error | 2 | 7.73 | 3.87 | |
| Total Error | 13 | 361.45 | 27.80 | |
| <i>Bulk Density</i> | | | | |
| Lack of Fit | 11 | 0.04 | 0.002 | 3.24 ^{**} |
| Pure Error | 2 | 0.002 | 0.001 | |
| Total Error | 13 | 0.04 | 0.003 | |

^{*}Degrees of Freedom

^{**}Not significant at the 95% significance level

CHAPTER 4

NUTRITIONAL AND RHEOLOGICAL CHARACTERIZATION OF SPRAY DRIED SWEETPOTATO POWDER

4.1 – ABSTRACT

Processing of sweetpotatoes into powder form is an approach to convert this highly nutritious vegetable into a functional ingredient. Previous studies revealed that spray drying of sweetpotato puree is technically feasible using a pre-treatment of alpha-amylase to reduce puree viscosity and the addition of maltodextrin to facilitate drying. To better determine potential applications of powders produced with various levels of amylase and maltodextrin, nutrient composition and rheological properties of the powders in solution were examined and compared to sweetpotato puree. Analysis on proximate composition, beta-carotene, vitamin C, and minerals was performed. Apparent viscosity and flow behavior of reconstituted powder solutions were also evaluated at different temperatures and shear rates. Spray drying significantly reduced the amount of beta-carotene and ascorbic acid in the final product. Additionally, the *all-trans* form of beta-carotene was further transformed to the *cis*-isomer during dehydration. Addition of maltodextrin increased the amount of non-sweetpotato solids in the powders thus reducing overall nutrient content. The viscosity of the reconstituted solutions was much lower than that of the puree at the same solid concentration. However, the reconstituted sweetpotato slurries did behave similarly to pregelatinized starch solutions. Thus, spray dried sweetpotato powders have the potential to enhance food systems as a thickener despite the need for increased nutrient retention.

4.2 – INTRODUCTION

Sweetpotatoes are highly nutritious vegetables; however, sweetpotato consumption is progressively declining especially in industrialized nations. Part of this decline may be attributed to the lack of uses for sweetpotatoes for consumers beyond the raw roots. Conversely, the white potato has seen a great increase in production and a large portion of this increase is due to processed products such as chips, fries, and frozen products. Thus, one way to expand sweetpotato consumption is to develop appealing processed products or alternative uses for sweetpotato roots (Kays 1985, Collins & Walter 1992).

One approach intended to increase consumption is to convert sweetpotato puree into dried powder to be used as a functional ingredient in food systems. Sweetpotatoes have been commercially dried into dehydrated flakes for many years. However, the resulting product has poor solubility, unattractive brown color, and limited applications. In general, fruit and vegetable powders have been described as value added ingredients in various food systems. Both freeze dried and drum dried fruit and vegetable powders deliver numerous functional and nutritional benefits. The characteristic flavors, colors, and nutrients as well as water binding properties of these powders make them an ideal addition to soups, sauces, marinades, baby foods, dips, extruded cereal products, fruit purees for confections, and fillings for frozen toaster snacks (Francis & Phelps 2003, Pszczola 2003).

Previous studies (Chapter 3) have demonstrated that sweetpotatoes can be spray dried into an appealing, functional product. For successful spray drying of

sweetpotato puree, alpha-amylase action was used as pre-drying treatment to reduce viscosity. Amylase reduces the viscosity of sweetpotato puree by hydrolyzing starch molecules to dextrans (Ice et al. 1980, Szyperski et al. 1986). Additionally, maltodextrin was added to the puree in various concentrations to act as a drying aid. Maltodextrin facilitated product recovery by raising the glass transition temperature of the product to reduce stickiness and partially encapsulating the material (Bhandari 1999, Re 1998). Both amylase addition and the use of maltodextrin produced a quality powder with appealing physicochemical characteristics.

Spray dried sweetpotato powders have the potential to be added to various food systems to provide a variety of benefits and functionality. In order to better determine the role of sweetpotato powder in different food applications, additional information about the nutrient composition and the functionality in reconstituted solutions of these powders is required. Sweetpotatoes are known as a rich source of carbohydrates, beta-carotene, ascorbic acid, and minerals. Unfortunately, many of these nutrients are lost or altered during thermal processing. In the production of dehydrated sweetpotato flakes, over 20% of the beta-carotene is lost while ascorbic acid losses have been reported between 50 and 70% (Chandler & Schwartz 1988, Arthur & McLemore 1955). Carbohydrate content also changes during processing as the action of amylase at elevated temperatures transforms the sweetpotato starch to dextrans and sugars (Walter et al. 1975). These chemical conversions will also lead to altered functionality and changes in the flow behavior of reconstituted powder solutions.

Further testing is required to determine the effect spray drying as well as amylase treatment and maltodextrin addition on nutrient composition and reconstituted powder solution viscosity. Significant retention of sweetpotato nutrients during processing is an ideal characteristic of a functional ingredient. Additionally, knowledge of the rheological properties of the reconstituted powders would be helpful in determination of potential product applications. Thus, the objective of this study was to compare the nutrient composition and rheological properties of sweetpotato powders spray dried under different conditions with sweetpotato puree.

4.3 – MATERIALS AND METHODS

4.3.1 – Materials

Sweetpotato puree from the Beauregard cultivar, an orange-fleshed variety, was manufactured by Bright Harvest Sweet Potato Co. (Clarksville, AK). The puree with 18.2% solids was procured in 20kg 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 and 60°C. Maltodextrin (MD) with a dextrose equivalent (DE) of 11 (MD 01960) was obtained from Cargill Inc. (Cedar Rapids, IA). Other chemicals used in experimentation were of analytical and HPLC grades.

4.3.2 – Spray dried powder production

Sweetpotato puree was spray dried using on a pilot scale dryer (Production Minor Spray Dryer, Niro Inc., Columbia, MD) equipped with a rotary atomizer set at 20,000rpm and co-current air-product flow. Drying air was heated using natural gas combustion. Feed was moved into the dryer using a progressive cavity pump (Metering Pump, Moyno, Inc., Springfield, OH) and outlet temperature was maintained by adjusting the feed rate of product into the dryer. A cyclone was used to separate the powder from the air and the powder was recovered at the bottom of the cyclone. Sweetpotato puree was spray dried with various pre-drying treatments using alpha-amylase action and maltodextrin addition and the resulting powders were compared to sweetpotato puree (Table 1).

Feed temperature (60°C), solids content (18.2%), inlet temperature (190°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 alpha-amylase (3.75ml/kg puree, 85.9 APA amylase units/kg puree) was allowed to act for 30 minutes at 60°C before the temperature of the puree was raised above 90°C to deactivate the enzyme. Maltodextrin was added to the puree and mixed until evenly distributed. In order to maintain the same amount of solids being fed to the dryer, water was added to the puree-maltodextrin mixture. For the combined amylase and maltodextrin treatment, the puree was treated with amylase and then the enzyme was inactivated. Maltodextrin was mixed with the treated puree just prior to spray drying. Each treatment was spray dried in duplicate and chemical and

rheological analyses performed on each replicate and the sweetpotato puree. All samples were stored at -20°C until analyzed.

4.3.3 – Nutrient Analysis

Moisture Content

Moisture content was determined by Karl-Fischer titration method using a Karl Fisher 701 Titrino (Metrohm Ltd., Herisau, Switzerland). Between 0.25 and 0.30g of spray dried powder were added to the system. Methanol was used as the solvent and Hydranal composite 5 was the reactant. The Karl Fisher unit was enclosed in a plexiglass dry box with compressed air pumped in to maintain low humidity. The extraction time was set at 180 seconds to enable the sample to fully dissolve. The moisture content of the puree samples was determined by drying a 5g sample in an oven at 70°C for 6 hours and then at 105°C overnight. All moisture measurements were performed in triplicate.

Sugar

Powder samples (100mg) were mixed with 5.0ml of 95% ethanol in a centrifuge tube. The tube was then incubated at 80-85°C for 10 minutes. After incubation, the contents were mixed on a vortex stirrer and another 5ml of ethanol were added to the tube. Next, the tube was centrifuged for 10 minutes at 3000rpm and the supernatant was collected. The remaining pellet was resuspended in 10ml of ethanol, stirred on a vortex mixer, and centrifuged to collect the supernatant. The sugar extracts were combined in a 25ml volumetric flask and filled to volume with ethanol. A 5ml aliquot of this solution was transferred to a small beaker and the

ethanol was allowed to evaporate off overnight. In preparation for High Performance Liquid Chromatography (HPLC), 1ml of the internal standard (cellobiose solution) was added to the beaker to dissolve the residue. Fifty microliters of this solution was diluted to 2ml with water in small test tube. Sugar analyses were conducted using a Dionex BioLC AD 50 HPLC system (Sunnyvale, CA). Ten microliter samples were injected and eluted through a Carbo PAC PA-1 column (250 x 4.6mm id) (Dionex Corporation, Sunnyvale, CA) at 30°C. The mobile phase consisted of 200mM sodium hydroxide at an isocratic flow rate of 1.0ml/minute. Peaks were detected by a Dionex PAD (pulse amperometric detector) and identified based on retention time. To determine sugar content, the peak heights of sucrose, glucose, fructose, and maltose were compared to that of the standard solution of cellobiose (Pattee et al. 2000).

Starch

Starch content was determined using an assay kit (Megazyme International Ltd, Bray, Co. Wicklow, Ireland) according to AOAC Method 996.11(AOAC 1995). Powder samples were first washed with ethanol to remove the sugars as described above. The remaining pellet was then treated with 2ml of dimethyl sulfoxide at 100°C to account for resistant starch. The samples were cooked with thermostable alpha amylase to partially hydrolyze and solubilize the starch. Subsequently, the samples were treated with amyloglucosidase for 30 minutes at 50°C to hydrolyze the starch dextrans to glucose. The samples were then transferred to 100ml volumetric flasks and filled to volume with distilled water. An aliquot of this solution was centrifuged at 3000rpm for 10 minutes and the supernatant transferred to a glass

centrifuge tube. The solution was mixed with a glucose determination reagent and incubated at 50°C for 20 minutes. The absorbance of the solution at 510nm was read on a spectrophotometer (Cary 300 UV-Visible Spectrophotometer, Varian Inc., Palo Alto, CA) against a reagent blank. Starch content was calculated based on the absorbance of the sample with reference to a glucose standard.

Fiber

Fiber content was determined using the methods as outlined in AOAC Methods 991.43 and 985.29 (AOAC 1995) using an assay kit [Megazyme International Ltd, (Bray, Co. Wicklow, Ireland)]. Total dietary fiber was determined on duplicate samples of the spray dried powders and freeze dried puree. Samples were first cooked at ~100°C with heat-stable alpha-amylase to induce gelatinization, hydrolysis, and depolymerization of starch. Subsequently, samples were incubated at 60°C with protease to solubilize and depolymerize proteins and amyloglucosidase to hydrolyze starch fragments. Next, each sample was treated with ethanol to precipitate soluble fiber and remove depolymerized protein and glucose. The residue was then filtered; washed with 78% ethanol, 95% ethanol, and acetone; dried; and weighed. One of the duplicate samples was analyzed for protein and the other used to determine ash content. The total dietary fiber was calculated as the weight of the dried residue less the weight of the protein and ash.

Protein, Fat, Ash

Protein, fat, and ash content determination of the sweetpotato puree and spray dried powders was performed by Silliker Laboratories (Stone Mountain, GA). Protein content was measured using Kjeldahl analysis using a protein factor of 6.25

according to AOAC Method 991.20.1. Fat content was determined using AOAC Method 933.05 and ash content by AOAC Method 925.51A (AOAC 1995).

Carotene

Beta-carotene was extracted from the puree and powder and analyzed using a HPLC system. For extraction, 5g of sample was mixed with approximately 2g of calcium carbonate, 1g of diatomaceous earth, and 25ml of methanol. Next, 50ml of a hexanes-acetone (1:1) mixture were added and stirred. The mixture was drawn under vacuum through a funnel with a fritted disk. The residue in the funnel was washed with 25ml of methanol and 50ml of the hexanes-acetone mixture two additional times or until the filter cake was colorless. The extract was transferred to a 250ml separatory funnel and washed with water. A few drops of saturated sodium chloride solution were added to the funnel to facilitate sharp delineation of the phases. The aqueous phase was released and the upper layer was transferred to a 50ml volumetric flask and filled to volume with hexane (Chandler & Schwartz 1988). Samples were stored in dark vials until HPLC testing.

HPLC analysis of the carotene content was performed according to the method of Yeum et al. (1996). The HPLC system consisted of a series 410 LC pump (Perkin Elmer, Norwalk, CT), a Waters 717 plus autosampler (Millipore, Milford, MA), a C30 carotenoid column (3 μ m, 150 X 4.6mm, YMC, Wilmington, NC), a column temperature controller (model 7950; column heater/chiller, Jones Chromatography, Lakewood, CO), a Waters 994 photodiode array detector, and a Waters 840 digital 350 data station. The mobile phase consisted of methanol:methyl-tert-butyl and ether:water. The eluent flow rate was 1ml/minute at

16°C. Peak identification was based on the retention time of respective carotene isomers. Carotenes were quantified by determining peak areas in the chromatograms as compared to known standards.

Vitamin C

Vitamin C activity was quantified by determining the amount of ascorbic and dehydroascorbic acid in the sample using HPLC. Samples (5g) were mixed with 30ml of a 5% metaphosphoric acid solution and centrifuged prior to HPLC analysis. Both ascorbic acid and dehydroascorbic acid were analyzed using a HPLC system (ThermoQuest San Jose, CA) consisting of a P2000 binary pump, AS 3000 autosampler, and SCM 1000 degasser. Samples were placed in the sample tray set at a temperature of 6°C with a light proof covering. 20µl samples were injected onto a 3µm reverse phase column (4.6 x 150mm) (µ Bondapack-NH₂ Z-module cartridge, Waters Associates, Milford, MA) and were separated at 35°C under isocratic conditions with an eluent flow rate of 1.2ml/minute. The mobile phase consisted of aqueous 0.005M KH₂PO₄ and acetonitrile (30:70 v/v). Peaks were monitored at 450nm by a UV 6000 LP Diode Array Detector. Standard solutions with concentrations from 0.5mg/ml to 10mg/ml were used for the calculations. ThermoQuest Chromatography Data Acquisition Software version 4.1 was used to collect and process the data. Results were calculated using the external calibration method. The R² of the standard curve was greater than 0.99 with the intercept forced through zero.

Minerals

Phosphorous, calcium, magnesium, potassium, iron, and sodium content was analyzed by the Department of Soil Science Analytical Services Lab at North Carolina State University. Samples underwent dry combustion and dissolution of the residue in acid. The mineral digest was then analyzed on a Perkin Elmer Ion Coupled Plasma (ICP) Spectrometer (Perkin Elmer Corp, Norwalk, CT). Duplicate analysis was performed on each treatment replication.

4.3.4 – Rheological Testing

Sample Preparation

Powder samples were reconstituted to the same solids content as the puree (18.2%). Five grams of powder were mixed with 27.5g of water in a 50ml test tube using a vortex mixer set on the highest speed for 2 minutes. Samples were allowed to stand for approximately one hour at room temperature before rheological measurements were taken.

Viscosity Determination

Viscosity of the puree was determined using a Stress Tech Rheometer [Reo Logica Instruments (Lund, Sweden)] with a serrated bob and cup geometry. The sample was covered with a thin layer of oil and a cover to help prevent moisture loss. Samples were pre-sheared for 60s at 25s^{-1} and allowed to equilibrate for 25s before testing began. Shear rate sweeps were performed on the samples at 25, 75, and 95°C with shear rate ramped up and down from 1 to 250s^{-1} . Samples were allowed to equilibrate for 60s at the set temperature before the shear rate sweeps

were performed. Two cycles of the shear rate ramp were performed at 75°C to look for different time dependent phenomena. For temperature ramps, puree and reconstituted solutions were sheared at a constant rate of 10s⁻¹ while the temperatures were increased from 25 to 75°C, continued from 75 to 95°C, and then cooled to 25°C at a rate of 1.5°C/minute.

Flow behavior was described by the Herschel-Bulkley (1) and power law models (2)

$$\sigma = \sigma_o + K \dot{\gamma}^n \quad (1)$$

$$\sigma = K \dot{\gamma}^n \quad (2)$$

where σ =shear stress, $\dot{\gamma}$ =shear rate, σ_o =yield stress, K=consistency coefficient, and n=flow behavior index (Steffe 1996).

4.3.5 – Statistical Analysis

Nutrient composition data was analyzed using the Statistical Analysis System (SAS Institute v.8.0, Cary, NC). Analysis of variance and means separation were calculated by the general linear model procedure. Differences between treatments ($p < 0.05$) were evaluated by the least squares mean procedure with a Tukey adjustment.

4.4 – RESULTS AND DISCUSSION

4.4.1 – Nutrient Composition

Proximate Analysis

Results of proximate analysis on sweetpotato puree and spray dried powders are found in Table 2. All powders were dried to an acceptable level below 5% moisture content and no differences in moisture content existed between spray drying treatments.

Starch content was higher in the spray dried samples containing maltodextrin (Table 2). The higher starch value in the maltodextrin samples can be attributed to the added maltodextrin, a starch hydrolyzate. In the starch testing method, the enzymes that are utilized to breakdown starch to glucose can also degrade maltodextrin to glucose. Since starch content is calculated from the absorbance of glucose, these samples would appear to have higher starch content. Typically, sweetpotato roots have approximately 30-70% starch content on dry basis (db) (Woolfe 1992). However, when sweetpotatoes are heated, the natural amylases become active and degrade the starch molecules into dextrans and sugars until the enzymes are inactivated. Walter et al. (1976) reported a decrease in starch concentration and increase in maltose and dextrans in dehydrated sweetpotato flakes as the length of time the puree was exposed to enzyme activity prior to drying was increased. Truong et al. (1986) reported a 4 -14% decrease in starch content when sweetpotatoes were cooked for 30 minutes. Arthur and McLemore (1955) reported

starch values of 33-43% in the raw roots which decreased to between 2-10% (db) in dehydrated flakes. Thus, the starch content was expected to be lower in spray dried powder pretreated with amylase for viscosity reduction. The amylase treatment does indeed have the lowest starch content, but this treatment does not significantly reduce the starch compared to the control powder. Total sugar content of the spray dried powders was similar to the values reported for dehydrated flakes (32-45% (db)) (Arthur & McLemore 1955). Each powder sample had a higher sugar content compared to the puree except for the maltodextrin-treated sample where the total sugar content was diluted by the addition of maltodextrin.

The addition of maltodextrin increased the amount of non-sweetpotato solids in the powder samples which in turn lowered the fiber, protein, and ash content of the maltodextrin-added powders. The control and amylase-treated samples were not significantly different than the puree for these components. Raw sweetpotatoes contain approximately 2-10% fiber (db) (Huang et al. 1999, Woolfe 1992, Purcell et al. 1989). The fiber content of the powder samples ranges from 3.5 to 7.8% and was lower, but not significantly different, than the fiber content of the puree except for the aforementioned added maltodextrin differences.

Protein values of the spray dried powders were also lower than that of the puree. The crude protein contents of sweetpotato roots has been reported between 1.2 -10% including a considerable amount of the biologically active amino acid, lysine. However, heat processing treatments such as canning and flaking have been related to a reduction in protein and amino acid content (Walter et al. 1984). Thus, the lower protein content of the powders could be attributed to the destruction

of lysine through interaction with the reducing groups of carbohydrates at high temperatures. The Maillard reaction is a complex set of reactions initiated by reaction of an amine group of a protein and a carbonyl group of a reducing sugar at elevated temperatures. During these reactions, lysine becomes biologically unavailable and also may further degrade by participating in the Strecker degradation reaction (Damodaran 1996).

The lipid content of the powders was significantly lower than that of the puree. Lipid oxidation of drum dried sweetpotato flakes has been a common problem (Walter & Purcell 1974). Similarly, greatly increasing the surface area through spray drying exposes more area for oxidation and degradation to take place thus reducing the lipid concentration.

Vitamin C & Minerals

Vitamin C activity of the puree was significantly reduced by spray drying (Table 3). Again, the addition of maltodextrin decreased the overall sweetpotato solids and thus the amount of vitamin C in those powders. Both ascorbic acid (AA) and dehydroascorbic acid (DHAA), the oxidized form of AA, contribute to vitamin C activity. However, if DHAA is further hydrolyzed to 2,3-diketogulonic acid, then vitamin C activity is lost. The rate of DHAA hydrolysis markedly increases with increasing temperature (Gregory 1996). Raw sweetpotatoes contain between 17 to 35 mg/100g of vitamin C on a fresh weight basis (fwb) (Woolfe 1992, Purcell et al. 1989, Bradbury & Singh 1986). However, these levels are significantly reduced by thermal processing. Bradbury and Singh (1986) reported baking sweetpotatoes for 30 minutes reduced AA and DHA by 45 to 55%. Boiling sweetpotatoes for less than

20 minutes decreased levels of AA and initially increased DHA. After 20 minutes, DHA concentration also began to decrease. A 50 to 70% decrease in vitamin C was reported for sweetpotato flakes drum dried at high temperatures. Sweetpotato flakes were reported to have ascorbic acid levels between 55 and 94mg/100g (db) with initial root levels of 103-105mg/100g (db) (Arthur & McLemore 1955). Bradbury and Singh (1986) reported levels of 48.6 and 23.8 mg/100g (db) for AA and DHAA in a representative cultivar from the South Pacific and levels of 10.8 and 12.6mg/100g (db) after cooking. The vitamin C values for the spray dried powders and the puree in the current study are much lower than these previously reported values. In addition to thermal treatments, some oxidation and hydrolysis over storage time could account for these low values.

Raw sweetpotatoes contain 10 to 64mg phosphorous (P); 110 to 403mg potassium (K); 20 to 41mg calcium (Ca); 10 to 22mg magnesium (Mg); 0.59 to 0.86mg iron (Fe); and 13 to 30mg sodium (Na) per 100g on a fresh weight basis (Picha 1985, Woolfe 1992). Processed sweetpotato products such as puree and dried powders have significantly higher levels of these minerals (Table 3). However, mineral content of the spray dried powders was significantly lower than that of the puree. Levels of the Dietary Reference Intakes for adults are also listed in Table 3. One hundred grams of sweetpotato powders provide between 14 and 28% of the DRI for magnesium and 20 to 39% for potassium. Levels of the other minerals fall below 10% of the DRI for these minerals.

Beta-carotene

Values for beta-carotene content of the sweetpotato puree and spray dried powders are listed in Table 4. Spray drying sweetpotato puree significantly decreased total amount of beta-carotene and caused further isomerization of the molecule. Carotenoids are easily isomerized by heat, acid, or light. In general, carotenoids exist in an *all trans* configuration. When exposed to heat, the molecule may transform to a *cis* configuration typically at the 9, 13, and 15 carbon positions. Also, dehydration processes may contribute to oxidative degradation due to exposure to oxygen and an increased surface to mass ratio (von Elbe & Schwartz 1996).

Raw sweetpotatoes contain between 0.5 and 45mg/100g (db) of beta-carotene (Kays 1992, Lessin et al. 1997, Purcell & Walter 1968). The original levels of beta-carotene can be reduced during the thermal processing of sweetpotatoes such as pureeing and dehydration (Table 4). In previous works, canning and pureeing actually showed an increase in total beta-carotene content but this increase was attributed to enhanced extraction efficiency (Chandler & Schwartz 1988, Lessin et al. 1997). Drum drying however, led to a loss in total beta-carotene content (Arthur & McLemore 1955).

Depending on the severity of the heat treatment, the *all-trans*-beta-carotene is more prone to isomerization than to degradation (Chandler & Schwartz 1988). As shown in Table 4, the puree in this study had 11.2% *cis* isomers while isomers in the spray dried powders ranged from 8.7 to 42%. Chandler and Schwartz (1988)

reported a very slight increase in *cis* isomers in the pureed product. However, drum drying showed a 20.5% loss in total beta-carotene and a 24% increase in *cis* isomers. Puree had total beta-carotene content of 48.6mg/100g (db) with 5.2% *cis* isomers while the drum dried flakes had total beta-carotene content of 35.0mg/100g (db) with 28.9% *cis* isomers. Most of these isomers existed at the 13 carbon position with only trace amounts at the 9 carbon position. Similarly, the results of the current study showed the greatest amount of isomerization at the 13 carbon (Table 4).

The beta-carotene content of the spray dried samples was much lower than the previously reported values for dehydrated flakes. Oxidation during extended storage (2-4 months) prior to testing may have contributed to additional losses in beta-carotene. Wagner and Warthesen (1995) reported degradation of beta-carotene during storage follows first order kinetics and that exposure to air accelerated the degradation.

Samples with added maltodextrin had the lowest amount of beta-carotene content as maltodextrin makes up a significant percent of the total solids content of the powder. These samples surprisingly also had an increased amount of isomerization and loss of beta-carotene as seen in Table 4 and the representative chromatograms (Figure 1). Maltodextrin is often used as an encapsulating agent to protect sensitive ingredients from the environment by form a coating or “wall” around the material. Previous researchers demonstrated the use of maltodextrin in reducing oxidative degradation of carotenes during storage depending on the concentration of maltodextrin, the level of hydrolysis of the maltodextrin, and drying technique

(Wagner & Warthesen 1995, Desobry et al. 1997). Therefore, the maltodextrin treated samples were expected to retain a higher percentage of beta-carotene. Despite the protective effects of maltodextrin however, Desobry et al. (1997) reported an 11% loss in beta-carotene during the spray drying of pure beta-carotene. Further, beta-carotene could have been oxidized during the storage time between powder production and testing. Wagner and Warthesen (1995) reported that maltodextrins with a higher dextrose equivalent (DE), or degree of hydrolysis gave better oxidative protection than lower DE maltodextrins. The maltodextrin with DE=11 used in this study may not have sufficiently protected the beta-carotene from oxidation during the storage time prior to testing.

Beta-carotene pigments impart cream, yellow, orange, and deep orange color in sweetpotato roots (Woolfe 1992). Previous researchers have found a correlation between Hunter color values and the beta-carotene content in sweetpotatoes. Simonne et al. (1993) reported a positive linear relationships between a and b values and beta-carotene while a negative linear relationship between beta-carotene and L value and hue angle was observed. In a previous study on spray dried sweetpotato powders (Chapter 3), similar results were noted. As beta-carotene decreased with spray drying treatment, L* values and hue angle increased while a* and b* values decreased.

Despite the significant decrease in total beta-carotene content, the spray dried sweetpotato powders may still contribute to vitamin A in the human diet. The Recommended Dietary Allowance (RDA) for vitamin A is 0.6-0.9mg/day which translates to approximately 7 – 10.8mg of beta-carotene (Food & Nutrition

Information Board 2004). Consuming 100g of the control and amylase-treated powders could significantly contribute to vitamin A activity.

4.4.2 – Rheological Properties

Shear rate ramps were performed at 25, 75, and 95°C to investigate shear and temperature effects on the apparent viscosity of 4 spray dried powders reconstituted to the same solid concentration (18.2%) as the original puree. Figure 2 shows a representative rheogram of the puree and the reconstituted powder solutions at 25°C while Table 5 summarizes model parameters and viscosities at 10, 50, and 100s⁻¹. The viscosity of all samples decreased with increasing temperature. At all temperatures, the viscosity of the puree was an order of magnitude greater than all of the powder solutions. The solutions of the control and amylase-treated samples had similar viscosities which were both greater than the samples containing maltodextrin (Figure 2).

The flow behavior of the puree was best fit to Herschel-Bulkley model showing pseudoplastic behavior with a yield stress while most of the powders did not have a yield stress and fit the power law model. For the puree, consistency coefficients (K) ranged from 36 to 61 Pa·sⁿ and flow behavior indexes (n) were between 0.15 and 0.36. These K values are slightly higher than those reported by other researchers for sweetpotato puree while the n values are in the same range. Kyereme (1999) reported consistency coefficients between 2.8 and 21.5 Pa·s and flow behavior indexes between 0.20 and 0.33 for sweetpotato puree measured at temperatures between 10 and 90°C. Rao et al. (1975) reported similar consistency

coefficient values between 1.79-24.8 Pa-s while flow behavior index values were slightly higher (0.33- 0.56) for seven different cultivars. The yield stress for the puree in this study was similar to those reported by Rao et al. (1975) (23 – 66.3 Pa) but much higher than the yield stress of 10 Pa reported by Kyereme (1999). The powder solutions had much lower consistency coefficient values (0.2 – 1.1 Pa-sⁿ) and higher flow behavior index values (0.21 – 0.81) than the puree. Solutions with maltodextrin had the lowest consistency coefficients and higher, nearing Newtonian (n=1), flow behavior indexes.

The solids concentration in the puree and reconstituted solutions was the same (18.2%); however, the puree viscosity was much greater than the powder viscosity at all temperatures. Abu-Jdayil et al. (2004) reported similar results for tomato paste and spray dried tomato powder solutions. In the tomato products, viscosity differences were attributed to changes to pectins and other long chain carbohydrate polymers found in the tomato paste during spray drying. These polymers are degraded by the heat of spray drying resulting in smaller molecules which affect the colloidal properties of the solution. Additionally, large differences in particle size between the dried tomato powder and tomato paste, which has suspended particles in colloidal serum, accounted for differences in yield stress and viscosity. Similarly for sweetpotatoes, starch molecules are degraded during processing losing the ability to swell and increase viscosity.

The powder samples containing maltodextrin were least viscous. Preliminary testing showed that a 10% maltodextrin solution had a viscosity of less than 0.005Pa-s while the viscosity of a 20% solution was less than 0.006Pa-s. In the

sweetpotato powders, maltodextrin comprised less than 2% of the total solid content and thus maltodextrin did not contribute to increased viscosity. At low concentrations, maltodextrin is known for having superior cold water solubility (BeMiller & Whistler 1996). In the previous study on spray dried sweetpotato puree (Chapter 3), the water solubility index of the powders increased as the amount of maltodextrin added increased. With more of the spray dried powder going into solution, there is less sweetpotato solids to create resistance to flow in the mixture. Additionally, as the maltodextrin encapsulates the sweetpotato material, there are interactions between the maltodextrin and the other polysaccharides present in the powders. This interaction does not allow these polysaccharides to fully extend in solution. Since longer molecules have a larger hydrodynamic volume which increases solution viscosity, the maltodextrin-polysaccharide interaction facilitates a decreased solution viscosity compared to the other samples.

The flow behavior of the spray dried sweetpotato powder solutions was similar to that of pregelatinized starch solutions. All the reconstituted solutions were shear-thinning and displayed slight thixotropy (Figure 3). The hysteresis loops at 75°C were similar for both shear ramp cycles. Additionally, most sweetpotato powder and pregelatinized starch solutions are best described by the power law model (Doublier et al. 1986, Anastasiades et al. 2002). For 8% pregelatinized wheat starch solutions, at a temperature of 60°C and shear rates between 0 and 662s⁻¹, apparent viscosity ranged from 0.99 to 0.02 Pa-s (Doublier et al. 1986). For modified maize starch pastes with solids concentrations between 3.5 and 4.5%, apparent viscosity ranged from 2 to .002 Pa-s over shear rate range of 1 to 200s⁻¹

at 40°C (Anastasiades et al. 2002). The viscosity of the sweetpotato powders at 18.2% solid concentration was in the same range but somewhat lower (0.3 – 0.02 Pa-s). Thus, sweetpotato powders behave similarly to pregelatinized starches in solutions; however, a much higher concentration of sweetpotato powder is required.

Similar to spray dried sweetpotato powders, the pseudoplastic behavior of pregelatinized starch solutions was also demonstrated by a flow behavior index of less than 1. The sweetpotato powders had flow behavior indexes between 0.2 and 0.8. For maize starch solutions with concentrations between 3.5 and 4.5%, the flow behavior index varied from 0.34 to 0.44 (Anastasiades et al. 2002). Flow behavior index values for 5 to 9.5% modified wheat starch solutions were slightly higher between 0.6 and 0.7 at 60°C (Doublier et al. 1986).

Subjecting the puree and spray dried powders to a temperature ramp under a shear rate of 10s^{-1} produced little variation in apparent viscosity (Figure 4). For example, the viscosity of control sample was reduced from 0.34 to 0.133 Pa-s as temperature was ramped from 25 to 95°C. Sample viscosity was higher during cooling from 95 to 25°C. In a study of corn starches modified by acid hydrolysis, Wang et al. (2003), reported that the starch solution transformed from a sol to a weak gel during cooling. While the puree, amylase, and maltodextrin samples behave similarly to the control sample during the temperature ramp, the sample with amylase and maltodextrin treatments displayed different behavior that requires further investigation. As the temperature was increased from 25 to 55°C, the apparent viscosity increased from 0.042 to 0.070 Pa-s. The viscosity then decreases until the temperature reaches 70°C and remains fairly constant (.048Pa-

s) until 95°C. Reducing the temperature from 95 to 25°C for the amylase and maltodextrin sample increases the viscosity from .048 to .098Pa-s similar to the other powder samples. During heating, the interaction between maltodextrin and polysaccharides present in sweetpotatoes may be slightly disrupted thus releasing these long chain molecules into solution and slightly increasing viscosity. Upon further heating, the solution viscosity decreases as elevated temperature weakens molecular interactions.

4.5 – CONCLUSIONS

Sweetpotato nutrients such as beta-carotene and ascorbic acid were significantly reduced during the spray drying of sweetpotato puree. In addition to thermal degradation of components, the addition of maltodextrin as a drying aid diluted the amount of nutrients in the resulting powder. Moreover, maltodextrin greatly increased the dispersibility of powder particles in solution. A decreased level of sweetpotato solids as well as the interaction between maltodextrin and sweetpotato polysaccharides contributed to decreased viscosity. Overall, the flow behavior of sweetpotato powders was different than sweetpotato puree at the same solids concentration due to molecular changes during spray drying. Sweetpotato powders in solution behaved similarly to pregelatinized starch solutions but required higher concentrations for the same effects.

Product development scientists are often looking for ingredients to improve food systems. Sweetpotato powder may have the potential to act as a functional

ingredient for enhancing natural color and flavor and acting as a thickening ingredient like pre-gelatinized starches in food systems. However, the results indicate that the nutrient composition of spray dried sweetpotato powders would have to be improved to make the powders more attractive to product developers and consumers. Thus, the optimal level of maltodextrin required to balance cost effective drying with powder quality will have to be determined. Additionally, further study is required on how to better retain beta-carotene and ascorbic acid during drying and subsequent storage. Further, critical concentrations of sweetpotato powders in solutions should be examined.

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Table 1. Experimental variables for sweetpotato spray drying

| Treatment | Amylase (ml/kg puree) | Maltodextrin (%) |
|------------------------|--------------------------|------------------|
| Puree | NA | NA |
| Control | 0 | 0 |
| Maltodextrin | 0 | 10 |
| Amylase | 3.75 | 0 |
| Amylase & Maltodextrin | 3.75 | 10 |

Table 2. Proximate analysis of sweetpotato puree and spray dried powder

| Treatment | Moisture (%) | Starch | Total Sugar | Total Dietary Fiber % of Dry Matter | Protein | Lipid | Ash |
|------------------------|-------------------|--------------------|----------------------|--|-------------------|-------------------|-------------------|
| Puree | 81.7 ^a | 33.12 ^b | 31.88 ^{b,c} | 11.29 ^a | 6.08 ^a | 2.33 ^a | 3.80 ^a |
| Control | 3.68 ^b | 32.00 ^b | 40.15 ^{a,b} | 7.04 ^{a,b} | 4.84 ^a | 0.62 ^b | 4.31 ^a |
| Maltodextrin | 2.61 ^b | 58.80 ^a | 28.41 ^c | 3.55 ^b | 3.29 ^b | 0.44 ^b | 2.37 ^b |
| Amylase | 3.11 ^b | 26.90 ^b | 42.55 ^a | 7.81 ^{a,b} | 5.02 ^a | 0.55 ^b | 3.90 ^a |
| Amylase & Maltodextrin | 2.51 ^b | 46.90 ^a | 36.19 ^{a,b} | 5.73 ^{a,b} | 3.08 ^b | 0.57 ^b | 2.62 ^b |
| CV | 4.76 | 5.86 | 4.33 | 16.38 | 11.02 | 15.25 | 10.10 |

Different letters within columns indicate a significant difference at $p < 0.05$.

Table 3. Vitamin C and mineral content of sweetpotato puree and spray dried powder (mg/100g) (db)

| Treatment | Vitamin C Activity [*] | P | Ca | Mg | K | Fe | Na |
|--|---------------------------------|--------------------|------------------|-------------------|-------------------|-------------------|--------------------|
| Puree | 5.79 ^a | 848 ^a | 630 ^a | 411 ^a | 7626 ^a | 15.8 ^a | 423 ^a |
| Control | 1.4 ^b | 202 ^b | 151 ^b | 99 ^b | 1822 ^b | 4.79 ^b | 122 ^{b,c} |
| Maltodextrin | 0.88 ^{b,c} | 133.5 ^b | 82 ^b | 61.5 ^b | 1058 ^b | 2.64 ^c | 73.0 ^c |
| Amylase | 1.32 ^{b,c} | 185 ^b | 124 ^b | 87.5 ^b | 1621 ^b | 2.89 ^c | 177 ^b |
| Amylase & Maltodextrin | 0.21 ^c | 123 ^b | 82 ^b | 51 ^b | 980 ^b | 1.76 ^c | 143 ^b |
| CV | 13.53 | 12.77 | 15.84 | 19.44 | 9.38 | 12.03 | 12.16 |
| Dietary Reference Intake ^{**} | 65-90 | 700 - 1250 | 1000 - 1300 | 360 - 420 | 4.7g | 8 - 18 | 1.3 - 1.5g |

Different letters within columns indicate a significant difference at $p < 0.05$.

^{*} Total vitamin C activity as the sum of ascorbic acid and dehydroascorbic acid

^{**} DRI values are in mg/day unless otherwise noted.

Table 4. *Cis/Trans* beta-carotene isomers in sweetpotato puree and spray dried powders (mg/100g) (db)

| Treatment | trans | 9 cis | 13 cis | Total Beta-Carotene Equivalent | % cis isomers |
|------------------------|--------------------|-------------------|---------------------|--------------------------------|---------------|
| Puree | 35.44 ^a | 0.28 ^a | 4.18 ^a | 38.0 ^a | 11.2 |
| Control | 10.54 ^b | 0.32 ^a | 0.68 ^{b,c} | 11.1 ^b | 8.7 |
| Maltodextrin | 0.31 ^c | 0.12 ^a | 0.11 ^c | 0.43 ^c | 42.1 |
| Amylase | 13.18 ^b | 0.35 ^a | 1.11 ^b | 14.0 ^b | 10.0 |
| Amylase & Maltodextrin | 0.815 ^c | 0.04 ^a | 0.12 ^c | 0.90 ^c | 16.4 |
| CV | 16.55 | 39.19 | 16.67 | 15.94 | |

Different letters within columns indicate a significant difference $p < 0.05$.

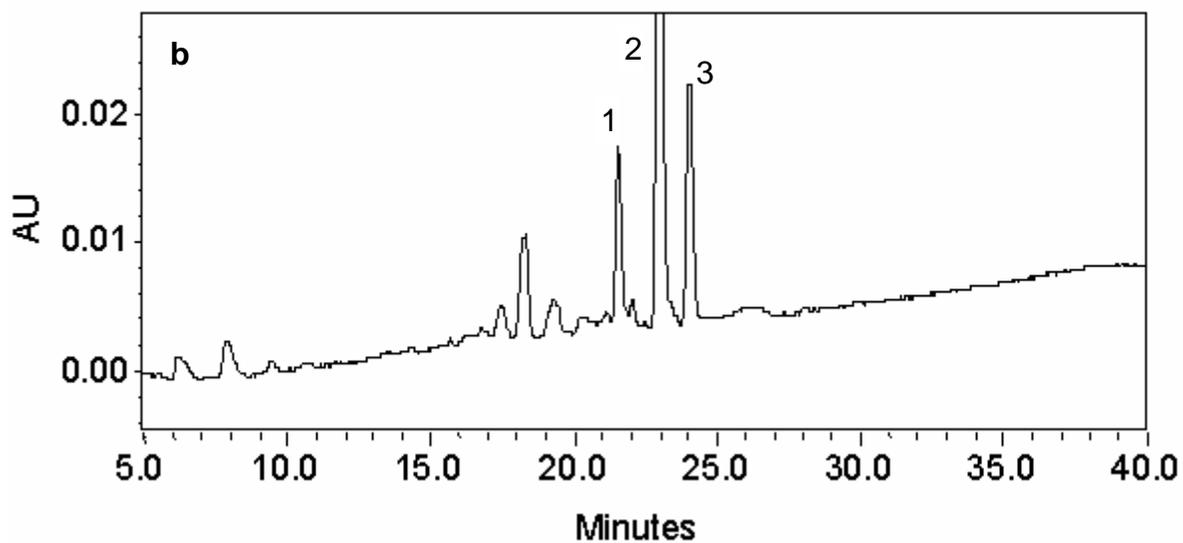
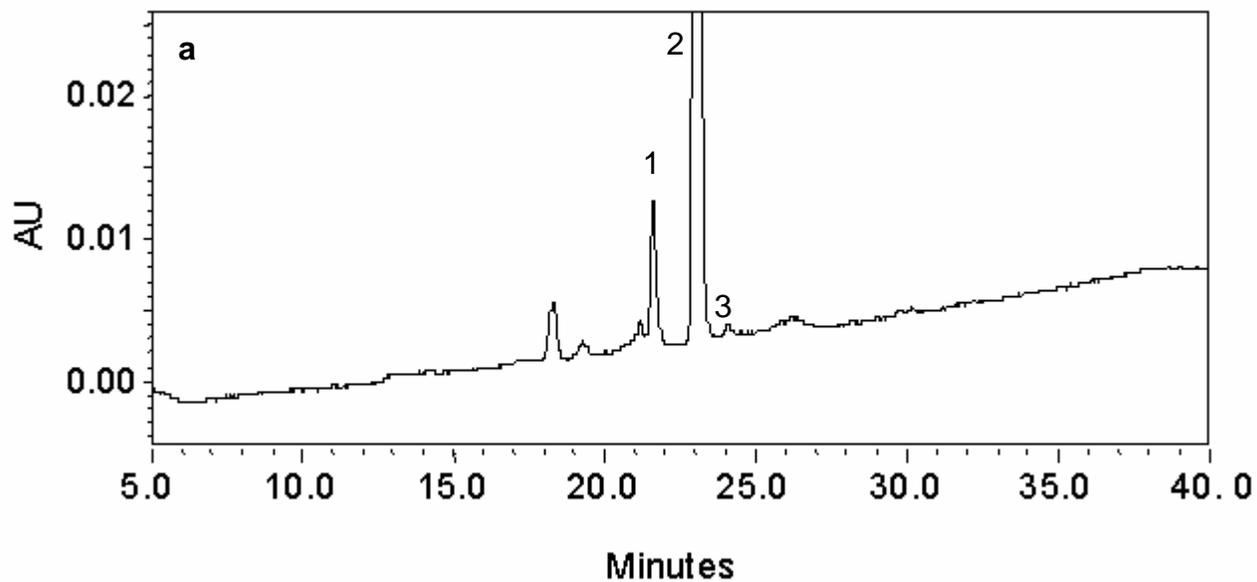


Figure 1. Representative chromatograms of the separation of beta-carotene isomers.
 1 = 13-*cis* 2 = all-*trans* 3 = 9-*cis*
 a.) sweetpotato puree b.) sweetpotato powder spray dried with maltodextrin

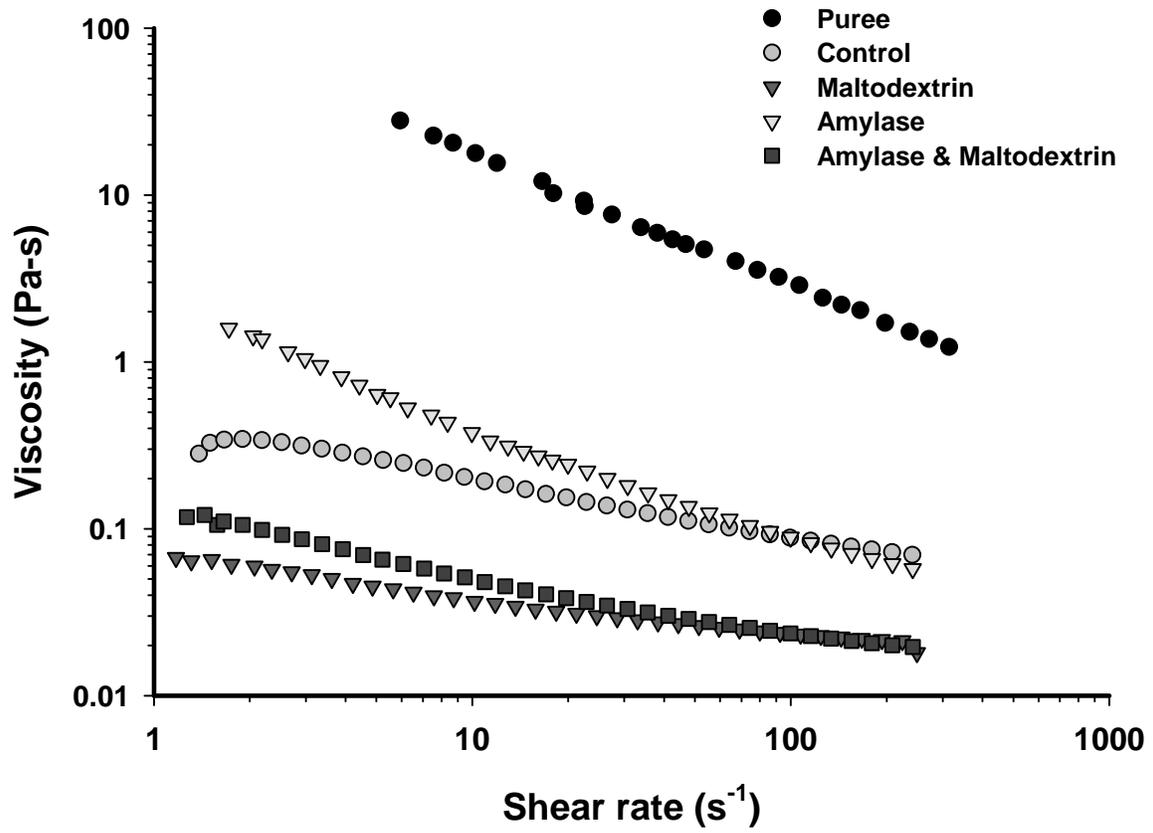


Figure 2. Viscosity of sweetpotato puree and reconstituted powders with 18.2% solids at 25°C sheared from 1 – 250 s^{-1}

Table 5. Herschel-Bulkley (HB) and power law (PL) parameters and viscosities of 18.2% solids sweetpotato puree and reconstituted spray dried powder solutions at 25, 75, and 95°C and at shear rates between 1 and 250s⁻¹.

| Sample | Temperature (°C) | σ_0 (Pa) | K (Pa-s) | n | R ² | η_{10} (Pa-s) | η_{50} (Pa-s) | η_{100} (Pa-s) |
|------------------------|------------------|-----------------|----------------|---------------|----------------|--------------------|--------------------|---------------------|
| Puree | 25 | 93.50 ± 2.12 | 36.92 ± 10.17 | 0.366 ± 0.030 | 0.98 | 17.81 ± 1.88 | 4.92 ± 0.22 | 2.81 ± 0.34 |
| | 75 | 30.00 ± 14.14 | 45.645 ± 2.661 | 0.313 ± 0.021 | 0.99 | 11.40 ± 0.87 | 3.69 ± 0.37 | 2.10 ± 0.23 |
| | 95 | 32.50 ± 17.68 | 61.300 ± 3.30 | 0.145 ± 0.010 | 0.85 | 6.00 ± 0.89 | 1.30 ± 0.20 | 0.764 ± 0.134 |
| Control | 25 | - | 0.403 ± 0.134 | 0.653 ± 0.033 | 0.99 | 0.187 ± 0.056 | 0.104 ± 0.027 | 0.085 ± 0.022 |
| | 75 | - | 0.623 ± 0.325 | 0.211 ± 0.068 | 0.99 | 0.099 ± 0.030 | 0.054 ± 0.016 | 0.043 ± 0.012 |
| | 95 | - | 0.211 ± 0.068 | 0.627 ± 0.013 | 0.99 | 0.081 ± 0.030 | 0.047 ± 0.017 | 0.038 ± 0.014 |
| Maltodextrin | 25 | - | 0.589 ± 0.427 | 0.805 ± 0.133 | 0.99 | 0.037 ± 0.014 | 0.026 ± 0.006 | 0.024 ± 0.004 |
| | 75 | - | 0.589 ± 0.416 | 0.718 ± 0.161 | 0.99 | 0.023 ± 0.013 | 0.014 ± 0.005 | 0.012 ± 0.003 |
| | 95 | - | 0.593 ± 0.414 | 0.725 ± 0.177 | 0.99 | 0.026 ± 0.019 | 0.015 ± 0.009 | 0.013 ± 0.006 |
| Amylase | 25 | - | 1.082 ± 1.006 | 0.350 ± 0.038 | 0.97 | 0.310 ± 0.182 | 0.130 ± 0.084 | 0.089 ± 0.055 |
| | 75 | - | 0.787 ± 0.521 | 0.338 ± 0.012 | 0.97 | 0.211 ± 0.157 | 0.076 ± 0.057 | 0.050 ± 0.036 |
| | 95 | - | 0.710 ± 0.422 | 0.387 ± 0.029 | 0.96 | 0.195 ± 0.166 | 0.065 ± 0.052 | 0.044 ± 0.033 |
| Amylase & Maltodextrin | 25 | - | 0.586 ± 0.375 | 0.647 ± 0.023 | 0.99 | 0.049 ± 0.005 | 0.028 ± 0.002 | 0.024 ± 0.002 |
| | 75 | - | 0.550 ± 0.333 | 0.450 ± 0.011 | 0.98 | 0.058 ± 0.010 | 0.023 ± 0.003 | 0.018 ± 0.002 |
| | 95 | - | 0.563 ± 0.351 | 0.549 ± 0.067 | 0.98 | 0.055 ± 0.005 | 0.024 ± 0.004 | 0.019 ± 0.003 |

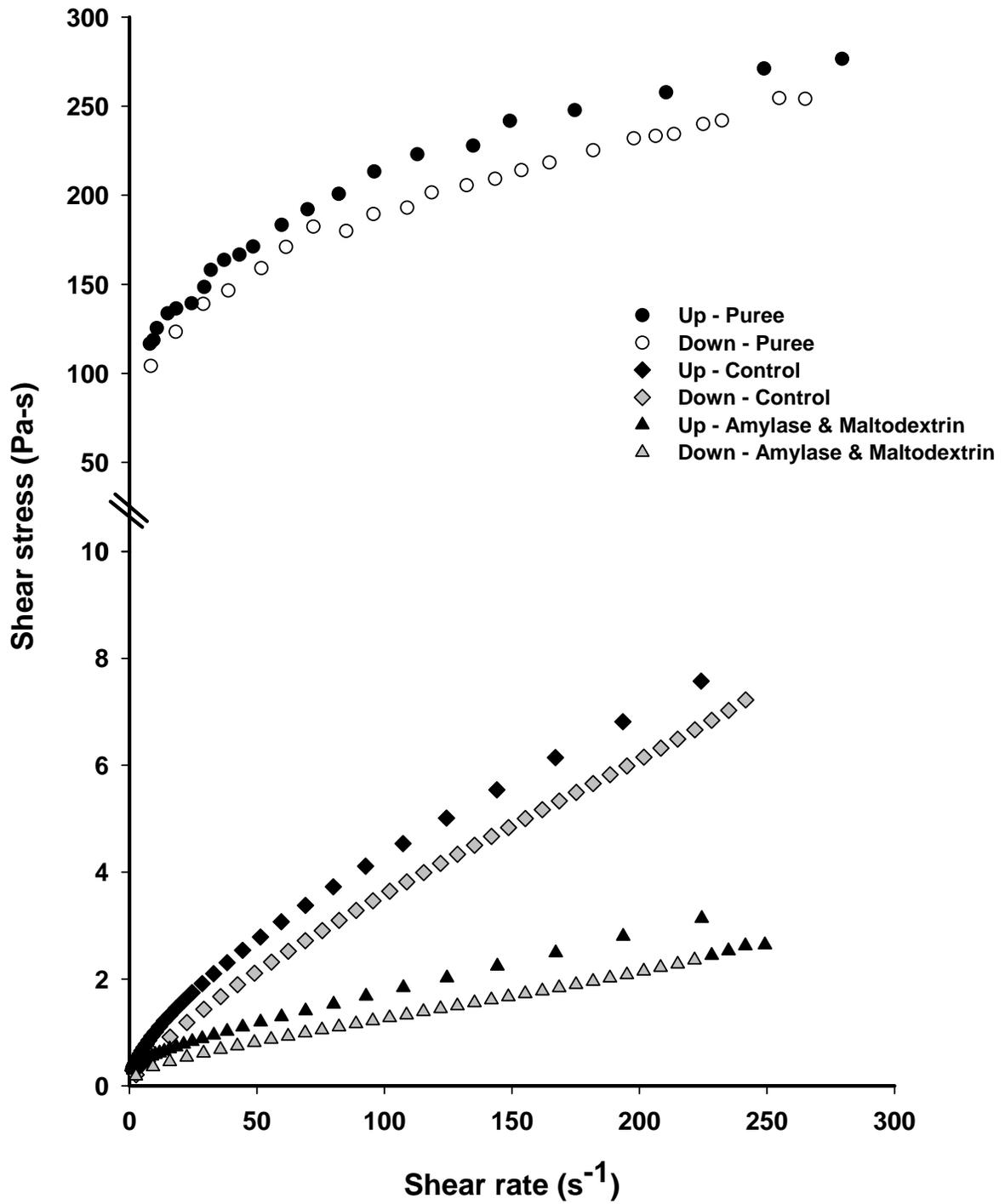


Figure 3. Shear rate ramps from $1\text{-}250\text{s}^{-1}$ of sweetpotato puree and powders at 75°C . Differences between the up and down ramps indicate time dependent behavior.

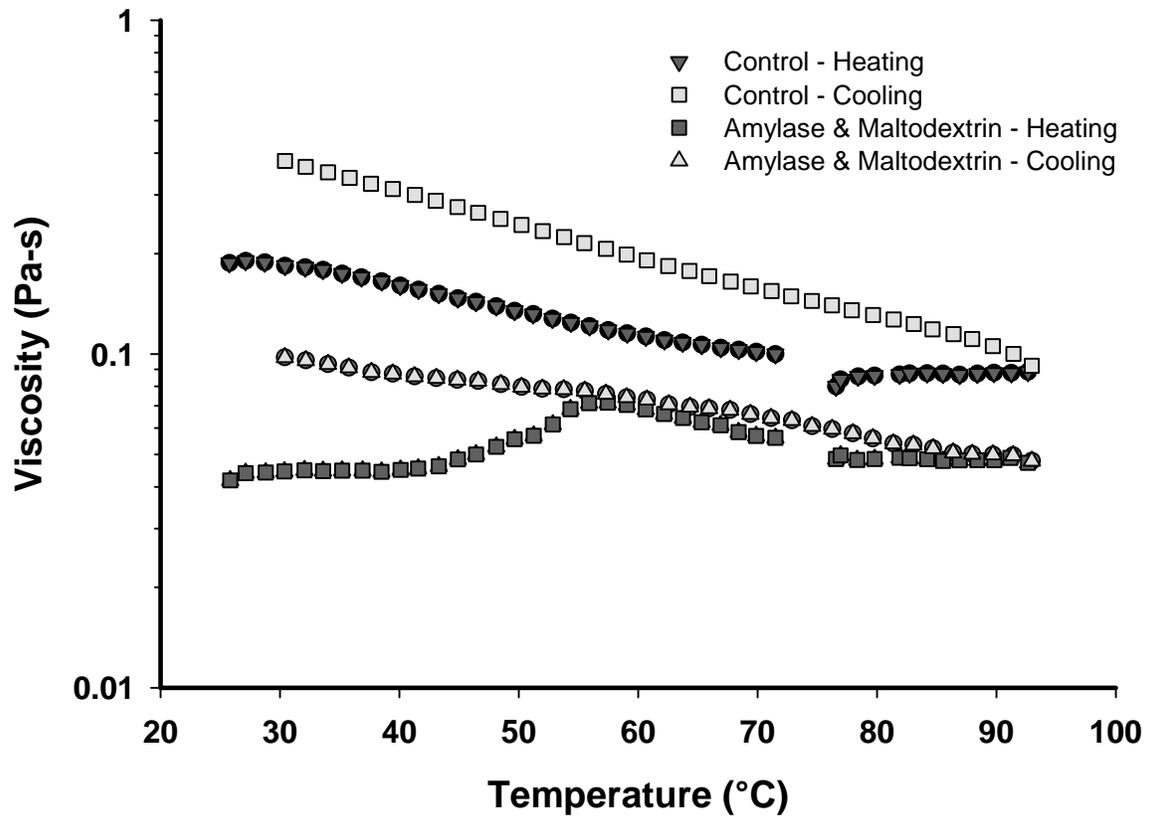


Figure 4. Viscosity of spray dried powders heated and cooled from 25 to 95°C at a constant shear rate (10s^{-1})