

MODELING AND OPTIMIZATION OF THE DEHYDRATION OF BEETS FOR USE AS A  
VALUE-ADDED FOOD INGREDIENT

by

AUDREY SANDRA VARNER

(Under the Direction of WILLIAM L. KERR)

ABSTRACT

Crushed beetroot was dried into powders with different dehydration methods. Powders were evaluated for betalain content and physical properties such as flowability, bulk density, hygroscopicity, and particle size. Modeling of drying with a vacuum belt drier was done to understand the dehydration mechanism. Powder making conditions such as maltodextrin level and drying temperature were optimized with a vacuum belt drier. Powders dehydrated with a vacuum belt dryer at 95°C were found to have the highest drying rate without betalain degradation or unfavorable physical properties. Vacuum belt dried powder was then compared against conventional spray-dried beet powder and golden standard freeze-dried powder, as well as tray-dried and drum-dried powders. Vacuum belt dried powder was found to have comparable betalain content to the freeze dried powder without any unfavorable physical properties. Use of a vacuum belt dryer is effective in creating a quality beet powder for use as a value-added food ingredient.

INDEX WORDS: Vacuum belt drying, spray drying, drum drying, freeze drying, beets, betalains, food powder

MODELING AND OPTIMIZATION OF THE DEHYDRATION OF BEETS FOR USE AS A  
VALUE-ADDED FOOD INGREDIENT

by

AUDREY SANDRA VARNER

AUDREY SANDRA VARNER, B.S. Chemical Engineering, University of Maryland, 2012

A Thesis Submitted to the Graduate Faculty of The University of Georgia in Partial Fulfillment  
of the Requirements for the Degree

MASTER OF SCIENCE

ATHENS, GEORGIA

2014

© 2014

Audrey Sandra Varner

All Rights Reserved

MODELING AND OPTIMIZATION OF THE DEHYDRATION OF BEETS FOR USE AS A  
VALUE-ADDED FOOD INGREDIENT

by

AUDREY SANDRA VARNER

Major Professor: William Kerr

Committee: Ronald Pegg  
Fanbin Kong

Electronic Version Approved:

Maureen Grasso  
Dean of the Graduate School  
The University of Georgia  
May 2014

## ACKNOWLEDGEMENTS

I would like to thank Dr. Kerr, Dr. Kong, and Dr. Pegg for their assistance with my research. I would also like to thank Carl Ruiz, Kathryn Kitchen, Megan Liu, Emily Wagener, Juzhong Tan, Julianne Bierwirth, and Adrian Kerrihard for their assistance.

## TABLE OF CONTENTS

	Page
ACKNOWLEDGEMENTS .....	iv
LIST OF TABLES .....	vii
LIST OF FIGURES .....	ix
CHAPTER	
1 INTRODUCTION .....	1
2 LITERATURE REVIEW .....	4
2.0 Introduction to beets .....	4
2.1 Beets as natural color additives.....	9
2.2 Dehydration methods .....	13
2.3 Properties of food powders .....	17
3 MODELING DEHYDRATION OF CHOPPED BEETS AND OPTIMIZATION OF POWDER PRODUCTION WITH A VACUUM BELT DRYER BASED ON CHEMICAL AND PHYSICAL PROPERTIES .....	20
Introduction.....	20
Materials and Methods.....	21
Results and Discussion .....	29
Conclusion .....	37

4	EVALUATION AND COMPARISON OF CHEMICAL AND PHYSICAL PROPERTIES OF BEET POWDERS PRODUCED BY DIFFERENT DRYING METHODS .....	53
	Introduction.....	53
	Materials and Methods.....	54
	Results and Discussion .....	59
	Conclusion .....	66
5	SUMMARY AND CONCLUSIONS .....	77
	REFERENCES .....	79

## LIST OF TABLES

	Page
Table 3.1: Models for dehydrating beetroot of two thicknesses at three temperatures .....	44
Table 3.2: Diffusion coefficient ( $D_{\text{eff}}$ ), drying ratio, productivity, and drying rate of beet powders of different thicknesses dried at different temperatures.....	47
Table 3.3: Water activity, moisture content, color, and bulk density of beet powders dried at different temperatures with different levels of maltodextrin .....	48
Table 3.4: Percent beet powder emerged from drum after 4 and 20 s .....	49
Table 3.5: Percent hygroscopicity and moisture sorption model for beet powders dehydrated at 95°C with the addition of three levels of maltodextrin.....	50
Table 3.6: Guggenheim-Anderson-de Boer (GAB) model of moisture isotherms of beet powders with three levels of maltodextrin dried at 95°C .....	51
Table 3.7: Total betalain quantification of beet powders dried in a vacuum belt dryer at three temperatures and with three maltodextrin levels .....	52
Table 4.1: Water activity, moisture content, color, and bulk density of beet powders prepared with different drying methods.....	72
Table 4.2: Flowability of beet powders prepared with different drying methods .....	73
Table 4.3: Percent hygroscopicity and moisture sorption model of beet powders prepared with different drying methods.....	74

Table 4.4: Guggenheim-Anderson-Boer (GAB) model of moisture isotherms of beet powders prepared with different drying methods.....75

Table 4.5: Total betalain quantification of beet powders prepared with different drying methods76

## LIST OF FIGURES

	Page
Figure 3.1: Schematic of vacuum belt dryer .....	38
Figure 3.2: Fit of Henderson and Pabis model to dehydration data.....	39
Figure 3.3: Diffusion coefficients of beet samples vs. 1/T .....	40
Figure 3.4: Flowability of beet powders dried at 75°C with the addition of different levels of maltodextrin .....	41
Figure 3.5: Dynamic hygroscopicity- moisture content of beet powders with three levels of maltodextrin as they adsorb moisture over time .....	42
Figure 3.6: Moisture isotherms of beet powders with three different levels of maltodextrin dried at 95°C .....	43
Figure 4.1: Scanning electron microscopy pictures (SEM) of beet powders .....	68
Figure 4.2: Flowability of beet powders prepared with different drying methods .....	69
Figure 4.3: Dynamic hygroscopicity of beet powders prepared with different drying methods ...	70
Figure 4.4: Moisture isotherms of beet powders prepared with different drying methods.....	71

## CHAPTER 1

### INTRODUCTION

*Beta vulgaris L.*, commonly known as the red beet or beetroot, is a root vegetable grown all over the world. While not a frequently consumed commodity, beets can be eaten raw or roasted, often served in soups or on salads. Beets are composed of 87.57% water, 9.56% carbohydrates (29.3% fiber and 70.7% sugar), 1.61% protein, and 0.17% lipids in addition to being a source of potassium, choline, vitamin C, and niacin (USDA, 2011). They also contain betalains, nitrogen-containing pigments, which are commonly added to foods as a source of red-purple natural color. Betalains are also great antioxidants, inhibiting cancer cell proliferation and increasing resistance of low-density lipoproteins from oxidation (Kapadia et al, 1996; Wu et al., 2006; Tesoriere et al., 2004; Gentile et al., 2004). Recent studies have shown that consumption of beet juice may improve performance in endurance sports (Muggeridge et al., 2014; Cermak et al., 2012; Murphy et al., 2012).

Due to the multitude of health benefits associated with consumption of beets, availability of a broader range of products would increase accessibility to betalains. One product that is available is powder from dehydrated beets. These powders have an extended shelf life, decreased risk of microbial hazards, function as a strongly colored pigment, and can be used as value-added ingredients in a variety of food products. However, dehydration of beetroot while maintaining high content of betalains is a challenge. Betalains, while stable across the broad pH range of 3 to 7, are extremely heat labile. Thermal degradation follow first-order reaction kinetics, as betalains

are degraded by isomerization, decarboxylation, or cleavage (Saguy et al, 1978; Herbach et al., 2004a; Huang et al., 1985; Drdak et al., 1990).

Dehydration of beets via spray drying, tray drying, and freeze drying have been previously studied. Spray-drying beets is commonly performed with a carrier such as maltodextrin to decrease powder hygroscopicity (Gokhale et al., 2011). Using higher temperatures when spray drying results in a higher drying rate but also greater loss of betalains than freeze drying (Cai et al., 2000; Janiszewski et al., 2013). Spray drying beet juice at 150°C led to a degradation of betacyanin pigments of 2.77% while tray drying beets at 53°C led to a 10% loss in color retention (Cai et al., 2000; Gokhale et al., 2012). Tray drying beets results in a higher yield of beet powder with a smaller energy investment (Gokhale et al., 2011).

A novel dehydration method, vacuum belt drying, which has not yet been evaluated for the dehydration of beets, could create high quality powders with minimal betalain loss. Vacuum belt drying allows for use of the whole beet, not just the juice like spray drying, reducing the amount of waste produced from processing. Because of the vacuum, beets will dehydrate faster than tray dried beets. Finally, due to gentle heating and ability to make the process continuous, vacuum belt-dried beets will dry faster and have a lower energy requirement than freeze-dried beets. Some products that have been successfully dried with a vacuum belt dryer include banana puree (Wang et al., 2007), rabbiteye blueberry slurries (Kim et al., 2012), *Panax notoginseng* extract (Liu et al., 2011), and fresh squeezed juices (Monzini et al., 1990).

This study will involve the dehydration of beets into powders using a vacuum belt-dryer at different temperatures, thicknesses, and levels of maltodextrin added, drum dryer, freeze dryer, spray dryer, and tray dryer. The objectives of this study are:

1. To model the dehydration of crushed beets with the vacuum belt dryer based on different drying temperatures and sample thicknesses
2. To optimize the dehydration of crushed beets with the vacuum belt dryer based on different drying temperatures and addition of different maltodextrin levels
3. To compare the physical properties of beet powders prepared with different forms of dryers (drum dryer, freeze dryer, spray dryer, tray dryer, vacuum belt dryer) based on bulk density, flowability, moisture isotherm, dynamic hygroscopicity, color, moisture content, and water activity
4. To compare the betalain content of beet powders prepared with different forms of dryers (drum dryer, freeze dryer, spray dryer, tray dryer, vacuum belt dryer).

## CHAPTER 2

### LITERATURE REVIEW

This review will go through the history, current use and market of beets. It will then review the nutritional profile of beets and go in depth explaining betalains, the important phytochemical associated with the health benefits from beets. The review will then cover the usage of betalains and their stability, including the effects of processing on betalains. Different dehydration methods currently used for drying beets will be reviewed before going into important physical properties that are important in evaluating the quality and functionality of food powders.

#### **2.0 Introduction to beets**

*Beta vulgaris* subsp. *vulgaris*, known as the common beet, garden beet, red beet, beetroot, or table beet is a root vegetable in the Chenopodiaceae family. Other vegetables in this family include spinach, chard, quinoa, and sugar beets (Goldman et al., 2008). In America, the *Beta vulgaris* genus was recently classified under Amaranthaceae (Neelwarne, 2012).

##### **2.0.1 History of beets**

Beets were named after the Greek letter *beta*, as the swollen roots appeared to resemble the letter. The genus originated in North Africa and spread through the Mediterranean Sea into Asia and Europe. The leafy greens from beetroots were first used for animal feed. Roots increased in size and pigment as the plant adapted to new soils as tribal people in Europe frequently moved around. Napoleon first had the idea to use beets as a source of sugar production, which led to a rising popularity in beets and creations of many cultivars. Sugar beets

were created by several selections followed by breeding and reselection from red beets, to increase the sugar content from 6% to 20%. The sugar beet now accounts for 30% of the world's sugar and is a widely cultivated crop in Europe, Canada, and America for use for production of table sugar (Neelwarne, 2012).

### **2.0.2 Beet market**

The vegetable market in the United States was valued at 10 billion US dollars in 2012, with onions, corn, and lettuce being the three commodities worth the most (USDA, 2013). Beets are grown in temperate areas all over the world, with main production in North America and Europe. In 2009, worldwide production of beets was 227 million tons, with 26.8 million tons grown in the United States of America, 25.9 million tons grown in Germany, and 24.9 million tons grown in the Russian Federation (FAOSTAT, 2011). Annual crop yield ranges from 50 to 70 ton per hectare, with beet growing season lasting from July until October (Neelwarne, 2012).

The two most popular varieties of beets are the Globe beetroot and the Egyptian beetroot, both dark red globe beetroots. Dark red globe beetroots are most popular with growers, including other varieties such as the Derwent Globe, Darkest Globe, and Detroit Dark Red. Other types of beets include Golden Beets, which do not bleed, and White Albina, which are sweeter than typical dark red beets. Both of these varieties produce tops, known as chard, which have pigments that color them white, pink, yellow, and red, and can be harvested and cooked (Neelwarne, 2012).

### **2.0.3 Nutritional profile of beets**

Beets are composed of 87.57 g water, 1.61 g protein, 0.17 g lipids, and 9.56 g carbohydrates per 100 g. Of the carbohydrates, beets are composed of 29.3% fiber (2.8 g total dietary fiber/ 100 g beet) and 70.7% sugar (6.76 g sugar/ 100 g beets). Beets also contain a

variety of minerals (mg mineral/ 100 g beets): calcium (16), iron (0.80), magnesium (23), phosphorus (40), potassium (325), sodium (78), zinc (0.35), Copper (0.075), manganese (0.329), and Selenium (0.0007) in addition to a variety of vitamins ( $\mu\text{g}$  vitamin/ 100g beets): vitamin C (4900), thiamin (31), riboflavin (40), niacin (334), pantothenic acid (155), vitamin B6 (67), total folate (109), total choline (6000), Betaine (128700), vitamin A (33), beta carotene (20), vitamin E (40), and vitamin K (0.2) (USDA, 2011).

Phytochemicals are chemical compounds naturally occurring in plants that contribute a function other than nutrition. The major phytochemicals found in beets are betalains, which have a high bioavailability, as Kanner et al. (2001) had subjects consume beet juice and was able to identify only 0.5-0.9% of ingested betacyanins in the urine, indicating that the rest were absorbed into the body. Betalains, rarely occurring in the diet due to limited plant food sources, will be discussed in further detail later in this review. Beets contain p-coumaric acid, feruloylamaranthin, and ferulic acid (Kujala et al., 2000). Two flavonoids are present in beets, quercetin and luteolin, at 0.13mg/ 100g and 0.37 mg/ 100g respectively (Bhagwat, 2013). Beets contain 0.4-0.5% galacturonan, highly methylated pectin (DM approximately 70%), and glucose polysaccharides (28-39%) as cellulose (Dongowski, 2001). Jiratanan et al. (2004) found a 5% increase in phenolic content of beetroot after they underwent a commercial canning process.

#### **2.0.4 Other health benefits associated with beets**

Beets and beet juice have been studied for their antioxidant, anticancer and antidiabetic effects as well as being a source of dietary nitrates that reduce blood pressure and may improve athletic performance.

Many fruits and vegetables with polyphenolic compounds function as antioxidants in vivo, or molecules that inhibit oxidation of other molecules that may lead to degenerative

diseases. Beets have a strong antioxidant effect due to their betalain content, as betalains contain a partly glucosized phenolic group and a cyclic amine group. The peel of beets contains the highest total phenolic content, followed by the crown, and finally the flesh (Kujala et al., 2000). The phenolic compounds act as free radical scavengers and prevent oxygen-induced and free radical-mediated oxidation of biological molecules (Pedreno et al., 2001). Kanner et al. (2001) found that even low concentrations of betalains can inhibit lipid peroxidation of membranes, with an IC<sub>50</sub> of <2.5 μM for inhibition of H<sub>2</sub>O<sub>2</sub>-activated metmyoglobin catalysis of low-density lipoprotein oxidation. Two betalain compounds, betanin and betanidin, were found to inhibit lipid oxidation. At pH >4, betanin was found to be 1.5-2.0 times more effective as antioxidants than anthocyanins as determined in the Trolox equivalent antioxidant capacity assay. At higher pH values, betanin becomes a better hydrogen and electron donor and therefore has increased free radical-scavenging activity (Gliszczynska-Swiglo et al., 2006).

Beets have been studied for their anticancer effects, however many studies have been performed on animals and there is limited research on human subjects. Kapadia et al. (1996) discovered a 60% reduction in lung tumors from the control, when a crude extract from red beetroot was fed to mice. Betanin consumption exhibited inhibitory effects on two-stage carcinogenesis of mouse pulmonary tumors and hepatic tumors (Konoshima et al., 2003). After rats were fed a diet of red beet fiber, there was a significant reduction in the incidence of precancerous lesions in the colons of the rats and the number of animals bearing tumors was reduced by 30% (Bobek et al., 2000).

Beets have also been evaluated for anti-diabetic effects. Ozsoy-Sacan et al. (2004) observed effectiveness of *Beta vulgaris* chard extract in controlling hyperglycemia in STZ diabetic rats. Female mice that ingested beet red pigment for 30 days did not show alloxan-

induced diabetes after being injected with alloxan, thus suggesting that the onset of diabetes was prevented due to the beets that were ingested (Yamasita, 2008).

Many consumers are interested in consuming beets or beet products as a source of dietary nitrogen. Beet juice contains a high level of dietary, inorganic nitrate, with 34-45 mmol/L (Webb et al., 2008). Consumption of beetroot juice has been shown to raise plasma nitrate and plasma nitrite, as well as increase heart rate and decrease blood pressure for three hours after consumption. The mechanism is as follows: Dietary nitrate is consumed by drinking beet juice or eating beets or beet-containing products. Nitrates are then concentrated in the salivary gland, absorbed from the stomach and small intestine, and excreted by the kidneys. In the mouth, bacteria reduce nitrates to nitrites. Acidic conditions in the stomach further reduce the nitrites to nitric oxides; nitrites and nitric oxides are brought into circulation by the liver and transported through the arterial circulatory system. Finally, nitrites are reduced to nitric oxide in resistance vessels where there is lower oxygen tension and therefore reduce the body's blood pressure (Webb et al., 2008). Other benefits of consuming dietary nitrogen include prevention of endothelial dysfunction, inhibition of platelet aggregation, and protection against ischemia reperfusion injury (Gilchrist et al., 2010). Some argue that a diet that involves too much dietary nitrogen may actually have negative health effects. Many studies have been performed to demonstrate that there may be a positive link between nitrate intake and cancer, however many of these studies are weak and imply that there may be some groups at risk but there is no overall trend (Gilchrist et al., 2010).

Finally, beetroot products including beet juice have been evaluated as an athletic performance enhancing super-food. A study performed by Murphy et al. (2012) showed that subjects ran 5 km faster with less perceived exertion after consuming baked beetroot when

compared to the placebo of cranberry relish. Male cyclists that consumed beetroot juice completed their time-trial 16.1 km bike ride faster after imbibing beet root juice than after the placebo, with average times of 1664 s and 1702 s (Muggeridge et al., 2014). However, some studies have also shown that beet products do not affect athletic performance. Club-level cyclists who consumed 0.5 L of beetroot juice before riding 50 miles as a time trial did not significantly improve their performance when compared against a placebo (Wilkerson et al., 2012). In another study, trained male cyclists who ingested 140 mL of concentrated beetroot juice before cycling a one-hour time trial did not show improved performance when compared to cyclists who ingested a placebo prior to their cycling (Cermak et al., 2012). The idea of using beet juice or other beet products to aid in athletic performance is still relatively new and further investigation is needed to determine if there is any correlation between beet consumption and exercise performance.

## **2.1 Beets as natural color additives**

Color is very important in food products, as it is the first characteristic that is noticed by consumers. It is primary screening for consumers to evaluate the quality of food, as they can see if fruit is bruised or vegetables are wilted. Colors are added to food for several reasons, including: to ensure uniformity of color across batches of a product, to give color to colorless foods, to restore the original color of a food which has been lost during processing, or to reinforce colors present in food which are less intense than consumers expect (Jackman et al., 1996). A survey conducted by Whole Foods found that 32% of consumers are willing to pay more for foods without artificial ingredients, preservatives, or colorings. The artificial food coloring market, valued at \$570 million in 2011, has been overpassed by the natural color market, valued at \$600 million in 2011 (Mintel, 2013). Natural pigments are synthesized and

accumulated in or excreted from living biological cells. Pigments can come from plants including algae, vertebrates, invertebrates, fungi, lichens, or bacteria (Jackman et al., 1996).

Many fruits and vegetables are used as sources of natural color in processed foods and beverages. Some natural color pigments are water-soluble, such as anthocyanins, carminic acid, and finally betalains, which come from beets. Other natural color pigments are fat-soluble, such as carotenoids and chlorophylls (Azeredo, 2009).

### **2.1.1 Betalains**

Beets get their color from nitrogen-containing pigments called betalains, common in roots, fruits, and flowers (Strack et al., 2003). Beets do not contain any anthocyanins, as betalains and anthocyanins are mutually exclusive. The average amount of betalains in beets is estimated to be 120 mg/ 100 g of fresh weight (Marmion, 1991). There are two groups of betalains: red-violet betacyanins and yellow-orange betaxanthins, both containing betalamic acid as a common chromophore. Betalains are synthesized with the condensation of betalamic acid from tyrosine. If the condensation occurs with an amino acid (e.g. Ser, Val, Leu, Iso, and Phe) or amino acid derivative, the betalain will be a betaxanthin. If the condensation reaction occurs with a derivative of dihydrophenylalanine (DOPA), then the resulting betalain will be a betacyanin (Moreno et al., 2008). The two most prominent pigments in beets are betanin (red) and vulgaxanthin I (yellow) (Azeredo, 2009). Betacyanins contain a cyclo-3,4-dihydroxyphenylalanine residue thus shifting the absorption maximum from 480 nm of betaxanthins up to 540 nm due to the extra conjugation (Strack et al., 2003). Betalains are relatively pH stable, as they are stable across the pH range of 3 to 7 (Jackman et al., 1996). However, betalains are not very stable across different levels of water activity, temperatures, exposure to oxygen, and light. Betalains degrade most rapidly at an intermediate water activity

level due to dilution effects at higher levels and decreasing mobility of reactants at lower values. Betalains also degrade more rapidly in an excess of oxygen. Attoe et. al. (1982) found that betanine degrades by a pseudo-first order rate and the activation energy of the loss of betanine was 30.7 kcal/ mol in the absence of oxygen. Oxidation of betalains is accelerated in the presence of light.

In beetroots, betalains function as enzyme inhibitors, viral resistors, and inhibitors of microbial growth and respiration (Jackman, 1996). Because the absorbance peaks of betalains occur in the 465-560 range, they may act as green light filters and therefore repress plant growth. Betalains also absorb UV light thus indicating that they may have a role in protecting the plant against harmful UV irradiation. Betalains may also act as nitrogen reservoirs for the plants. The fact that betalains evolved later in evolution and exist in plants that are mutually exclusive of anthocyanins may imply that these plants chose to switch to betalains because the pigments are stable across a wide pH range and there exists a wide spectrum of colors available with just small changes to the biosynthetic pathway after betalamic acid synthesis (Neelwarne, 2012).

### **2.1.2 Uses of betalains**

Due to the strong color of the betalain pigments, beet extracts are used as natural food colorants. In the United States and European Union, the only allowed source of betalain colorant is the beetroot. According to 21 CFR 73.40 and 21 CFR 73.260, dehydrated beets (beet powder) and beet juice can be “used safely in amounts consistent with good manufacturing practices” and are exempt from certification. The European Union classifies beetroot red as E162 and allows usage of it as a natural colorant at *quantum satis*, or as much as needed, not setting a maximum amount allowed for use (Jackman et al., 1996).

Beetroots are commonly processed into juice, liquid concentrate, color extract, and powder. Juice is created by hydraulic pressing beets, traditionally prepared by blanching, chopping, pressing, and filtering of raw beets, resulting in less than 50% retention of betalains. Beet juice can be converted into a concentrate by centrifuging, pasteurizing, then concentrating the juice until it is about 70% sugar and 0.5% betanin. Color extracts are produced to increase the color content and reduce the flavor by fermenting the sugar into alcohol and then removing the alcohol. Beetroot juice and powder are the two most common forms of betalains used for coloring foods, with juice containing 1% betalain content and powders containing 0.4 to 0.7% (Jackman, 1996).

Applications are limited due to beets' sensitivity to heat and light, as well as concentration of odorous compounds such as geosmin. Betalains can be used as a source of color in products that undergo minimal thermal treatment, with a relatively short shelf life, that are packaged and stored under reduced levels of light and oxygen. Some examples of food products and the level of their usage are: Yogurt (0.08%), ice cream (0.25-0.27%), dry powder beverages (1.0-1.5%), hard candies (0.15%), jellies (0.18%), water ices (0.5-1.0%) and marzipan (0.45%) (Agrawal, 2013). Yellow-orange betaxanthins can be used in water-soluble applications in place of carotenoids (Azeredo, 2009). Beetroot is also used to color some comminuted meat products with low moisture content that do not contain SO<sub>2</sub> such as salami sausages (Jackman et al., 1996).

### **2.1.3 Effects of processing on betalain content**

Betalains are heat labile and undergo thermal degradation. Ravichandran et al. (2013) found that there was a decrease in betaxanthin content of 18%, 23%, and 33% and a decrease in betacyanin content of 6%, 22%, and 51% after heating beets at 80°C for 60s, 120s, and 180s

respectively. When thermally processed, betalains are converted to decarboxylated derivatives including 2-decarboxy-betacyanins and 2,17-bidecarboxybetanin and dehydrogenated byproducts including 14,15- dehydrogenated betacyanins, 2,17-bidecarboxy-2,3-dehydro-neobetanin, 2,15,17-tridecarboxy-2,3-dehydro-neobetanin, and 2-decarboxy-2,3-dehydro-neobetanin (Nemzer et al., 2011).

## **2.2 Dehydration methods**

Dehydration of foods is the oldest method of preservation, including drying fruit in the sun and smoking fish and meat (Cohen et al., 1995). Developing countries such as India have nearly 40% post-harvest loss due to lack of cold storage chains and unorganized transport. Therefore, dehydration of vegetables can improve profitability for farmers (Gokhale et al., 2011). Dehydrated vegetables are produced in many varieties- sliced, diced, flaked, chopped, minced, or as a powder- and are mostly used in packaged mixes such as soups and sauces (Arsdel et al., 1973). Foods such as fruits and vegetables are dried for a variety of reasons including increased storage stability and therefore increased shelf life, minimized weight for easier transportation, and minimized packaging requirements (Sagar et al., 2010). Dehydration of food products also makes food safer by reducing microbial hazards. Pathogenic bacteria cannot grow below a water activity of 0.85-0.86 and yeasts and molds cannot grow below a water activity of 0.80 (Rahman, 2007). Sensory properties, including flavor, color, aroma, and texture, can change during dehydration (Sagar et al., 2010).

Beets can be dried in a variety of ways, depending on the end product desired. The most commonly used methods of dehydration are spray drying and tray drying. Spray drying, freeze drying, drum drying, tray drying, and vacuum belt drying are all methods which are used for the

dehydration of vegetables and are reviewed below as methods to be evaluated for the dehydration of beets into powder.

### **2.3.1 Spray drying**

Spray drying involves the atomization of a liquid into small droplets, which then make contact with heated air, thus drying the liquid to a fine powder. The most significant applications of spray drying in the food industry are milk, coffee, tea, eggs, whey protein, enzymes, and microorganisms (Barbosa-Cánovas et al., 1996). Co-current processes have hot air flow in the same direction as the liquid is sprayed, thus exposing the dry powder products to moderate temperatures, 50-80°C, as opposed to counter-current processes where the hot air flows against the direction of the liquid being sprayed, exposing the dry powder product to higher temperatures (Gharsallaoui et al., 2007). Higher spray drying air temperatures result in a higher drying rate and higher powder productivity, however, it causes more betacyanin losses. Compared to freeze drying, spray drying led to a 2.77% degradation of betacyanin pigments at 150°C, 3.85% at 165°C, 4.14% at 180°C, 6.08% at 195°C and 7.66% at 210°C (Cai et al., 2000). Janiszewski et al. (2013) found a 26.7-29.3% retention rate of beet root pigments after spray drying beet juice. Spray drying requires the addition of maltodextrin or another binding agent thus resulting in a less brightly colored powder. The powder produced from spray drying beets without a carrier is very hygroscopic which is undesirable as it may lead to lump formation (Gokhale et al., 2011).

### **2.3.2 Freeze drying**

Freeze drying is a two-step process- a product is first frozen and then the ice undergoes sublimation into vapor due to low pressure conditions (Sagar et al., 2010). Freeze dried products retain their original shape and texture well, and are light, dry, and porous. Freeze drying is an expensive method of dehydration due to the high energy cost of freezing the product, use of

vacuum, and slow drying rate (Barbosa-Canovas et al., 2005). Foods that have been freeze-dried generally have higher quality characteristics such as high porosity, low bulk density, better rehydration properties, and better retention of flavor and aroma when compared to foods that have been dried with alternative processes (Krokida et al., 1998).

### **2.3.3 Drum drying**

Drum drying is a method of dehydration that utilizes rotating metal cylinders to dry liquids or slurries by heating upon contact. Foods that are commonly drum dried include milk, soup mixes, baby foods, potato slurries, and instant cereals (Barbosa-Cánovas et al., 1996). Drum dryers are classified as single drum dryer, twin drum dryer (drums rotate away from each other at the top), and double drum dryer (two drums rotate towards each other at the top). Drum dryers can dry very viscous foods such as pastes or starches, are highly energy efficient, and create products with good porosity, however, drum dryers cannot process corrosive materials, expose products to high temperatures and products high in sugar may be difficult to scrape off of the drums (Tang et al., 2003).

### **2.3.4 Tray drying**

Convective drying of beets involves a smaller investment and results in high yield of a better quality product than spray drying (Gokhale et al., 2011). Gokhale et al. (2012) found optimal conditions of 53°C temperature, 7-mm thickness, and 63% air recirculation ratio for 90% color retention of *Beta vulgaris* with convective dehydration. Crushed beet was found to be better than shredded beets or sliced beets for drying due to its increased bulk density and therefore increased loading capacity (Gokhale et al., 2011).

### 2.3.5 Vacuum belt drying

Vacuum drying is often used for dehydration of heat sensitive materials as lower temperatures may be used when the product is under vacuum (Sagar et al., 2010). Liu et al. (2009) found that moisture ratio decreased with an increase in drying temperature and decreased with an increased belt speed. Vacuum belt drying was found to be an ideal process for dehydration of an herbal extract when compared with other drying methods because it was advantageous in producing a product with lower moisture content in a shorter amount of time than freeze drying or vacuum drying with the same pH value as the raw extract (Liu et al., 2011). A vacuum belt dryer which uses both conduction and radiation for heat transfer can lead to a thermal efficiency increased by 20% when compared to a conventional vacuum dryer (Hayashi et al., 1983).

Many food products have been dehydrated with vacuum belt drying, however the dehydration of beets by vacuum belt dryer has not previously been studied. Liu et al. (2009) dried *Panax notoginseng* extract with a vacuum belt dryer at temperatures of 90, 100, and 110°C and belt speeds of 4, 7, & 10 cm/ min and found  $D_{\text{eff}}$  values of 4.86 to  $11.0 \times 10^{-7}$  m<sup>2</sup>/s. Liu et al. (2010) found optimal powder properties such as porosity and hydrogen peroxide scavenging activity of vacuum belt-dried *Panax notoginseng* extract when compared to spray-dried, freeze-dried, and vacuum-dried extracts. Wang et al. (2007) evaluated volatiles in dried banana powder and discovered that vacuum belt drying banana puree which passed under 5 plates set to temperatures beginning at 210°C and decreasing to 50°C had a better retention of volatiles than air drying banana puree at a constant temperature of 75°C. Fresh squeezed juices were dried with a vacuum belt drier to preserve their colors and tastes (Monzini et al., 1990). Rabbiteye blueberry

slurries were dried with a vacuum belt dryer to produce high-quality powders with high anthocyanin content (Kim et al., 2012).

## **2.4 Properties of food powders**

Food powders are used for a variety of purposes; as spices, sweeteners, additives, mixes for convenience foods such as cake mixes, flours, additives such as vitamins or pigments, and instant foods such as soups and beverages. Food powders are usually of low value as they are perceived to be of low quality (Fitzpatrick et al., 2005). Vegetables or vegetable juice can be dehydrated and ground into powders for use as ingredients or preservation due to extended shelf life. Important powder classification criteria such as flowability and hygroscopicity are difficult to quantify because they are dependent on many physical and physiochemical phenomena (Peleg, 1983).

### **2.4.1 Bulk density**

Bulk density is the mass of particles that occupy a certain volume. Most food powders have bulk densities between 0.3 and 8.0 g/cm<sup>3</sup>. Bulk density depends on the particle size and shape, strength of interparticle attractive forces, cohesion, and density of the particles. Higher moisture content in powders leads to a decreased bulk density while addition of anticaking agents increases the bulk density of powders (Peleg, 1983). A general trend with powders shows an increased bulk density with decreased particle size (Grabowski et al., 2006). Liu et al. (2011) found that vacuum belt dried *Panax notoginseng* extract had the lowest bulk density at 0.304 g/cm<sup>3</sup> compared to spray dried and freeze dried, with bulk densities of 0.316 g/cm<sup>3</sup> and 0.389 g/cm<sup>3</sup> respectively. Cai et al. (2000) found that increased temperatures during spray drying led to decreased bulk density of *Amaranthus* betacyanin pigments, probably due to the higher temperature creating a higher surface area-to-volume ratio.

### **2.4.2 Flowability**

Peleg (1983) defines powder flow as the relative movement of a bulk of particles among neighboring particles or along the container wall surface. Resistance to flow primarily comes from friction; therefore compaction of a food powder from even small pressure may cause lack of flow. Factors that affect the flowability of a powder include particle surface properties, particle shape and size distribution, and geometry of the system (Barbosa-Canovas et al., 2005). The amount of moisture present affects the flowability of a powder, as the greater the water content, the more difficult it is for the powder to flow due to a higher cohesiveness (Teunou et al., 1999).

### **2.4.3 Hygroscopicity**

Many food powders spontaneously agglomerate with contact with a moist atmosphere or high storage temperatures. This process occurs with the initiation step of formation of liquid bridges between particles. Anticaking agents are very fine powders of inert chemicals added to powders to improve flowability. Anticaking particles work by covering the surfaces of the host particles, reducing interparticle interactions and inferring with formation of liquid bridges (Peleg, 1983). Tonon et al. (2008) found that the lowest hygroscopicity values were found with higher flow rates and decreased temperatures when spray drying acai. The hygroscopicity was also higher with lower particle moisture content, due to a larger moisture gradient between the powder and its ambient surroundings. The hygroscopicity of spray-dried betacyanin powders with 15 DE maltodextrin ranged from 45.4 to 49.2 g/ 100 g and was significantly less than spray-dried betacyanin powders without added maltodextrin, 118.3g / 100g. The hygroscopicity of spray-dried betacyanin powders with 25 DE maltodextrin was the most hygroscopic at 69.4 g/ 100g while the powder with 10 DE maltodextrin was the least hygroscopic at 40.9 g/ 100 g. This

implies that using maltodextrin as a carrier can help reduce the hygroscopicity of a powder and thus improve upon its storage and functional properties.

Carriers with lower glass transition temperatures caused higher hygroscopicities of spray-dried powders due to shorter chain lengths and more hydrophilic groups on lower molecular weight maltodextrins, therefore maltodextrins with lower DE values are better for reducing hygroscopicity in powders (Cai et al., 2000).

#### **2.4.4 Solubility/ water absorptivity**

The ability of a powder to dissolve into water is an instant property. Ideal powders, which are intended for rehydration, dissolve quickly and completely rather than floating or clumping (Hogekamp et al., 2003). The addition of maltodextrin to a sweet potato powder made by spray-drying sweet potato puree increased the solubility index of the powders due to the inverse relationship between the concentration of maltodextrin added and the mean diameter of the powder particles. The surface stickiness of the particles was reduced with the addition of maltodextrin due to reduced particle cohesion and wall adhesion, therefore resulting in less agglomerate formation and a lower water-holding capacity (Grabowski et al., 2006). A general trend is that more porous products rehydrate faster. However, Hawlader et al. (2008) found that vacuum-dried papaya had a larger porosity than air, nitrogen, or carbon dioxide drying due to its puff effect when under low pressure but had a reduced rehydration capability because the puff volume cannot hold water.

## CHAPTER 3

# MODELING DEHYDRATION OF CHOPPED BEETS AND OPTIMIZATION OF POWDER PRODUCTION WITH A VACUUM BELT DRYER BASED ON PHYSICAL AND CHEMICAL PROPERTIES

### **Introduction**

Red beetroot has been used as a source of natural color for years. Direct consumption of beets has recently increased, particularly as a means of increasing dietary nitrates that may enhance health or performance. Drinking beet juice is associated with lowering blood pressure (Webb et al., 2008). Many studies have been performed to evaluate the effect of consuming beetroot juice before athletic performance (Muggeridge et al., 2014; Murphy et al., 2012; Wilkerson et al., 2012; Cermak et al., 2012). Beets are also known for having a high antioxidant content (Pedreno et al., 2001).

Betalains, the phenolic compounds responsible for some of the health benefits in beet consumption, are also responsible for the red color of beets. Betalains are not common in the human diet, as edible sources are limited to red and yellow beetroot, cactus fruits, chard and amaranth (Azeredo, 2009). In order to consume betalains, one must eat beets, beet juice, or beetroot powder. With the short shelf life of beets and beet juice as well as required refrigeration, beetroot powders are ideal for consumers to ingest dietary betalains. Beetroot powder can be used as a value-added ingredient in a variety of products including pasta, smoothies, beverages, soup mixes, and more.

Vacuum belt drying is a non-traditional method that can be used for dehydrating fruits and vegetables into powders. Products dried by vacuum belt dryer are of high quality due to low thermal impact and produced in relatively short amounts of time. Many food products have been dehydrated with vacuum belt drying, however the dehydration of beets by vacuum belt dryer has not previously been studied. Liu et al. (2010) dried *Panax notoginseng* extract with a vacuum belt dryer and found optimal powder properties such as flowability and bulk density. Banana puree was dried with the vacuum belt drier to evaluate volatile compounds (Wang et al., 2007). Fresh squeezed juices were dried with a vacuum belt drier to preserve their colors and tastes (Monzini et al., 1990). Rabbiteye blueberry slurries were dried with a vacuum belt dryer to produce high-quality powders with high anthocyanin content (Kim et al., 2012).

The purpose of this study was to model the dehydration of crushed beets in a vacuum belt dryer and to optimize conditions such as sample thickness, drying temperature, and maltodextrin content to obtain a powder with maximum betalain content and optimal powder properties.

## **Materials and Methods**

### Sample Preparation

Beets (*Beta vulgaris vulgaris*) were obtained from Atlanta State Farmer's Market from Victory Farms (Hudsonville, MI). Whole beets were chopped into 1-mm chunks using a bowl chopper (Model 33, Schneidmischer, Wallau/Lahn, Germany) with 1% ascorbic acid (w/w) to minimize enzyme activity. Ascorbic acid was purchased from Prinova US LLC (Carol Stream, IL). Chopped beets were stored in vacuum-sealed, oxygen- and light-barrier bags at -40 °C until the time of testing. Packages of beets were thawed for 24 hr at 4°C before use. Maltodextrin (M100, Grain Processing Corporation, Muscatine, IA) was added to beet samples at three levels, 0 g/ g beet solids, 0.3 g/ g beet solids, and 0.6 g/ g beet solids. Maltodextrin is an inert carrier

frequently used to aid in dehydration. It was used to reduce the hygroscopicity of powders with high sugar contents and may protect sensitive compounds from processing conditions (Tonon, 2008).

#### Vacuum belt drying

Beet samples were dried with a laboratory scale vacuum belt dryer (VBD) (Zwag, CH-5312 Zchokke Warman Ltd. Bucher, Dottingen, Switzerland). The VBD contained a steel housing, which included a low friction, Teflon-coated fiberglass conveyor belt that passed over three conductive heating plates and one cooling plate (Figure 3.1). The temperature of each plate and the speed of the conveyor belt were all individually controlled. For all trials, heating zones 1-3 were set at 75, 85, or 95°C. The system was connected to a vacuum pump (VT Aqua Seal 80 CFM, Dekker Vacuum Technologies, Michigan City, IN), which was operated with pressurized water. 175 g beets were spread to a thickness of 0.58 cm in aluminum pans that were loaded directly onto the belt. Samples were dried under a vacuum of 13.3 kPa for 10 min then at 1.6-2.0 kPa for the remaining time. At 75, 85, and 95, beets were dried for 160, 130, and 115 min, respectively, until they reached a water activity ( $a_w$ ) of  $0.25 \pm 0.05$ . After drying, samples were taken out of the VBD and cooled to room temperature then ground for 20 s with a Nutribullet (Nutribullet LLC, Pacomia, CA) extraction blade. Powders were stored in vacuum-sealed, oxygen- and light-barrier bags at -40°C until the time of testing.

For generating dehydration curves for mathematical modeling, the system was modified so that one conduction plate, which was set at 75, 85, or 95°C, was attached to an analytical balance (HP 4200C Avery Weigh-Tronix, Fairmont, MN) that continuously recorded the mass of the product. For all trials, samples were loaded into 7.6-cm diameter round frames of height 0.58 cm or 1.07 cm onto thin aluminum pans and set on the plate. A vacuum of 1.6-2.0 kPa was

pulled and samples were dried for up to 275 min, until they each reached a final moisture content of less than 0.1 g water/g dry solids.

### Statistical Analyses

JMP Pro 11 (SAS Institute Inc., Cary, NC) was used to run Analysis of Variance (ANOVA) to analyze all data. Tukey's HSD post-hoc testing was used at a p-value of 0.05 to determine differences between different treatments.

### Mathematical Modeling

Dehydration occurs by diffusion of water across a concentration gradient, from a high moisture content area (such as the surface of the beet sample) to a low moisture content area (such as the air around it). The effective diffusion coefficient is temperature dependent and can be described by the Arrhenius equation in Equation 3.1:

$$D_{eff} = D_0 \exp\left(\frac{-E_a}{R_g T}\right) \quad (3.1)$$

where  $D_0$  is the diffusion coefficient,  $E_a$  is the activation energy,  $R_g$  is the universal gas constant, and  $T$  is the temperature.

Fick's Second Law is used to predict the change in moisture content over time due to diffusion. It is given by Equation 3.2:

$$\frac{\partial x}{\partial t} = D_{eff} \frac{\partial^2 x}{\partial z^2} \quad (3.2)$$

where  $x$  is the moisture content at time  $t$  and  $z$  is the spatial dimension in which diffusion occurs.

Solving this equation by integration for the geometry of an infinite slab results in Equation 3.3:

$$MR = \frac{x_t - x_e}{x_i - x_e} = \frac{8}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{(2n-1)^2} e^{\frac{-(2n-1)^2 \pi^2 D_{eff} t}{4L^2}} \quad (3.3)$$

where MR is the dimensionless moisture ratio,  $x_t$  is the average moisture content at any time t,  $x_e$  is the average moisture content at equilibrium,  $x_i$  is the average initial moisture content, and L is the thickness of the slab. In our cases, the equilibrium moisture content may be taken as to be zero due to vacuum conditions. From Equation 3.3, the moisture ratio (MR) and time were graphed and the following several theoretical, semi-theoretical, and empirical models were applied to determine the best fitting model to represent the dehydration of beets.

$$\text{Newton} \quad MR = e^{-kt} \quad (3.4)$$

$$\text{Page} \quad MR = e^{-kt^n} \quad (3.5)$$

$$\text{Henderson and Pabis} \quad MR = a \cdot e^{-kt} \quad (3.6)$$

$$\text{Two-Term} \quad MR = a \cdot e^{-kt} + c \cdot e^{-gt} \quad (3.7)$$

$$\text{Wang and Singh} \quad MR = 1 + a \cdot t + b \cdot t^2 \quad (3.8)$$

$$\text{Two-Term Exponential} \quad MR = a \cdot e^{-kt} + (1 - a) \cdot e^{-kat} \quad (3.9)$$

$$\text{Verma et al.} \quad MR = a \cdot e^{-kt} + (1 - a) \cdot e^{-kgt} \quad (3.10)$$

$$\text{Modified Henderson and Pabis} \quad MR = a \cdot e^{-kt} + b \cdot e^{-gt} + c \cdot e^{-ht} \quad (3.11)$$

$$\text{Logarithmic} \quad MR = a \cdot e^{-kt} + c \quad (3.12)$$

$$\text{Midilli-Kucuk} \quad MR = a \cdot e^{-kt^n} + b \quad (3.13)$$

$$\text{Modified Henderson and Perry} \quad MR = a \cdot e^{-kt^n} \quad (3.14)$$

Modeling was carried out in MATLAB 7.11.0 (Mathworks Inc., Natick, MA). The models were evaluated based on  $R^2$  (correlation coefficient).

### Drying Ratio, Productivity, and Drying Rate

The drying ratio, productivity, and drying rate of samples of two thicknesses (0.58, 1.07 cm) and dried at three temperatures (75, 85, 95 °C) were measured according to Equations 3.15-3.17:

$$Drying\ Ratio = \frac{(w_0+1)}{(w_f+1)} \quad (3.15)$$

$$Productivity = \frac{Feed\ Rate}{Drying\ Ratio} \quad (3.16)$$

$$Drying\ Rate = Feed\ Rate - Productivity \quad (3.17)$$

Where  $w_f$ = moisture content of powder (dry basis) and  $w_0$ = moisture content of feed (dry basis) (Cai et al., 2000).

### Assessment of Beet Powders

#### Water Activity

The water activity of each sample was measured by a water activity meter (Aqualab Series 3, Pullman, Washington).

#### Moisture Content

For moisture analysis, 1.5 to 2.5 g of each beet powder were placed in 5-cm diameter aluminum sample pans. Samples were dried in a vacuum oven (Model 1430 MS, VWR Scientific Products, Radnor, PA) at 70°C until they reached constant mass to measure moisture content by a gravimetric analysis. Moisture content (MC) was calculated on a wet basis from initial ( $m_i$ ) and final ( $m_f$ ) sample masses:

$$MC \left[ \frac{g\ water}{100\ g\ powder} \right] = \frac{m_i - m_f}{m_i} \times 100 \quad (3.18)$$

#### Color

Color measurements were taken of the beet powders using a chroma meter (Model CR-410 Minolta Co Ltd., Tokyo, Japan). The HSL color space was used, with  $L^*$  representing lightness (low values mean the product is dark, high values mean the product is light),  $c^*$  representing chroma (low values mean low brightness, high values mean high saturation), and  $h$

representing hue (degree of color on color wheel with 0° representing red, 120° representing green, 240° representing blue).

#### Bulk Density

Bulk density was measured by transferring a 10 g portion of beet powders to a 25-mL graduated cylinder. The cylinder was held on a digital vortex mixer (Fisher Scientific, Hampton, NH) for 60 s at 600 rpm. The volume of the powder was read and the bulk density of the sample was determined by the mass of the powder divided by the volume of the powder (Grabowski, et al., 2006). Three replications of each powder were measured.

#### Flowability

Flowability was measured as described by Jaya et al. (2004). Five g of each beet powder were loaded into a rotating drum cylinder, 9 cm in length, 12-cm diameter with two 4-mm slits, 7 cm in length, on opposite sides. The drum was powered by a direct-drive DC motor power supply (BK Precision Model 1710, Yorba Linda, CA) and operated at 30 rpm. The quantity of powder that fell through the slits was recorded continuously for 20 s on an analytical balance (HP 4200C Avery Weigh-Tronix, Fairmont, MN). The total percentage of powder that came out of the drum after 4 and 20 s was also recorded.

#### Dynamic Hygroscopicity

Approximately 1.5 g of each powder was spread evenly onto a Petri dish, maximizing its surface area. The Petri dish was placed on top of an analytical balance (AG204, Mettler Toledo, Greifensee, Switzerland) in an environmental chamber set to 22°C and 75% relative humidity. The mass of the powder was recorded every 5 min until constant masses were obtained. Percent hygroscopicity of powders (HG) was calculated according to:

$$HG [\%] = \frac{\frac{b}{a} + w_i}{1 + \frac{b}{a}} \quad (3.19)$$

where  $a$  (g) is the initial powder weight,  $b$  (g) is the increase in weight of powder, and  $w_i$  is the initial moisture content of the sample.

The data was fit to a moisture sorption model:

$$\ln\left(\frac{m_e - m_i}{m_e - m}\right) = k \cdot t + b \quad (3.20)$$

where  $m_e$  is the equilibrium moisture content,  $m_i$  is the initial moisture content,  $m$  is the moisture content at time  $t$ , and  $k$  is a coefficient that describes the speed of the adsorption of moisture.

### Moisture Isotherms

Approximately 1.5 g of each sample was spread evenly on a plastic plan and placed in an isopiestic chamber containing a saturated salt solution at 22°C. The different solutions and their relative humidity levels were as follows: lithium chloride, 11%; magnesium chloride, 32%, potassium carbonate, 43%; sodium chloride, 75%; and potassium chloride, 85%. Samples were left in the chambers and allowed to pick up moisture until they reached a constant mass for ~4 weeks. The dry basis moisture content ( $M_{db}$ ) of each sample was determined by the following:

$$M_{db}\left[\frac{g \text{ water}}{g \text{ sample}}\right] = \frac{w_f - w_s}{w_s} \quad (3.21)$$

where  $w_f$  is the total mass of wet samples and  $w_s$  is the mass of solids in the original samples.

The adsorption isotherms were fit to the Guggenheim-Anderson-de Boer (GAB) model:

$$M_{db} = \frac{m_0 k c a_w}{(1 - k a_w)(1 - k a_w + c k a_w)} \quad (3.22)$$

where  $m_0$  is the monolayer moisture content,  $k$  is related to adsorption of multiple layers of moisture, and  $c$  is a constant related to the surface enthalpy.

### Extraction of Betalains

For extraction of betalains, 0.1 g of beet powder was added to 10 mL water. The samples were mixed with a vortexer (Standard Mini Vortexer, VWR Scientific Products, Radnor, PA) until homogeneous and then centrifuged with a centrifuge (Centrifuge Model 228, Fisher Scientific, Hampton, NH) for 10 min at 3300 rpm. The supernate was collected and the insoluble portion was re-extracted 2x more and the supernates were combined and brought to a final volume of 50 mL with water in a volumetric flask at room temperature (Kujala et al., 2000; Nemzer et al., 2011).

### Total Betalain Quantification

Betalain extracts were diluted with 0.05 M phosphate buffer, pH 6.5 so that the samples exhibited an absorption between 0.4 and 0.5 AU at  $\lambda_{\max}=538$  nm. A spectrophotometer (Spectronic Genesys 2, Thermo Electron Corporation, Madison, WI) was used to measure the absorption of the samples were recorded at 476, 538, and 600 nm. Corrected light absorptions of betanin and vulgaxanthin-I were calculated according to von Elbe et al. (2001):

$$x = 1.095 \times (a - c) \quad (3.23)$$

$$z = a - x \quad (3.24)$$

$$y = b - z - \frac{x}{3.1} \quad (3.25)$$

where

x=light absorption of betanin minus colored impurities

y=light absorption of vulgaxanthin-I corrected for contribution of betanin and colored impurities

z=light absorption of impurities

a= light absorption of sample at 538 nm

b= light absorption of sample at 476 nm

c=light absorption of sample at 600 nm

## Results and Discussion

Drying temperature and thickness of sample both had effects on the dehydration of beetroot into powder with the vacuum belt dryer. Crushed beets took a longer time to dry at lower temperatures (275 min @ 75°C; 235 min @ 85°C; 175 min @ 95°C for thickness of 1.07 cm). The thicker samples (1.07 cm) took approximately 250% more time to dry than the thinner samples (0.58 cm); in 275 and 110 min at 75°C, respectively.

### Mathematical Modeling

Eleven models, semi-theoretical and empirical, were applied to fit the data and describe the dehydration of beetroot to powder. None of the models were a poor fit, with the R<sup>2</sup> values ranging from 0.905 to 0.991. The models are summarized in Table 3.1.

The Henderson and Pabis Model, a semi-theoretical model, fit the data well with an average R<sup>2</sup> value of 0.974 for the six different temperature/thickness combinations. Figure 3.2 displays the data and fitted models. The strong fit of this model (high R<sup>2</sup> value) indicates that there is a relationship between both sample thickness and temperature and the amount of time that it takes for the sample to reach the designated moisture content.

As the coefficient k from the Henderson and Pabis Model (Equation 3.6) represents the coefficient  $\frac{-(2n-1)^2\pi^2D_{eff}t}{4L^2}$  from Equation 3.3, it is evident that there exists an inverse-square relationship,  $k \sim \frac{1}{L^2}$ , and therefore the thicker that the sample (L) is, the lower the coefficient k will be. With the increase in thickness of a sample and in turn a lower coefficient k, the amount of time, t, which it takes to reach a desired moisture content and therefore moisture ratio increases, as stated in Equation 3.6. This is evident in the k values experimentally determined, as the k values for samples of thickness 0.58 cm are greater than those of samples of thickness 1.07

cm. The ratio of  $\frac{L_{thin}^2}{L_{thick}^2} = \frac{0.58^2}{1.07^2} = 0.3$ , however, the ratio of k values between samples of thickness 1.07 cm and 0.58 cm for all three temperatures at which samples were dried at is approximately 0.5. This is due to other factors contributing to the k values such as the coefficient of diffusion,  $D_{eff}$ . The  $D_{eff}$  values for each model were calculated by Equation 3.6 and ranged from  $1.32 \times 10^{-5}$  to  $3.95 \times 10^{-5} \text{ cm}^2/\text{s}$ , increasing with temperature at which the samples were dried and decreasing with increased thickness of the samples. This is, of course, an oversimplification of the drying process as the rate of diffusion constantly changes as drying occurs. These values are higher than the diffusion coefficient of diced sugar beets, which have a value of  $0.4\text{-}1.3 \times 10^{-6} \text{ cm}^2/\text{s}$  (Doulia et al., 2000).

The temperature at which the sample is dried at also affects the coefficient k in the Henderson and Pabis Model. From the Arrhenius Equation (Equation 3.1), it is evident that the effective diffusion coefficient is dependent on temperature,  $D_{eff} \sim \exp(\frac{1}{T})$  and the relationship shows that as temperature is increased,  $D_{eff}$  decreases. After once again replacing the coefficient k in the Henderson and Pabis Model with its corresponding coefficient from Equation 3.3, it is evident that the relationship  $MR \sim \exp(D_{eff} \cdot t)$  exists. With an increase in temperature at which a sample is dried at, the  $D_{eff}$  value is increased and therefore the coefficient k is increased. An increased k value indicates that the amount of time that it takes for the sample to be dried to the desired moisture content is decreased. This is evident with a trend of increasing k values for samples of the same thicknesses which are dried at increasing temperatures (e.g. k= 0.01075, 0.01364, 0.01716 for samples of thickness 0.42 cm at 75, 85, and 95°C)

#### Drying Ratio, Productivity, and Drying Rate

The drying ratio, productivity, and drying rate of each sample was measured when the powder was dehydrated to  $MR=0.10$ . Results are summarized in Table 3.2. The drying ratio for

all samples was 4.61. The productivity of powders increased with increasing temperature at which samples were dried and decreased thickness of sample. The drying rate of powders ranged from 10.42 to 19.57 g/ hr. Powders dried at higher temperatures had faster drying rates, as did samples which were less thick. The sample that was 0.58 cm in thickness and dried at 95°C had the fastest drying rate and the sample that was 1.07 cm in thickness and dried at 75°C had the slowest drying rate. Cai et al. (2000) discovered a higher drying rate and higher powder productivity with increased inlet-air temperature of spray-dried *Amaranthus*.

#### Water Activity

The water activity of six samples from each temperature-maltodextrin level content was tested. The water activity levels varied from 0.24 (95°C, 0.3 g maltodextrin/g dry solids) to 0.32 (75°C, 0.6 maltodextrin/ g dry solids). The average water activity of the powders dried at each temperature were not significantly different (0.261, 0.257, 0.254 at 75, 85, 95° C), however the average water activity of powders with 0 g maltodextrin/g dry solids (0.261) was lower than the average water activities of powders with 0.3 and 0.6 g maltodextrin/g dry solids (0.322, 0.303).

#### Moisture Content

The moisture content of the samples is displayed in Table 3.3. Samples contained 0.016 to 0.019 g water/ g powder and no samples had moisture contents that were significantly different from each other. This implies that the level of maltodextrin added to the sample and the temperature at which the beets were dried at did not affect the moisture content of the powders. Grabowski et al. (2006), Abadio et al. (2004), and Goula et al. (2004) found that the moisture content of powders made by spray drying sweet potato puree, pineapple juice, and tomato paste decreased with increased maltodextrin concentration. However, the previously mentioned

experiments used higher quantities of maltodextrin implying that the addition of more maltodextrin to the beet samples may have yielded similar results.

### Color

The lightness ( $L^*$ ), chroma ( $c^*$ ), and hue angle ( $h$ ) were measured on each sample ( $n=9$ ). Results are summarized in Table 3.3. The lightness of samples values, with  $L^*=0$  indicating black to  $L^*=100$  indicating white, ranged from 24.8 to 29.3. The lightness of the raw beets prior to dehydration was much lower, at 19.79. This implies that samples got darker from the removal of water during dehydration. The  $L^*$  values did not differ based on the temperatures at which samples were dried at, with values of 25.7, 25.6, and 24.8 for samples dried at 75, 85, and 95°C. The lightness of the samples was greater for samples with maltodextrin levels of 0.3 and 0.6 g maltodextrin/ g dry solids (29.2, 27.7) than for samples with 0 maltodextrin (25.7). This is due to the addition of maltodextrin, which has a lightness value of 96.6. The hue angles, with values from 346-358°, were in the purple-red to red range for samples dried in all conditions. This characteristic reddish color of beets comes from their betalain content, as betalains are heat labile compounds and are subject to degradation under elevated temperatures, resulting in a change in color from red to brown. The chroma and hue angle did not vary between samples; as neither the temperature that samples were dried at nor did samples' maltodextrin content affect these values. This indicates that the amount of heat supplied to the beets during processing was not enough for significant degradation of the betalain pigments.

### Bulk Density

The temperature at which beets were dehydrated at did not affect the bulk density of the powders, as the average bulk densities of powders dried at 75, 85, and 95 degrees were 0.67, 0.67, and 0.66 g/mL. The addition of maltodextrin, however, did affect the bulk density of the

powders as the bulk densities of powders with 0, 0.3, and 0.6 g maltodextrin/ g dry solids were 0.68, 0.74, and 0.74. This is due to the fact that maltodextrin helps prevent the formation of clumps, which occupy more volume. In general, addition of anticaking agents increases the bulk density of powders (Peleg, 1983). Goula et al. (2008) found that an increase in maltodextrin content in spray-dried tomato powders had decreased bulk density, possibly due to the maltodextrin minimizing the effect of thermoplastic particles from sticking together. Cai et al. (2000) found that spray-dried beet powders with higher maltodextrin contents had lower bulk densities.

### Flowability

Flowability of powders was measured with a rotating cylindrical drum; Table 3.3 displays the percentage of powder that emerged from the cylinder in 4 and 20 s and Figure 3.4 displays plots of the amount of powder which flowed out of the cylinder over time. Flowability is an important property of powders as movement through pipes and packaging depends on it. The total amount of powder that emerged from the cylinder ranged from 85.2% to 95.0% of the total 5 g. The full five g of powder did not emerge from any due to adhesion to the surfaces of the cylinder and moisture absorption from ambient surroundings.

The amount of powder that flowed out of the drum after 4 s varied from 70.5 to 84.5%. Maltodextrin content, with averages of 85.9, 90.2, and 92.8% of total powder emerging from the drum from powders with 0, 0.3, 0.6 g maltodextrin/ g dry solids, was a significant factor. The more maltodextrin present, the more powder fell out of the drum after 4 s as well. This result comes from maltodextrin's anti-caking effect which helped the powder remain more free-flowing. Flowability of soy sauce powder with the addition 20% and 40% maltodextrin was not found by Wang et al. (2012) to be statistically different. Kim et al. (2012) discovered that

increased maltodextrin levels resulted in more dried blueberry slurry powder flowing out of the drum.

The different temperatures at which powders were dried at did not have a large effect on the flowability of the powders. After 4 s, there was no difference in the amount of sample that came out of the drum. After 20 s, 91.3% of powder dried at 85°C came out of the drum but only 85.2% of powder dried at 95°C. This difference may be due to large variability between samples. Yan (2012) found the flowability of vacuum belt-dried apple pomace powder to be highest for samples dried at 80°C and lowest from samples dried at 110°C.

#### Dynamic Hygroscopicity

The hygroscopicity of three samples of each maltodextrin level of powders dried at 95°C was evaluated; results are displayed in Table 3.5 and Figure 3.5. All powders got darker in color and caked during the eight hr they spent at 75% relative humidity. All powders rapidly adsorbed moisture for the first three hr in the chamber then slowly adsorbed moisture until finally reaching equilibrium at approximately 8 hr.

While the powder with 0.6 g maltodextrin/ g dry beet solids adsorbed less moisture than the powders with 0 g and 0.3 g maltodextrin/ g dry solids (19.4, 22.0, 21.8 g H<sub>2</sub>O/ 100 g powder), the equilibrium moisture contents of the beet powders were not significantly different. The k values obtained from the moisture isotherm models ranged from 0.0139 to 0.0159 but were not significantly different; implying that the level of maltodextrin added to the sample did not affect how quickly it absorbed moisture. Cai et al. (2000) discovered that the addition of 10 DE maltodextrin to spray-dried beet powders resulted in a lower hygroscopicity than the control powder without maltodextrin. Kim et al. (2012) found that increasing levels of maltodextrin in vacuum-belt dried blueberry slurries led to less hygroscopic powders and different temperatures

at which samples were dried did not have an effect on the hygroscopicity of the powders. It was discovered that hygroscopicity decreased with an increase in maltodextrin content by Goula et al. (2008) with spray-dried tomato powders and Rodriguez-Hernandez et al. (2005) with cactus pear powder.

### Moisture Isotherms

Samples with the addition of different levels of maltodextrin that were dried at 95°C were placed in chambers of relative humidity levels of 11-85%; results are displayed in Figure 3.6 and a summary of the models which were fit to the data is displayed in Table 3.6. Powders adsorbed moisture for 25 days, at the end of which powders at high relative humidity levels such as 75% and 85% caked and became darker in color while powders at low relative humidity levels maintained their color and free-flowing properties. The moisture isotherm curves generated were J-shaped due to the crystalline structure of the sugar-containing powder. They follow the shape of the Type 3, or Flory-Huggins isotherm, which accounts for adsorption of a solvent or plasticizer above the glass transition temperature (Mathlouthi et al., 2003).

The Guggenheim-Anderson-de Boer Model (GAB) was fit for all three data sets, with strong  $R^2$  values of 0.98-99. The GAB model showed monolayer values of 0.0888, 0.0709, and 0.0642 g water/ g dry solid for powders with the addition of 0, 0.3, and 0.6 g maltodextrin/g dry solids. The monolayer moisture content of a powder represents the amount of water strongly adsorbed to the food matrix, which is important in determining the stability of food products (Catelam, 2011). The addition of maltodextrin leads to a decrease of active sites on beet particles which are available to form bonds with water. This is in agreement with Catelam et al. (2011) for spray-dried and freeze-dried passion fruit pulp, Moraga (2006) for freeze-dried kiwi, and Tonon et al. (2009) for spray-dried acerola and acai powders.

### Total Betalain Quantification

The total betalain content of beet powders was measured by red betacyanin (reported as betanin) and yellow betaxanthin (reported as vulgaxanthin-I) content and is reported in Table 3.4. The betacyanins in beets consist of 75-95% betanin and the betaxanthins comprise roughly 95% vulgaxanthin (Francis, 1999; Piatelli, 1981). Betacyanin content ranged from 0.29 to 0.55 mg betanin per g powder, dry basis. Betacyanin content was highest for samples without the addition of maltodextrin (0.55 mg betanin/g powder, dry basis) and lower for samples with 0.3 and 0.6 g maltodextrin/ g dry solid (0.41, 0.32 mg betanin/ g powder, dry basis). Although betacyanins are heat labile compounds, there was no difference in betanin content between samples dried at 75, 85, and 95°C. The betaxanthin content of the beet powders ranged from 0.11 to 0.27 mg vulgaxanthin/ g beet powder, dry basis. The betaxanthin content was also highest for samples without the addition of maltodextrin (0.27 mg vulgaxanthin/ g powder, dry basis) than for samples with the addition of 0.3 and 0.6 g maltodextrin/ g dry solids (0.20, 0.14 mg vulgaxanthin/ g powder, dry basis). The betaxanthin content of the beet powders also did not differ between samples dried at different temperatures. The difference in betacyanin and betaxanthin content of powders due to the addition of maltodextrin, is because maltodextrin is a polysaccharide that does not contain any betalains.

The amount of betacyanins and betaxanthins present in the beet powders was also measured on the basis of dry beet solids, to determine if the addition of maltodextrin changed the betalain content of the powders or just diluted the amount. The betacyanin content varied from 0.47 to 0.58 mg betanin/ g beet solids, however none of the samples were significantly different. The betaxanthin content ranged from 0.18 to 0.29 mg vulgaxanthin/ g beet solid, with only the lowest and highest value (powder dried at 95°C with 0.3 g maltodextrin/ g beet solids and

powder dried at 95°C with 0.6 g maltodextrin/ g dry beet solid) different from one another.

Betain, the most prominent betalain in beets, can undergo many forms of degradation during thermal processing, including isomerization, decarboxylation, or cleavage by heats and acids (Herbach et al., 2004a). The lack of difference between betacyanin and betaxanthin content between samples dried at different temperatures suggests that the amount of heat transferred to the samples during dehydration was not enough to degrade the pigment.

### **Conclusion**

Crushed beets were dried into powders with a vacuum belt dryer at three temperatures (75, 85, 95°C) and with the addition of three levels of maltodextrin (0, 0.3, 0.6 g maltodextrin/ g beet solids). Thinner samples had higher drying rates. Temperature was an important factor in drying rate, however it did not affect physical properties or betalain content of powders. Level of maltodextrin was an important factor in flowability and hygroscopicity of powders, higher levels lead to increased flowability and decreased hygroscopicity. The betalain content of powders with maltodextrin had a lower betalain content than powders without maltodextrin on a total dry solids basis but not different on a total beet solids basis. Beet powder dried at 95°C with 0 g maltodextrin/ g dry solids is optimal within test conditions for creating a value-added food ingredient as it dries quickly and has favorable physical properties and betalain content.

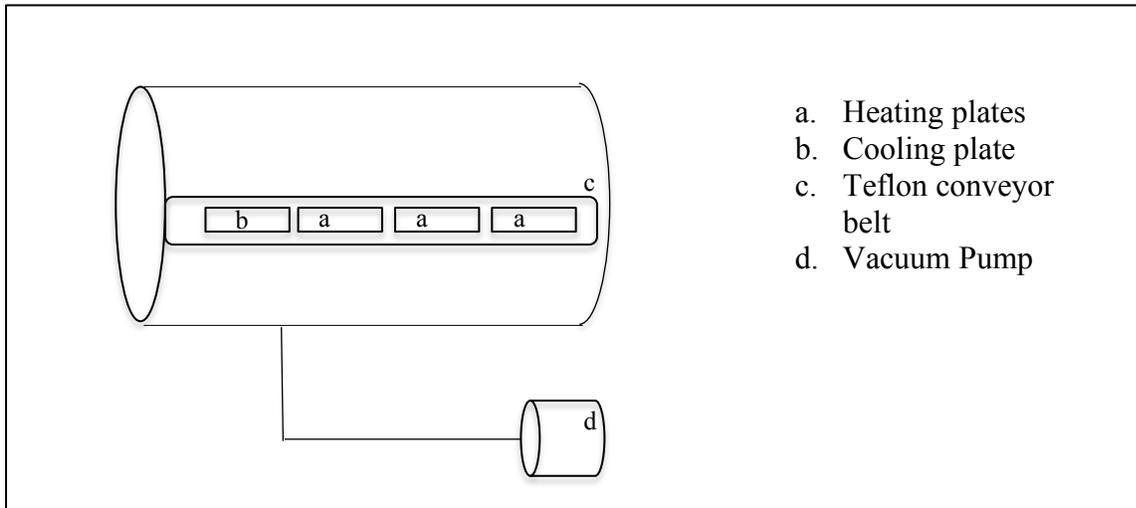


Figure 3.1. Schematic of vacuum belt dryer.

Figure 3.2: Fit of Henderson and Pabis model to dehydration data

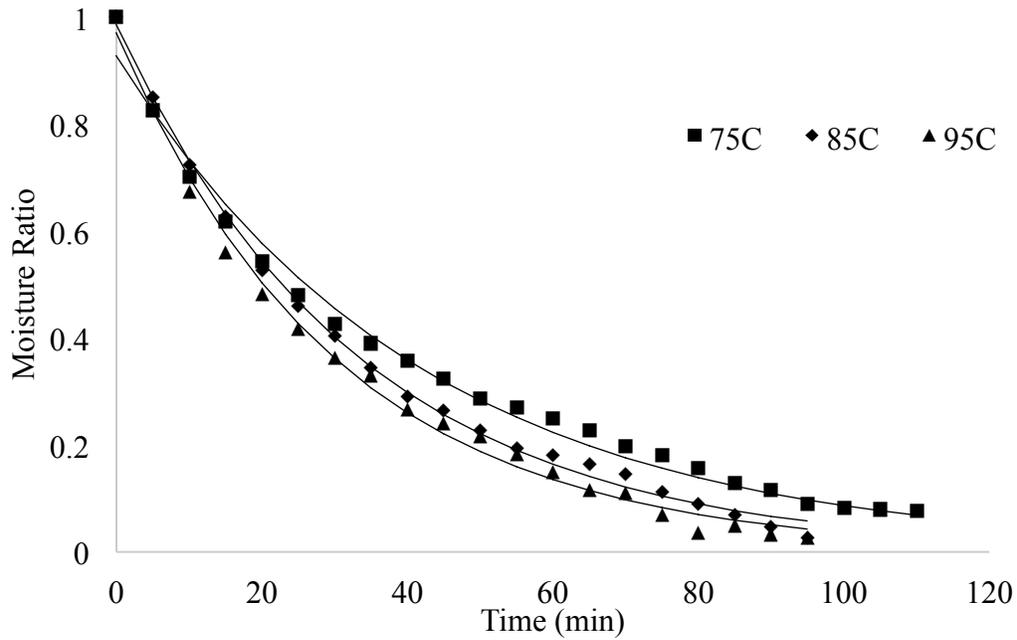


Figure 3.2a: Model of dehydration of beets, thickness 0.58 cm.

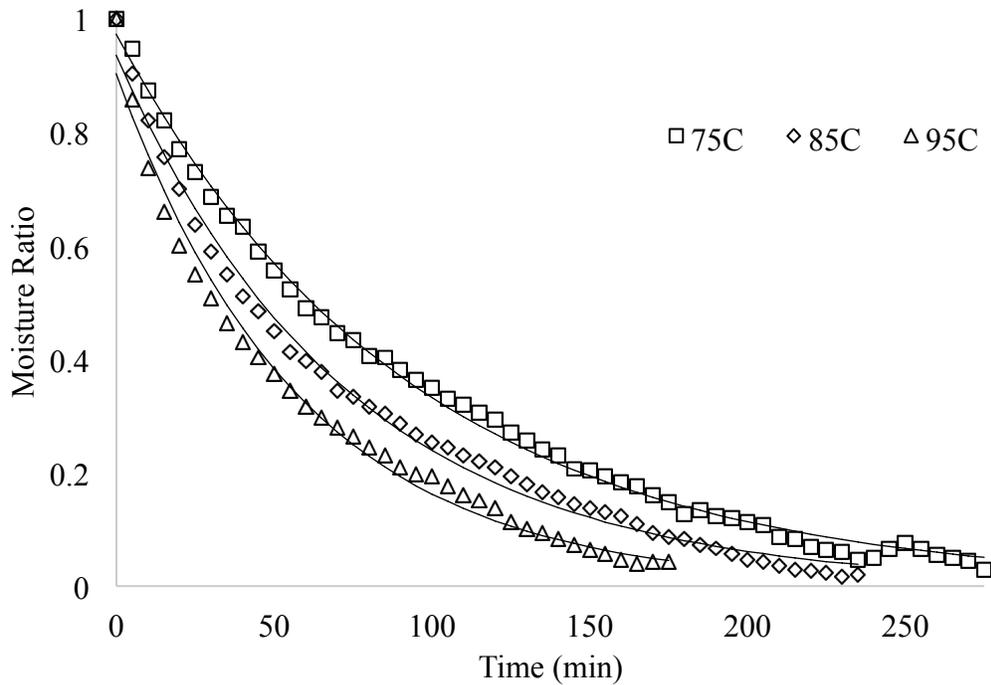


Figure 3.2b: Model of dehydration of beets, thickness 1.07 cm.

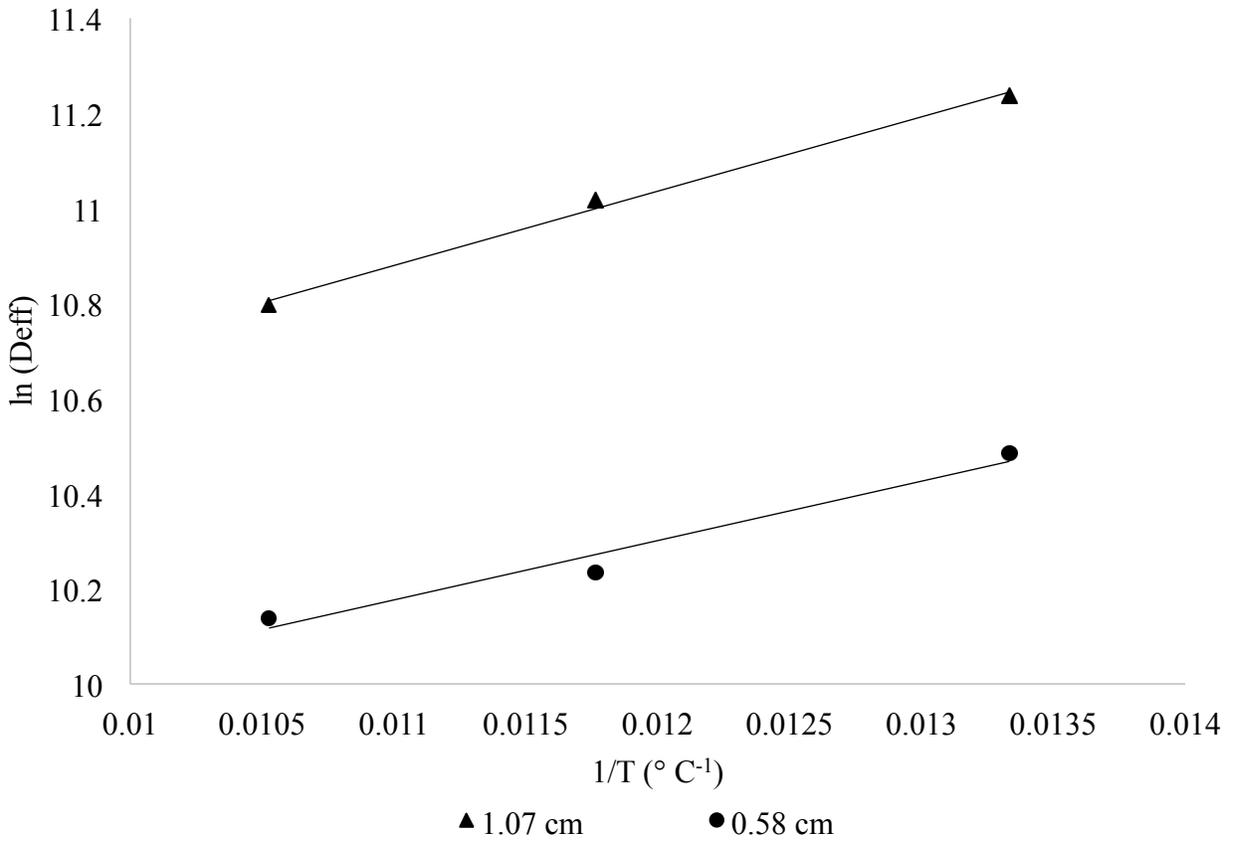


Figure 3.3: Diffusion coefficient of beet samples vs.  $1/T$ .

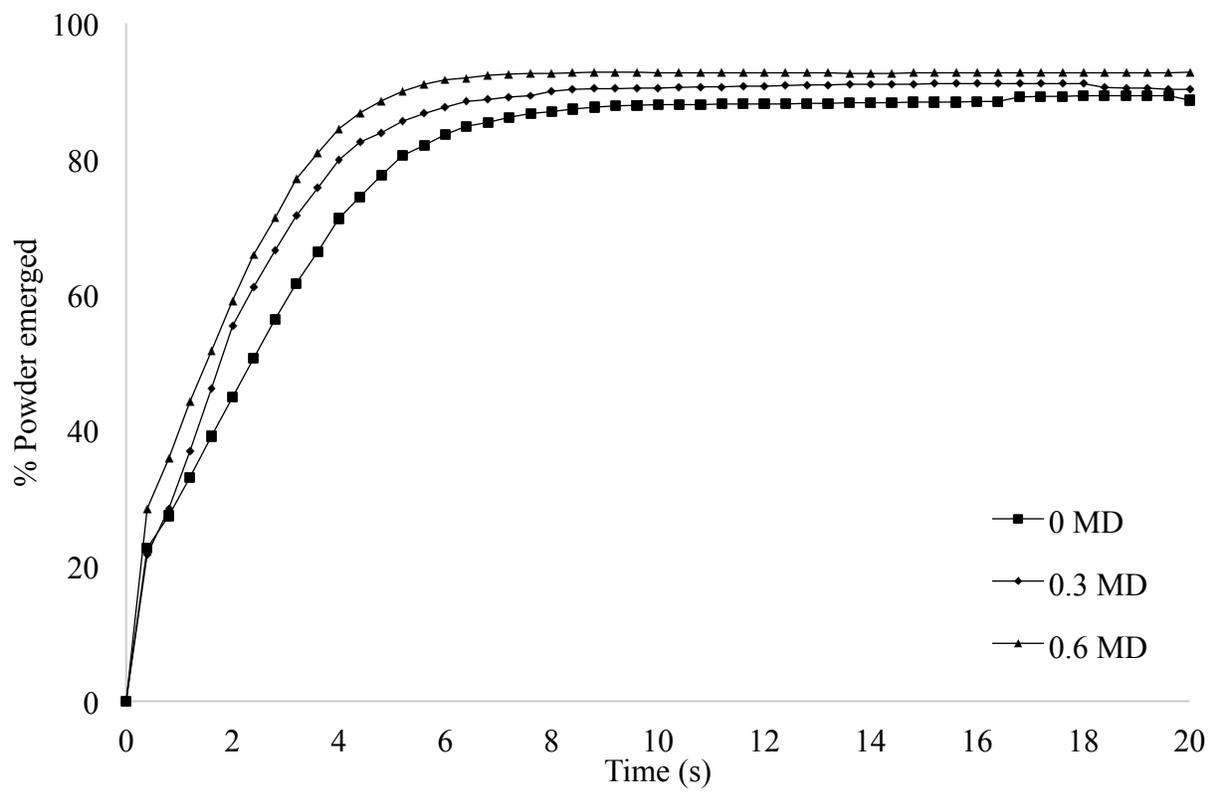


Figure 3.4 Flowability of beet powders dried at 75°C with the addition of different levels of maltodextrin.

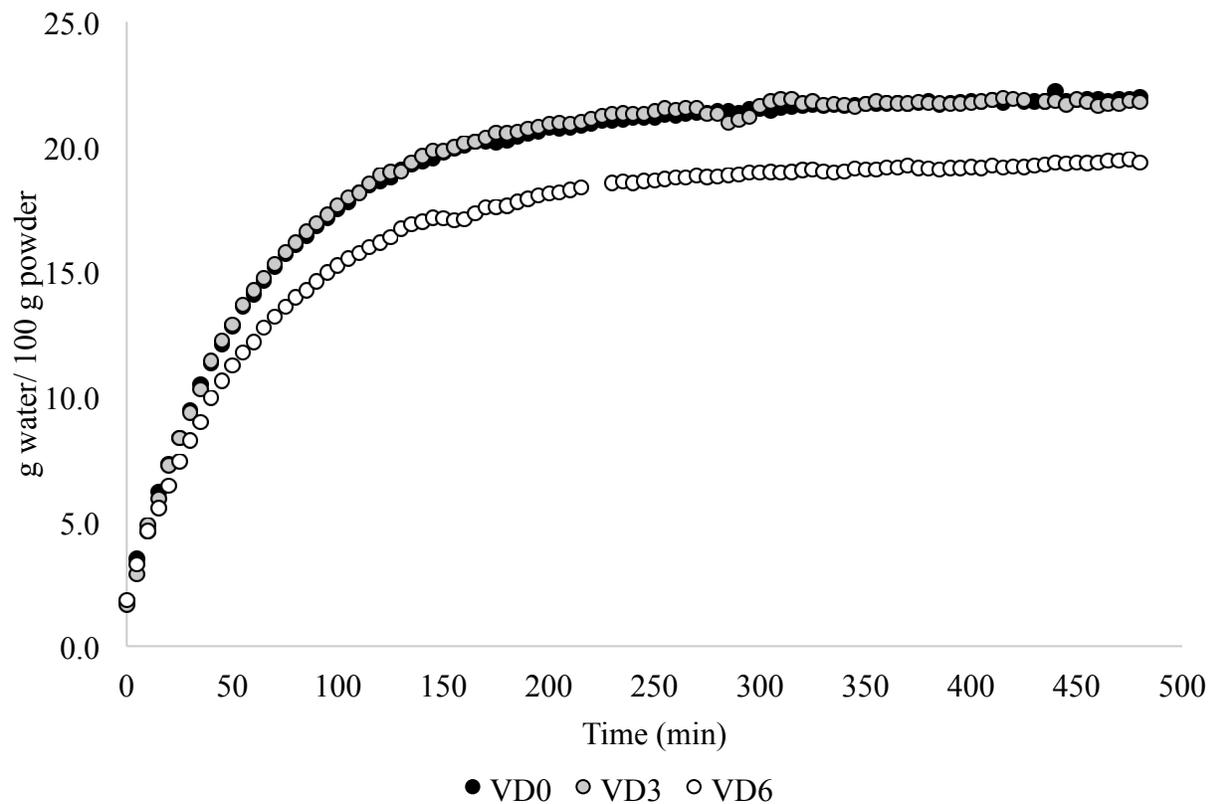


Figure 3.5: Dynamic hygroscopicity- moisture content of beet powders with three levels of maltodextrin as they adsorb moisture over time.

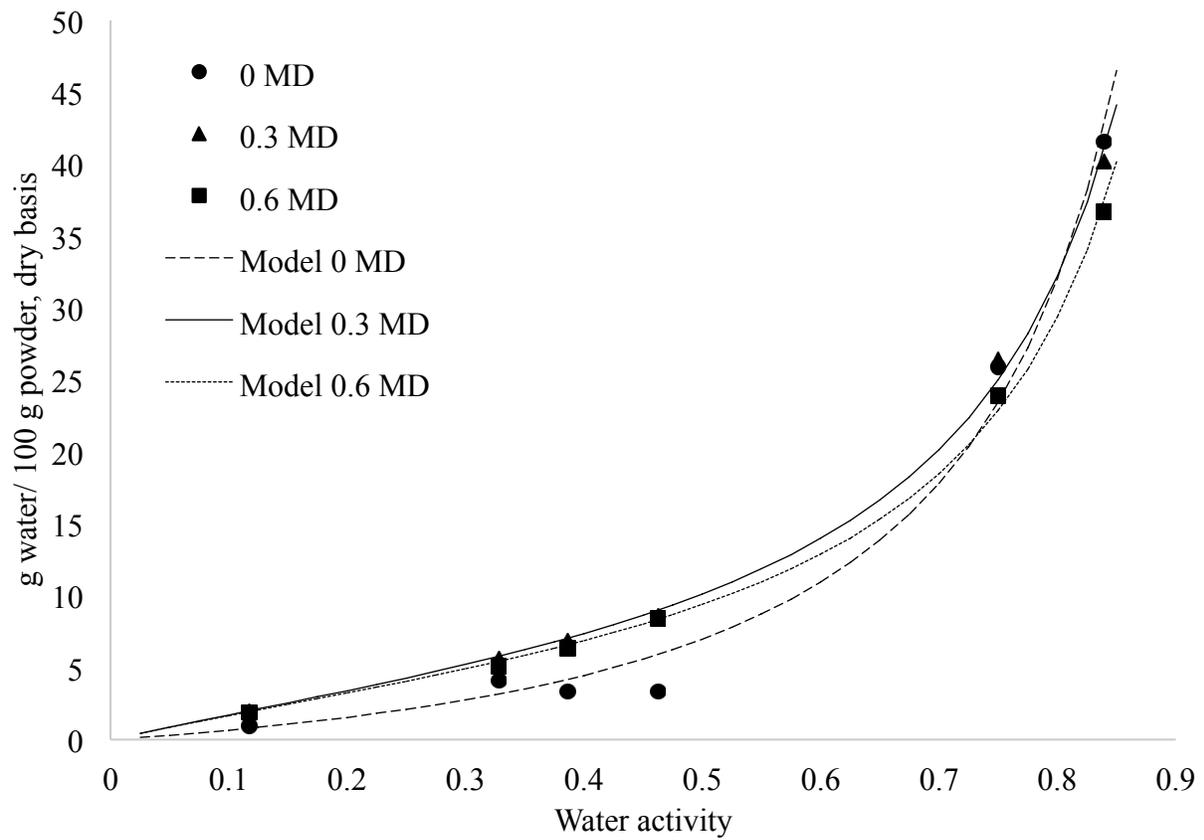


Figure 3.6: Moisture isotherms of beet powders with different levels of maltodextrin dried at 95°C.

Table 3.1: Models for dehydrating beetroot of two thicknesses at three temperatures

Model Name	Equation	Thick-ness (cm)	T (°C)	Coefficients and Constants	R <sup>2</sup>
Newton	$MR = \exp(-kt)$	0.58	75	k= 0.0258	0.941
		0.58	85	k= 0.03034	0.978
		0.58	95	k=0.0339	0.972
		1.07	75	k=0.01107	0.979
		1.07	85	k=0.01468	0.980
		1.07	95	k=0.01924	0.964
Page	$MR = \exp(-kt^n)$	0.58	75	k=0.05125, n=0.8205	0.956
		0.58	85	k=0.03471, n=0.9632	0.979
		0.58	95	k=0.0441, n=0.9268	0.974
		1.07	75	k=0.01373, n=0.9538	0.981
		1.07	85	k=0.0265, n=0.8663	0.988
		1.07	95	k=0.04488, n=0.7960	0.986
Henderson and Pabis	$MR = a \cdot \exp(-kt)$	0.58	75	a=0.9274, k=0.02373	0.949
		0.58	85	a=0.9866, k=0.02991	0.978
		0.58	95	a=0.9707, k=0.03287	0.973
		1.07	75	a=0.9731, k=0.01075	0.981
		1.07	85	a=0.9353, k=0.01364	0.985
		1.07	95	a=0.9025, k=0.01716	0.978
Two-term	$MR = a \cdot \exp(-kt) + b \cdot \exp(-gt)$	0.58	75	a=0.8162, k=0.0292, b=0.1855, g=0.1532	0.957
		0.58	85	a=0.9544, k=0.02904, b=0.0466, g=0.1508	0.979
		0.58	95	a=0.9139, k=0.03109, b=0.0890, g=0.2089	0.975
		1.07	75	a=0.9521, k=0.01052, b=0.0538, g=0.1182	0.981
		1.07	85	a=0.8458, k=0.01245, b=0.1582, g=0.0783	0.989
		1.07	95	a=0.7949, k=0.01516, b=0.2089, g=0.1293	0.988
Wang and Singh	$MR = 1 + at + bt^2$	0.58	75	a=-0.02018, b=0.0001127	0.905
		0.58	85	a=-0.02302, b=0.0001427	0.956
		0.58	95	a=-0.02453, b=0.0001576	0.939
		1.07	75	a=-0.00835, b=0.00001869	0.957
		1.07	85	a=-0.01048, b=0.00002879	0.924
		1.07	95	a=-0.01395, b=0.00005167	0.899

Model Name	Equation	Thick-ness (cm)	T (°C)	Coefficients and Constants	R <sup>2</sup>
Two-term Exponential	$MR = a \cdot \exp(-kt) + (1 - a) \cdot \exp(-kat)$	0.58	75	a=0.1758, k=0.1214	0.957
		0.58	85	a=0.6318, k=0.03622	0.978
		0.58	95	a=0.5266, k=0.04698	0.973
		1.07	75	a=0.6597, k=0.10283	0.980
		1.07	85	a=0.1537, k=0.08101	0.989
		1.07	95	a=0.1832, k=0.08647	0.985
Verma et al.	$MR = a \cdot \exp(-kt) + (1 - a) \cdot \exp(-kgt)$	0.58	75	a=0.1841, k=0.1516, g=0.1380	0.957
		0.58	85	a=0.04665, k=0.1441, g=0.2013	0.979
		0.58	95	a=0.08602, k=0.205, g=0.1517	0.975
		1.07	75	a=0.04775, k=0.1104, g=0.09526	0.981
		1.07	85	a=0.1541, k=0.0783, g=0.1591	0.989
		1.07	95	a=0.1663, k=0.01895, g=1.0116	0.964
Modified Henderson and Pabis	$MR = a \cdot \exp(-kt) + b \cdot \exp(-gt) + c \cdot \exp(-ht)$	0.58	75	a=-11.82, k=0.4003, b=12.45, g=0.4182, c=-0.3498, h=0.6891	0.925
		0.58	85	a=1.231, k=0.3938, b=-0.9673, g=0.2415, c=0.00866, h=0.2559	0.961
		0.58	95	a=2.009, k=-0.052, b=0.05026, g=1.612, c=-1.838, h=-0.1069	0.976
		1.07	75	a=-1.986, k=-0.5323, b=0.4663, g=0.4876, c=1.766, h=-0.586	0.973
		1.07	85	a=-14.1, k=0.1694, b=14.25, g=0.1758, c=0.07243, h=1.106	0.977
		1.07	95	a=1.37, k=0.5506, b=-0.1923, g=0.5535, c=-0.96, h=0.4656	0.970

Model Name	Equation	Thick-ness (cm)	T (°C)	Coefficients and Constants	R <sup>2</sup>
Logarithmic	$MR = a \cdot \exp(-kt) + c$	0.58	75	a=0.9001, k=0.02763, c=0.04878	0.952
		0.58	85	a=0.9879, k=0.02972, c=0.00223	0.978
		0.58	95	a=0.9748, k=0.03218, c=0.00719	0.973
		1.07	75	a=0.9841, k=0.01018, c=0.01932	0.981
		1.07	85	a=0.9299, k=0.01419, c=0.01204	0.986
		1.07	95	a=0.8864, k=0.0194, c=0.0357	0.979
Midilli-Kucuk	$MR = a \cdot \exp(-kt^n) + bt$	0.58	75	a=1.125, k=0.05995, n=0.7216, b=-0.1201	0.956
		0.58	85	a=1.065, k=0.04092, n=0.8857, b=-0.05928	0.979
		0.58	95	a=0.12, k=0.05589, n=0.7985, b=-0.1133	0.977
		1.07	75	a=1.118, k=0.01971, n=0.8331, b=-0.1082	0.982
		1.07	85	a=1.119, k=0.3703, n=0.7469, b=-0.1029	0.991
		1.07	95	a=1.133, k=0.05663, n=0.6833, b=-0.1252	0.988
Modified Henderson and Perry	$MR = a \cdot \exp(-kt^n)$	0.58	75	a=0.9939, k=0.04967, n=0.8271	0.956
		0.58	85	a=0.998, k=0.03432, n=0.9657	0.979
		0.58	95	a=0.9930, k=0.04253, n=0.9350	0.974
		1.07	75	a=0.9818, k=0.01196, n=0.9791	0.981
		1.07	85	a=0.9877, k=0.023389, n=0.8875	0.987
		1.07	95	a=0.9877, k=0.0421, n=0.8085	0.986

Table 3.2: Diffusion coefficient ( $D_{\text{eff}}$ ), drying ratio, productivity, and drying rate of beet powders of different thicknesses dried at different temperatures

Temperature (°C)	Thickness (cm)	$D_{\text{eff}}$ ( $\text{cm}^2/\text{s}$ )	Drying ratio	Productivity (g/hr)	Drying Rate (g/hr)
75	0.58	$2.80 \times 10^{-5}$	4.61 <sup>a</sup>	3.5 <sup>d</sup>	12.4 <sup>d</sup>
85	0.58	$3.59 \times 10^{-5}$	4.61 <sup>a</sup>	4.3 <sup>bc</sup>	15.4 <sup>bc</sup>
95	0.58	$3.95 \times 10^{-5}$	4.61 <sup>a</sup>	5.4 <sup>a</sup>	19.6 <sup>a</sup>
75	1.07	$1.32 \times 10^{-5}$	4.61 <sup>a</sup>	3.0 <sup>d</sup>	10.8 <sup>d</sup>
85	1.07	$1.64 \times 10^{-5}$	4.61 <sup>a</sup>	3.6 <sup>cd</sup>	12.9 <sup>cd</sup>
95	1.07	$2.05 \times 10^{-5}$	4.61 <sup>a</sup>	4.6 <sup>ab</sup>	16.4 <sup>ab</sup>

<sup>a</sup> Values within a column with same letters indicates values are not statistically different ( $p < 0.05$ ).

Table 3.3: Water activity, moisture content, color, and bulk density of beet powders dried at different temperatures with different levels of maltodextrin

Temp. (°C)	g maltodextrin/ g dry solids	a <sub>w</sub>	g water/ 100 g powder	Color			Bulk density (g powder/ mL)
				L*	c*	h (°)	
75	0	0.26 <sup>bcd</sup>	1.78 <sup>a</sup>	25.7 <sup>bc</sup>	28.8 <sup>a</sup>	357.5 <sup>a</sup>	0.68 <sup>bc</sup>
	0.3	0.30 <sup>ab</sup>	1.71 <sup>a</sup>	29.2 <sup>a</sup>	27.9 <sup>a</sup>	355.9 <sup>a</sup>	0.75 <sup>a</sup>
	0.6	0.32 <sup>a</sup>	1.64 <sup>a</sup>	27.7 <sup>ab</sup>	29.9 <sup>a</sup>	356.4 <sup>a</sup>	0.74 <sup>a</sup>
85	0	0.26 <sup>bcd</sup>	1.86 <sup>a</sup>	25.6 <sup>bc</sup>	28.7 <sup>a</sup>	357.2 <sup>a</sup>	0.67 <sup>c</sup>
	0.3	0.27 <sup>abcd</sup>	1.80 <sup>a</sup>	27.6 <sup>abc</sup>	29.3 <sup>a</sup>	354.8 <sup>a</sup>	0.72 <sup>abc</sup>
	0.6	0.30 <sup>abc</sup>	1.72 <sup>a</sup>	29.0 <sup>a</sup>	30.6 <sup>a</sup>	356.3 <sup>a</sup>	0.74 <sup>a</sup>
95	0	0.25 <sup>cd</sup>	1.74 <sup>a</sup>	24.8 <sup>c</sup>	28.9 <sup>a</sup>	357.6 <sup>a</sup>	0.67 <sup>c</sup>
	0.3	0.24 <sup>d</sup>	1.62 <sup>a</sup>	29.3 <sup>a</sup>	28.6 <sup>a</sup>	346.0 <sup>a</sup>	0.73 <sup>a</sup>
	0.6	0.27 <sup>abcd</sup>	1.78 <sup>a</sup>	29.3 <sup>a</sup>	30.6 <sup>a</sup>	356.6 <sup>a</sup>	0.73 <sup>ab</sup>

<sup>a</sup> Values within a column with same letters indicates values are not statistically different (p<0.05).

<sup>b</sup> Raw beet sample: L\*, c\*, h =19.79, 15.49, 359.02

Maltodextrin: L\*, c\*, h = 96.56, 1.97, 209.56

Table 3.4: Percent beet powder emerged from drum after 4 and 20 s

Temperature (°C)	g maltodextrin/ g dry solids	% Powder emerged after 4 s	% Powder emerged after 20 s
75	0	70.5 <sup>a</sup>	85.9 <sup>bc</sup>
	0.3	79.8 <sup>a</sup>	90.2 <sup>abc</sup>
	0.6	84.5 <sup>a</sup>	92.8 <sup>ab</sup>
85	0	79.0 <sup>a</sup>	91.3 <sup>abc</sup>
	0.3	84.2 <sup>a</sup>	92.1 <sup>abc</sup>
	0.6	83.8 <sup>a</sup>	95.0 <sup>a</sup>
95	0	76.4 <sup>a</sup>	85.2 <sup>c</sup>
	0.3	84.6 <sup>a</sup>	94.6 <sup>a</sup>
	0.6	78.7 <sup>a</sup>	93.8 <sup>ab</sup>

<sup>a</sup> Values within a column with same letters indicates values are not statistically different (p<0.05).

Table 3.5: Percent hygroscopicity and moisture sorption model for beet powders dehydrated at 95°C with the addition of three levels of maltodextrin

g maltodextrin/ g dry solids	k (min <sup>-1</sup> )	R <sup>2</sup>	m <sub>e</sub> (g water/ 100 g powder)	% hygroscopicity
0	0.0147 <sup>a</sup>	0.998	22.0 <sup>a</sup>	18.0 <sup>a</sup>
0.3	0.0159 <sup>a</sup>	0.999	21.8 <sup>a</sup>	18.0 <sup>a</sup>
0.6	0.0139 <sup>a</sup>	0.998	19.4 <sup>a</sup>	16.2 <sup>a</sup>

<sup>a</sup> Values within a column with same letters indicates values are not statistically different (p<0.05).

Table 3.6: Guggenheim-Anderson-de Boer (GAB) model of moisture isotherms of beet powders with three levels of maltodextrin dried at 95°C

g maltodextrin/ g dry solids	M <sub>0</sub> (g water/ 100 g powder)	c	R <sup>2</sup>
0	8.88	0.648	0.989
0.3	7.09	2.488	0.998
0.6	6.42	2.713	0.998

<sup>a</sup> Values within a column with same letters indicates values are not statistically different (p<0.05).

Table 3.7: Total betalain quantification of beet powders dried in a vacuum belt dryer at three temperatures and with three maltodextrin levels

Temperature (°C)	g maltodextrin/g dry solids	mg betanin/g powder, dry basis	mg vulgaxanthin/g powder, dry basis	mg betanin/g beet solids, dry basis	mg vulgaxanthin/g beet solids, dry basis
75	0	0.55 <sup>a</sup>	0.27 <sup>a</sup>	0.55 <sup>a</sup>	0.27 <sup>ab</sup>
75	0.3	0.41 <sup>bc</sup>	0.20 <sup>bcde</sup>	0.53 <sup>a</sup>	0.26 <sup>ab</sup>
75	0.6	0.32 <sup>cd</sup>	0.14 <sup>def</sup>	0.51 <sup>a</sup>	0.23 <sup>ab</sup>
85	0	0.49 <sup>ab</sup>	0.24 <sup>abc</sup>	0.48 <sup>a</sup>	0.24 <sup>ab</sup>
85	0.3	0.40 <sup>bcd</sup>	0.18 <sup>cdef</sup>	0.51 <sup>a</sup>	0.23 <sup>ab</sup>
85	0.6	0.32 <sup>cd</sup>	0.14 <sup>ef</sup>	0.51 <sup>a</sup>	0.22 <sup>ab</sup>
95	0	0.52 <sup>a</sup>	0.26 <sup>ab</sup>	0.52 <sup>a</sup>	0.26 <sup>ab</sup>
95	0.3	0.44 <sup>abc</sup>	0.23 <sup>abcd</sup>	0.58 <sup>a</sup>	0.29 <sup>a</sup>
95	0.6	0.29 <sup>d</sup>	0.11 <sup>f</sup>	0.47 <sup>a</sup>	0.18 <sup>b</sup>

<sup>a</sup> Values within a column with same letters indicates values are not statistically different (p<0.05).

## CHAPTER 4

### EVALUATION AND COMPARISON OF CHEMICAL AND PHYSICAL PROPERTIES OF BEET POWDERS PRODUCED BY DIFFERENT DRYING METHODS

#### **Introduction**

Beetroot contains phytochemicals called betalains, which are naturally occurring red and yellow pigments. Due to their stability across the pH scale, betalains are used as a source of natural color in food such as yogurts, ice cream, candies, and jellies in either powder or liquid form. The dehydration of beets into powder is a method of increasing and diversifying products available for consumption. However, betalains are heat labile, thus creating a challenge for processors.

The dehydration of beets through forced air convective drying and spray drying have been studied extensively. Gokhale et al. (2012) found optimal conditions of 53°C temperature, 7-mm thickness, and 63% air recirculation ratio for 90% color retention of *Beta vulgaris* with convective dehydration. Beets are generally spray dried with maltodextrin or another carrier to reduce the hygroscopicity of the dried powder. Cai et al. (2000) studied the loss of betacyanins with spray-dried beet juice at different temperatures with different levels of maltodextrin and found only 2.7% degradation at 150°C. Janiszewski et al. found a 26.7-29.3% retention of pigments after spray drying beet juice.

A novel method used to dehydrate different fruit and vegetable products, vacuum belt drying, has been used to make high quality powders. Vacuum belt drying is a semi-continuous

dehydration method which uses low heating and vacuum conditions to dry products at relatively low temperatures. Many vacuum belt dried products have been studied, including banana puree (Wang et al., 2007), fresh squeezed juices (Monzini et al., 1990), *Panax notoginseng* extract (Liu et al., 2011), and blueberry puree (Kim et al., 2012).

The objective of this study was to evaluate crushed beetroot powders dehydrated by six different drying methods, based on their betalain content and physical properties.

## **Materials and Methods**

### Sample preparation

Beets were obtained from Atlanta State Farmer's Market from Victory Farms (Hudsonville, MI). Whole beets were chopped into 1 mm chunks using bowl chopper (Model 33, Schneidmischer, Wallau/Lahn, Germany) with 1% ascorbic acid (w/w). Ascorbic acid was purchased from Prinova US LLC (Carol Stream, IL). The chopped beet product was stored in vacuum-sealed packages in dark at -25°C in dark until needed. Vacuum packages of beets were stored for 24 hours at 4°C before use.

### Drum drying

Samples were loaded between the tops of the drums of a double drum dryer (Model 214-501, Stokes Processing Equipment, Ringwood, Australia). The drums rotated in opposite directions in unison, at 1.13 rpm, with a drum temperature of 100°C. Dried pieces were removed from the drums with a scraper and collected.

### Freeze drying

Samples were freeze-dried using a freeze dryer (REVO Millrock Technology Model RD53S5 Freeze Dryer, Kingston, NY). The program used brought the sample from -40°C to

20°C in 24 hr and the samples were under pressures of 0.013-0.024 kPa. After drying, samples ground for 20 s with a Nutribullet (Nutribullet LLC, Pacomia, CA) extraction blade.

#### Tray drying

Samples were dried according to modified conditions used by Gokhale et al. (2012). Samples were dried in a convective air oven (ALKAR Food Processing Equipment, Lodi, WS) at 53°C for 350 min. The sample was spread to a thickness of 0.5± 0.2 cm. Fan speed was set to 848 rpm, an air velocity of 230 m/ min. After drying, samples were cooled to room temperature and then ground for 20 s with a Nutribullet (Nutribullet LLC, Pacomia, CA) extraction blade.

#### Spray drying

Samples were spray dried according to modified conditions used by Cai et al. (2000). Chopped beets were juiced with a juicer (Cuisinart Model CJE-1000, East Windsor, NJ) before spray drying. 10 DE Maltodextrin (M100, Grain Processing Corporation, Muscatine, IA) was added to the juice to reach a total solids content of 30% (w/w). M100 maltodextrin was obtained from Grain Processing Corporation (Muscatine, IA). Beet juice was spray dried with a spray dryer (Mini Spray Dryer B-290, Büchi, Flawil, Switzerland) with an inlet temperature of 150°C and outlet temperature 89± 2°C. The aspirator was set at 100% and the pump was set to 5%.

#### Vacuum belt drying

Beet samples were dried with a 20.3-cm wide laboratory scale vacuum belt dryer (Zwag, CH-5312 Zchokke Warman Ltd. Bucher, Dottingen, Switzerland) complete with a low friction, Teflon coated fiberglass conveyor belt that passes over three conductive heating plates. These heating zones were all set to 95°C. Either 0 or 0.6 g 10 DE Maltodextrin (M100, Grain Processing Corporation, Muscatine, IA) / g dry solids were added to 175 g of beet samples. Beet samples were loaded into thin aluminum pans at a thickness of 0.05 cm so samples would

maintain their shape throughout dehydration. Pans were placed directly on the belt and dried for 115 min. After drying, samples were taken out of the VBD and cooled to room temperature then ground for 20 s with a Nutribullet (Nutribullet LLC, Pacomia, CA) extraction blade.

#### Moisture Content

Samples were placed in aluminum sample pans and dried in vacuum oven (Model 1430 MS, VWR Scientific Products, Radnor, PA) at 70°C until they reached constant weight to measure moisture content by gravimetric analysis. Moisture content is expressed in terms of percent wet basis, g water per g wet material.

#### Color

Color measurements were taken of the beet powders using a chroma meter (Model CR-410 Minolta Co Ltd., Tokyo, Japan). The HSL color space was used, with L\* representing lightness (low values mean the product is dark, high values mean the product is light), C\* representing chroma (low values mean low brightness, high values mean high brightness), and h representing hue (degree of color on color wheel with 0° representing red, 120° representing green, 240° representing blue).

#### Water Activity

The water activity of each sample was measured by a water activity meter (Aqualab Series 3, Pullman, Washington).

#### Flowability

Flowability was measured as described by Jaya et al. (2004). Five g of each sample were loaded into a rotating drum cylinder, 9 cm in length with a 12 cm diameter with two 4 mm slits, 7 cm in length, on opposite sides. The drum was powered to a direct-drive DC motor power supply (BK Precision Model 1710, Yorba Linda, CA) and operated at 30 rpm. For each sample,

5 g of powder was loaded into the drum. The amount of powder that fell through the slits was recorded continuously for 20 s on an analytical balance (HP 4200C Avery Weigh-Tronix, Fairmont, MN). The total percentage of powder that came out of the drum after 20 s was also recorded.

### Bulk Density

Bulk density was measured by loading 10 g of beet powders into a 25-mL graduated cylinder. The cylinder was held on a digital vortex mixer (Fisher Scientific, Hampton, NH) for 60 s at 600 rpm. The volume of the powder was read and the bulk density of the sample was determined by the mass of the powder divided by the volume of the powder (Grabowski et al., 2006). Three replications of each powder were measured.

### Dynamic Hygroscopicity

Approximately 1.5 g of each powder was spread evenly onto a petri dish, maximizing its surface area. The petri dish was placed on top of an analytical balance (AG204, Mettler Toledo, Greifensee, Switzerland) in an environmental chamber set to 22°C and 75% relative humidity. The mass of the powder was recorded every 5 min until constant masses were obtained. Percent hygroscopicity of powders (HG) was calculated according to:

$$HG = \frac{\frac{b}{a} + w_i}{1 + \frac{b}{a}} \quad (4.1)$$

where  $a$  (g) is the initial powder weight,  $b$  (g) is the increase in weight of powder, and  $w_i$  is the initial moisture content of the sample.

The data was fit to a moisture sorption model:

$$\ln \left( \frac{m_e - m_i}{m_e - m} \right) = k \cdot t + b \quad (4.2)$$

Where  $m_e$  is the equilibrium moisture content,  $m_i$  is the initial moisture content,  $m$  is the moisture content at time  $t$ , and  $k$  is a coefficient that describes the speed of the adsorption of moisture.

#### Particle size

Samples were prepared by being mounted onto stubs and coated with a 0.25-nm layer of gold. A variable pressure scanning electron microscope (Model 1450EP, Carl Zeiss MicroImaging Inc., Thronwood, NY) was used to evaluate the particle size and shape for each powder. Pictures of each powder were taken with the primary detector while the sample was under vacuum.

#### Extraction of Betalains

For extraction of betalains, 0.1 g of beet powder was added to a 10-mL portion of water. The samples were mixed with a vortexer (Standard Mini Vortexer, VWR Scientific Products, Radnor, PA) until homogeneous and then centrifuged with a centrifuge (Centrifuge Model 228, Fisher Scientific, Hampton, NH) for 10 min at 3300 rpm. The supernate was collected and the insoluble portion was re-extracted 2x more and the supernates were combined and brought to a final volume of 50 mL with water in a volumetric flask at room temperature (Kujala et al., 2000; Nemzer et al., 2011).

#### Total Betalain Quantification

Betalain extracts were diluted with 0.05 M phosphate buffer, pH 6.5 so that the samples had an absorbance between 0.4 and 0.5 AU at 538 nm. A spectrophotometer (Spectronic Genesys 2, Thermo Electron Corporation, Madison, WI) was used to measure the absorption of the samples were recorded at 476, 538, and 600 nm. Corrected light absorptions of betanin and vulgaxanthin-I were calculated according to von Elbe et al. (2001):

$$x = 1.095 \times (a - c) \quad (4.3)$$

$$z = a - x \quad (4.4)$$

$$y = b - z - \frac{x}{3.1} \quad (4.5)$$

where

x=light absorption of betanin minus colored impurities

y=light absorption of vulgaxanthin-I corrected for contribution of betanin and colored impurities

z=light absorption of impurities

a= light absorption of sample at 538 nm

b= light absorption of sample at 476 nm

c=light absorption of sample at 600 nm

## **Results and Discussion**

### Water activity

The water activity values of all powders ranged from 0.16 to 0.30, all low enough to eliminate risk of microbial growth (Barbosa-Canovas et al., 1996). The water activities of the freeze-dried powder and the spray-dried powder were the lowest, at 0.21 and 0.16 respectively. The process of freeze-drying involves the dehydration of a product via sublimation, when the sample is under extremely low pressure and at a temperature under its glass transition temperature. This is the industry standard method for the removal of moisture from products. As a result, all unbound water was removed from the freeze-dried powder. During the process of spray-drying, moisture was removed from the sample by atomization of beet juice into a fine mist and then drying it with heat, however due to the dilution with maltodextrin, there were less sites available for water molecules to bind to the dry beet solids and create a monolayer of bound moisture, thus resulting in a low water activity. The remaining powders, produced with the tray

drier, drum drier, and vacuum-belt drier, were in the 0.25 to 0.30 range. The water activity of the vacuum belt-dried powder with added maltodextrin did not differ from the water activity of the powder without maltodextrin, implying that there was not enough maltodextrin added to the sample to have a significant effect.

#### Moisture content

The moisture content of all samples was between 0.015 to 0.02 g water/ g powder. They did not differ based on how samples were dried or what level of maltodextrin was added to them.

#### Color

The lightness, chroma, and hue angle of all beet powders was measured. The lightness of the spray-dried powder was 48.5 while the lightness values of all other powders were in the range of 26.0 to 33.7. The spray-dried powder was a lot lighter than the other powders due to the large amount of maltodextrin that was added to it, as maltodextrin has a lightness value of 96.6. The vacuum-belt dried powder without any maltodextrin added had lightness of 26.0 while the powder with maltodextrin was lighter, at 30.0.

All powders had chroma values between 27.8 and 36.0. The freeze-dried and spray-dried powders had the highest  $c^*$  value, indicating the most saturated color while the tray-dried powder had the lowest  $c^*$  value. This is due to the betalain degradation in the tray-dried powder. The  $h$  value, or hue angle, ranged from 353.4 to 360.7, indicating that all beet powders were in the purple- red range. The spray-dried powder has the lowest  $h$  value, meaning it was purpler than the other powders. This could be due to the spray-dried powder only containing beet juice and not beet solids like the other powders.

### Flowability

Flowability of all powders was measured by the percent of powder that emerged from the drum at 4 s and at 20 s. After 4 s, 35.6% to 78.7% of the 5 g of powder emerged from the cylinder. Greater than 75% of the powder emerged at 4 s for the tray-dried powder and both vacuum belt-dried powders; 50% or less emerged at 4 s for the drum-dried, freeze-dried, and spray-dried powders. This is due to both the size and shape of the particles and the hygroscopicity of the powders. The spray-dried powder and the freeze-dried powder had the lowest water activities and adsorbed moisture from the air, causing agglomeration and adhesion to the walls of the drum. Drum-dried powders also had lower water activity values and also had larger particle sizes and different shapes, also causing resistance to flow. At 20 s the total amount of powder which flowed out of the drum was between 85.6 and 93.8% for all samples. Drying method was not a factor that affected the total amount of powder that emerged from the drum. Drying method did not affect the total amount of powder which emerged, just how rapidly the powder flowed out.

### Bulk Density

The vacuum belt dried powders, both with and without the addition of maltodextrin, as well as the tray-dried powder had the highest bulk densities; 0.73, 0.67, 0.69 g beet powder/ mL. These powders were all dehydrated in pans and then grinded in the Nutribullet for the same amount of time, thus leading to similarly sized particles and therefore bulk densities that are not different. The freeze-dried powder was also prepared in a pan and grinded by the Nutribullet, however, it had the lowest bulk density at 0.43 g powder/ mL. Due to the low water activity and high hygroscopicity of this powder, it is likely that it began to adsorb moisture from the air and

began to stick together. These aggregates of powder were larger in volume than the individual powder particles and therefore did not pack as well into the same volume.

It is probable that this is also the case with the spray-dried powder, which had a bulk density of 0.55 g powder/ mL. The powder exited the spray dryer as very fine particles with a very low water activity, therefore it also adsorbed water from the humidity in the air. The drum-dried powder had a low bulk density as well, 0.46 g powder/ mL, because of the shape of its particles. Rather than particles being spherical like the other powders, drum-dried beet powder particles are more similar to flat sheets which formed as the dried beets were scraped off the drums of the drum dryer. Liu et al. (2011) found that vacuum belt-dried *Panax notoginseng* extract powder had a lower density than spray-dried and freeze-dried powders.

#### Dynamic Hygroscopicity

All powders reached constant mass in the 75% relative humidity chamber after 8 hours. All powders rapidly adsorbed moisture for the first two hours. Equilibrium moisture content of the spray-dried powder was the lowest at 16.5 g water/ g powder, followed by the vacuum belt dried powder with maltodextrin at 19.4 g water/ g powder, and finally all other powders had equilibrium moisture contents between 22.0 and 23.0 g water/ g powder. The two powders adsorbed less moisture due to the levels of maltodextrin added before dehydration. The spray dried powder and vacuum belt dried powder with maltodextrin added also had the lowest  $k$  values, or rates of moisture gain. The freeze dried powder had the highest  $k$  value, indicating that it adsorbed moisture most rapidly, because it had the lowest initial water activity and therefore the greatest concentration gradient between the powder and its surroundings.

Percent hygroscopicity ranged between 17.9 to 18.7% for all powders without maltodextrin and 13.6 and 16.2% for the powders with maltodextrin added. Once again,

maltodextrin has a significant effect on percent hygroscopicity and dehydration is not a major factor. Kim et al. (2012) found that increasing levels of maltodextrin in vacuum belt-dried blueberry slurry powders decreased the hygroscopicity of the powders.

### Moisture Isotherms

Powders were kept in chambers to adsorb moisture until they reached equilibrium. The Guggenheim-Andersen-Bohr model was fit to the data; results are displayed in Figure 4.3 and Table 4.4. Powders in chambers at low relative humidity levels remained amorphous and free flowing while powders in chambers at high relative humidity levels caked and got darker in color. All samples increased in moisture content slowly at higher water activities up to 0.5, and then rapidly increased in moisture content at water activities above 0.5. The spray-dried powder underwent transition to a glassy state. This is due to the depression of its glass transition temperature below 22°C at an increased moisture content. At a temperature above  $T_g$ , the powder is in a liquid-like state with increased mobility to flow, which leads to crystallization (Roos, 1995).

The moisture isotherms for drum-dried, spray-dried, tray-dried, and vacuum belt dried-0.6 maltodextrin powders all were J-shaped and took the form of type III isotherms. These isotherms are generally associated with crystalline materials, as beet powders contain high sugar contents. The freeze-dried powder and vacuum belt-dried powder with maltodextrin had shapes in between the sigmoidal type II isotherm and j-shaped type III isotherm. Type II isotherms are associated with materials in amorphous states, as these powders have regions of both amorphous and crystalline structure.

This moisture present at very low water activities below 0.2 (zone 1), is associated with forming a monolayer over polar sites in the powder and is not very mobile. At intermediate water

activities (zone 2) between 0.2 and 0.85, additional water is bound to remaining polar sites available as powders begin to swell. Additional water adsorbed, as powders enter the high water activity range (zone 3) above 0.85, is what constitutes the bulk water, this is what is available as a solvent, can be frozen, and can support the growth of microorganisms (Reid et al., 2008). Type II isotherms adsorb moisture quickly in zone 1, slowly in zone 2, and quickly in zone 3. Type III isotherms adsorb moisture more in zone 1, more quickly in zone 2, and very quickly in zone 3.

The freeze-dried, tray-dried, and vacuum belt-dried powders without maltodextrin had the highest monolayer moisture contents. These powders had the maximum number of sites for water to bind to the dehydrated beet solids and were all finely grinded. The vacuum belt-dried powder with maltodextrin and spray-dried powders had lower monolayer moisture contents due to maltodextrin's dilution of available sites for water to bind to. Finally drum-dried powder had the lowest monolayer moisture content due to its particle size and shape, which had a reduced the surface area to volume ratio therefore allowing for less water molecules to bind.

#### Particle size

Particle sizes of the beet powders dehydrated by different drying methods were analyzed with SEM; pictures of results are in Figure 4.1. Samples dried in pans (freeze-dried, tray-dried, vacuum belt-dried samples) were ground with a Nutribulle to created powders, thus resulting in particle sizes ranging from 10-200 microns. Due to atomization of beet juice prior to spray drying, the particles were more uniform in shape and size, small spheres at the order of magnitude of 10 microns. The drum dried powder particles were shaped like flat sheets as the product was dried on hot rollers and scraped off. These particles were larger, ranging from 200-600 microns.

### Total Betalain Quantification

The amount of betacyanins and betaxanthins in each of the beet powders dried with different methods on a total dry solids and dry beet solids basis was measured to determine the effect of the addition of maltodextrin and different processing conditions on betalain content. Results are displayed in Table 4.5. Powders contained 0.18 to 0.55 mg betanin/ g powder and 0.073 to 0.32 mg vulgaxanthin/ g powder.

The sample which was freeze dried as well as the sample that was vacuum belt dried without maltodextrin had both the highest betacyanin and betaxanthin content (0.61, 0.52 mg betanin/ g powder; 0.32, 0.26 mg vulgaxanthin/ g powder). This is due to processing conditions that did not expose samples to high temperatures or heat them for a long time, as betalains are heat labile and degrade at raised temperatures. The freeze dried powder was never heated as all drying occurred via sublimation at low temperatures and pressures and the vacuum of the vacuum belt dryer allowed for the reduction of the boiling point of water and therefore dehydration occurred at lower temperatures.

Tray-dried and drum-dried powders, both not containing any maltodextrin, had comparable levels of both betacyanins and betaxanthins (0.37, 0.31 mg betanin/ g powder; 0.15, 0.14 mg vulgaxanthin/ g powder). These two methods of dehydration exposed beets to atmospheric pressure and either a high temperature (drum drying) or low heat for an extended period of time (tray drying), both of which contribute to the degradation of betalains. The betacyanin content of vacuum belt dried powder with added maltodextrin contained the same betacyanin content as the drum-dried and tray-dried powders on a total dry solids basis. The betacyanin and betaxanthin content of powders spray-dried and vacuum belt-dried without the addition of maltodextrin on a per g dry beet solid basis were the same.

Spray-dried beet powder had a significantly lower betacyanin content than the other powders and a betaxanthin content comparable to the tray-dried, drum-dried, and vacuum belt-dried with maltodextrin powders on a per total dry solids basis. On a per beet solid basis, however, spray-dried beet powder had betacyanin and betaxanthin content comparable to the vacuum belt-dried powders and a betacyanin content even comparable to the freeze-dried powder. This also implies that maltodextrin had a dilution effect rather than spray drying causing degradation. During the spray drying process, beet juice was atomized and exposed to temperatures up to 150°C for a very short amount of time, which must have not been enough to initiate degradation of betalain compounds. It is important to note that the spray-dried beet powder was made by dehydrating beet juice, while all other drying methods used crushed beets, including all beet solids such as the beet peels.

Betacyanin content of 0.55 to 0.62 mg betacyanin per g beetroot solids was found by Azerado et al. (2007) when examining the effect of microencapsulation on betalain content. Nemzer et al. (2011) reported 0.18 to 0.32% violet betalains in betanin equivalents for spray-dried powder, 0.89 to 1.26% for freeze-dried powder, and 0.56 to 0.61% for air-dried beet powder when evaluating red beet root dried extracts. They also reported 0.06 to 0.18% yellow pigments in vulgaxanthin I equivalents for spray-dried extracts, 0.57 to 0.89% for freeze-dried extracts, and 0.32 to 0.35% for air-dried extracts. Kujala et al. (2000) found a higher betanin content of 38.7, 21.1, and 10.2 mg betanin/ g dry weight of lyophilized beetroot peel, crown, and flesh, respectively.

## **Conclusion**

Crushed beetroot was dehydrated with six different drying methods to compare physical properties and betalain content. All powders had similar moisture contents, between 1.5 and

2.0%, while the freeze-dried and spray-dried powders had lower water activities than the other powders. The two powders with added maltodextrin were the least hygroscopic, they adsorbed the least amount of moisture at equilibrium, as they had less available sites for water molecules to bind. Drum-dried powder had the lowest amount of monolayer moisture, as it had the smallest surface area to volume ratio meaning less area to make contact with the humidity in the air. The most powder emerged from a drum in testing flowability of powders for the tray-dried and vacuum-dried powders, but dehydration method was not a significant factor in the total amount of powder which emerged from the drum. The tray-dried and both vacuum belt-dried powders had the highest bulk density, while the spray-dried and drum-dried powders had the lowest. This is related to particle size and agglomeration of particles. Finally, the betalain content was the highest in freeze-dried and vacuum belt-dried powders due to minimal heat used; spray-dried powder had a comparable betalain content on a per g dry beet basis but much lower on a per g powder basis due to the dilution effects of maltodextrin.

Based on the lack of betalain degradation and favorable physical properties of the vacuum-belt-dried powder, it should be considered as an alternative method for creating a value-added beet powder. The vacuum belt-dried powders had properties comparable with the freeze-dried powder, the golden standard of drying, and dried in much less time, thus indicating reduced production cost. The vacuum belt dried powders also were less diluted with maltodextrin, thus less powder can be added to a product to have the same color effect. Further evaluation could be done to see if spray-drying beet juice and vacuum belt-drying the pomace could maximize yield of high quality beet powders.

Figure 4.1: Scanning electron microscopy pictures (SEM) of beet powders

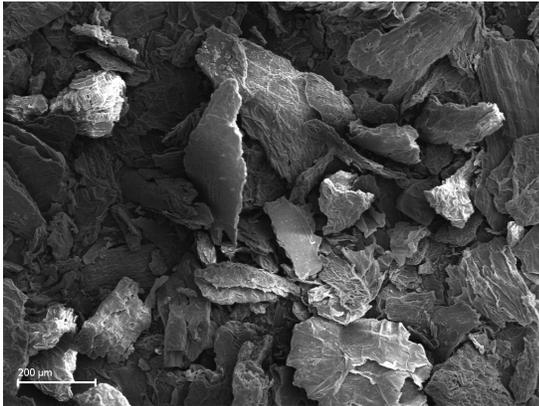


Figure 4.1a: Drum-dried powder

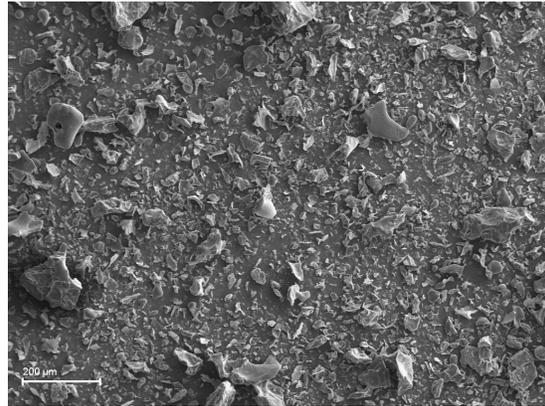


Figure 4.1b: Freeze-dried powder

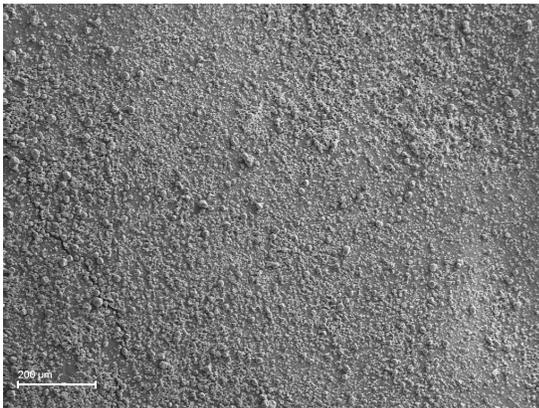


Figure 4.1c: Spray-dried powder

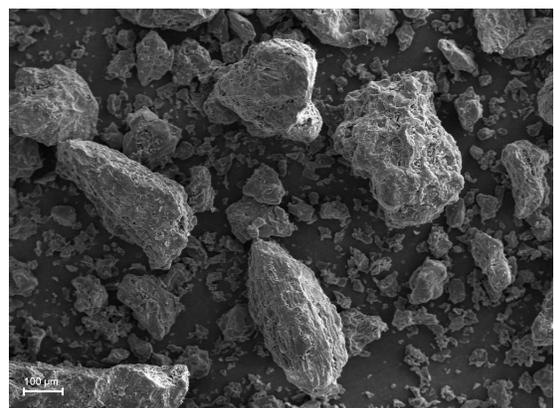


Figure 4.1d: Tray-dried powder

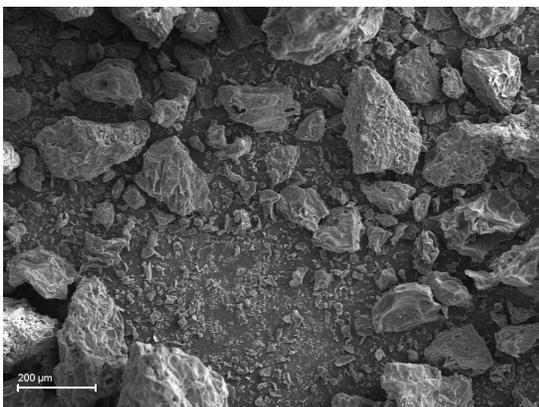


Figure 4.1e: Vacuum belt-dried powder,  
0 g maltodextrin/ g beet solids

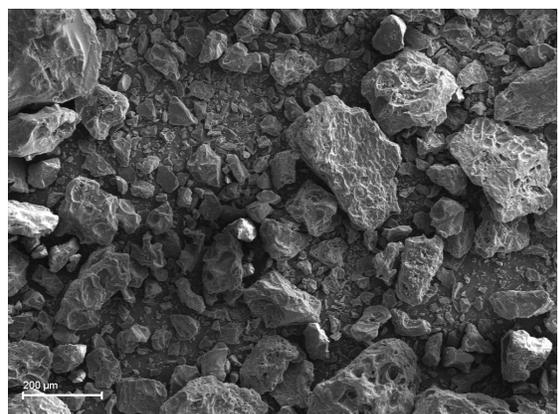


Figure 4.1f: Vacuum belt-dried powder  
0.6 g maltodextrin/ g beet solids

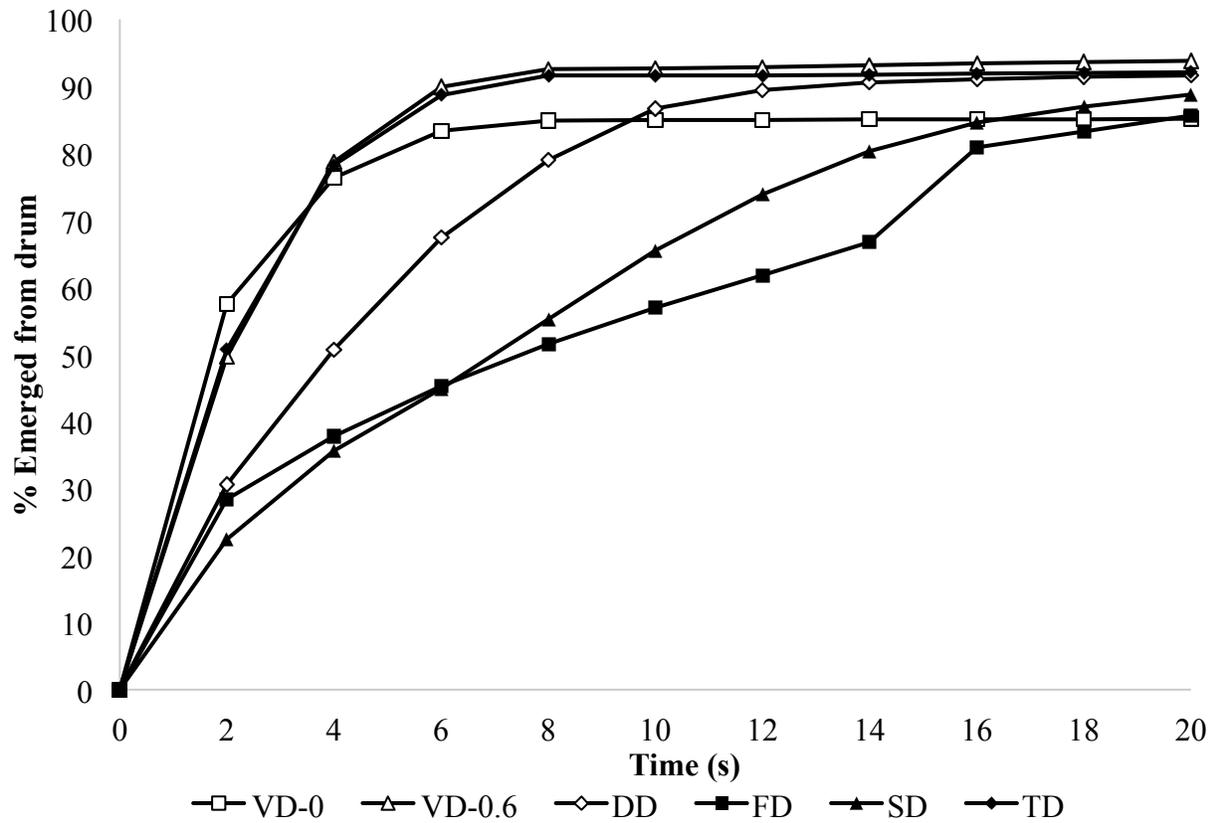


Figure 4.2: Flowability of beet powders prepared with different dehydration methods<sup>1</sup>

<sup>1</sup>DD= Drum-dried, FD= freeze-dried, SD= spray-dried, TD= tray-dried, VD-0= vacuum belt-dried without addition of maltodextrin, VD-6= vacuum belt-dried with 0.6 g maltodextrin/ g beet solid

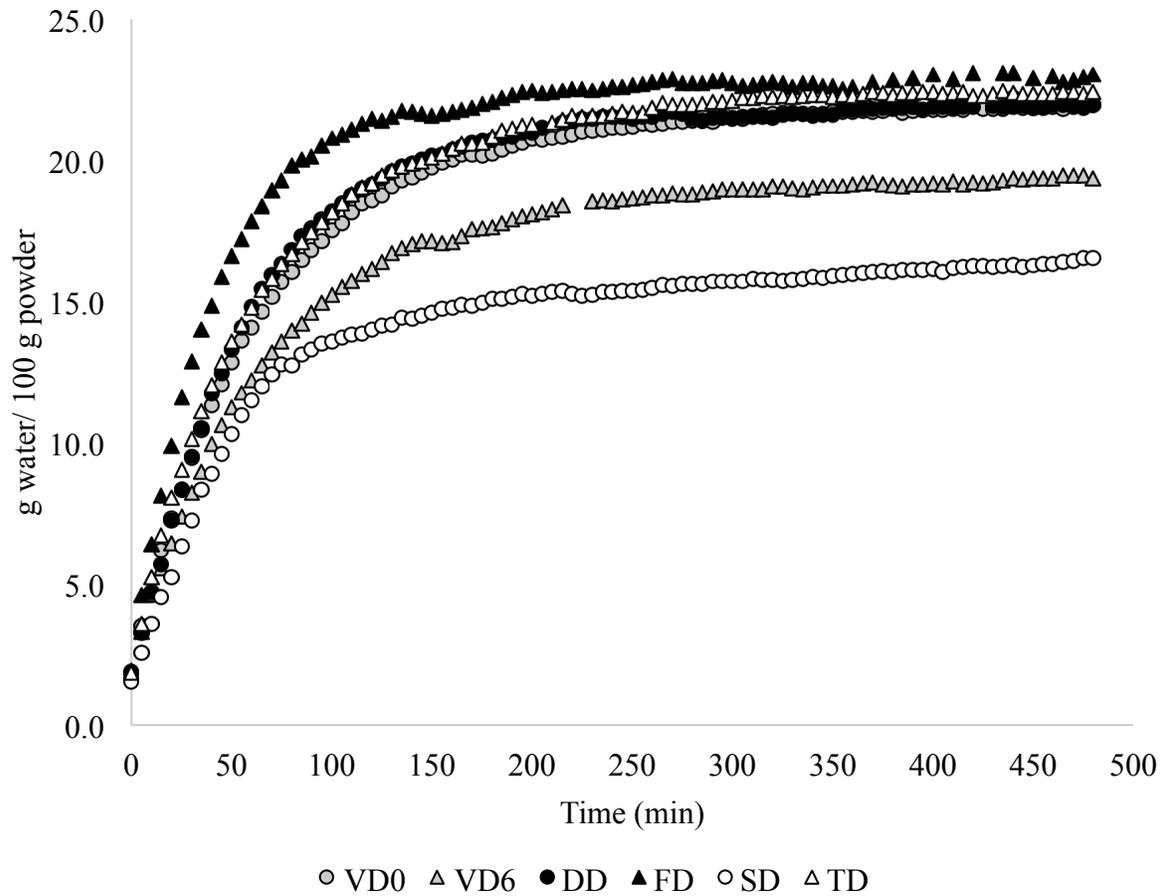


Figure 4.3: Dynamic hygroscopicity of beet powders prepared with different drying methods<sup>1</sup>

<sup>1</sup>DD= Drum-dried, FD= freeze-dried, SD= spray-dried, TD= tray-dried, VD-0= vacuum belt-dried without addition of maltodextrin, VD-6= vacuum belt-dried with 0.6 g maltodextrin/ g beet solid

Figure 4.4: Moisture Isotherms of beet powders prepared with different drying methods

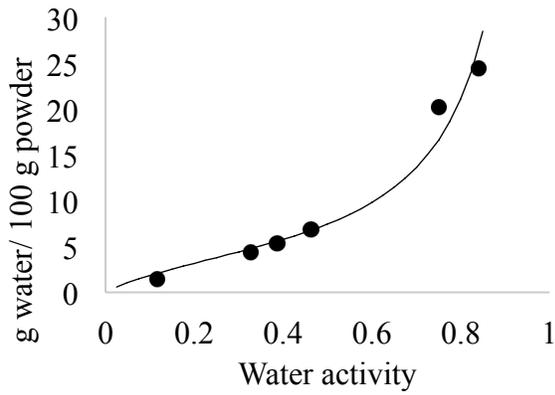


Figure 4.4a: Drum-dried powder

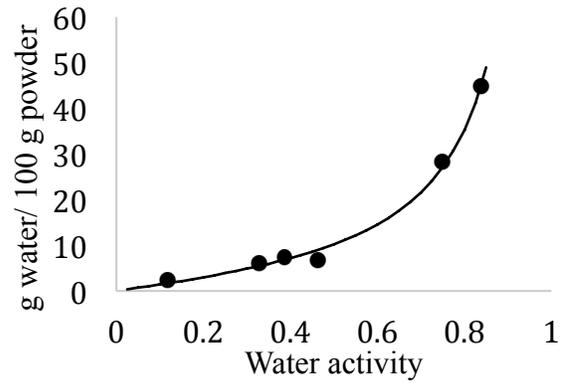


Figure 4.4b: Freeze-dried powder

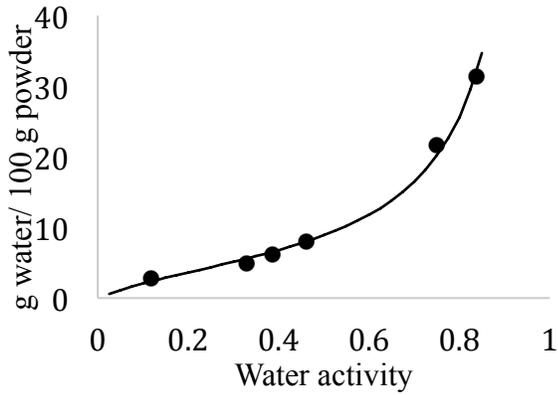


Figure 4.4c: Spray-dried powder

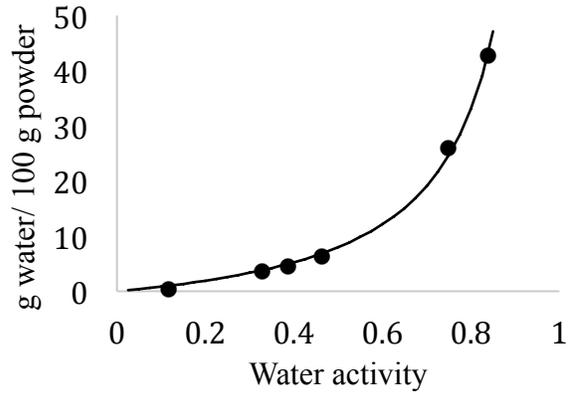


Figure 4.4d: Tray-dried powder

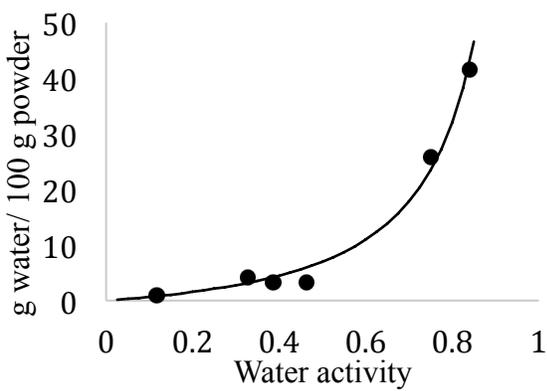


Figure 4.4e: Vacuum belt-dried powder,

0 g maltodextrin/ g beet solids

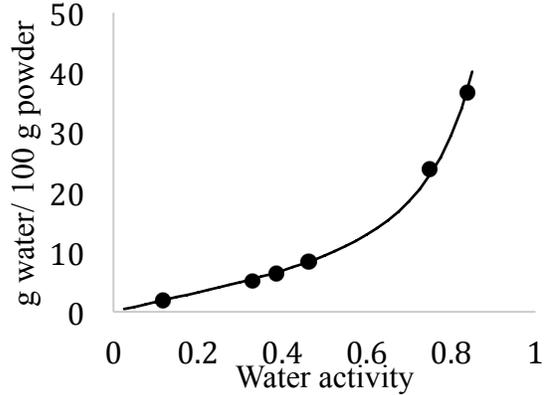


Figure 4.4f: Vacuum belt-dried powder

0.6 g maltodextrin/ g beet solids

Table 4.1: Water activity, moisture content, color, and bulk density of beet powders prepared with different drying methods

Drying Method <sup>1</sup>	a <sub>w</sub>	g water/ 100 g powder	Color			Bulk Density (g/ mL)
			L*	c*	h (°)	
DD	0.29 <sup>a</sup>	1.88 <sup>a</sup>	33.7 <sup>b</sup>	34.4 <sup>a</sup>	367.7 <sup>a</sup>	0.46 <sup>c</sup>
FD	0.21 <sup>ab</sup>	1.91 <sup>a</sup>	27.5 <sup>b</sup>	36.0 <sup>a</sup>	360.0 <sup>a</sup>	0.43 <sup>c</sup>
SD	0.16 <sup>b</sup>	1.51 <sup>a</sup>	48.5 <sup>a</sup>	35.6 <sup>a</sup>	353.4 <sup>b</sup>	0.55 <sup>bc</sup>
TD	0.30 <sup>a</sup>	1.93 <sup>a</sup>	27.5 <sup>b</sup>	27.8 <sup>bc</sup>	358.1 <sup>ab</sup>	0.69 <sup>a</sup>
VD-0	0.27 <sup>a</sup>	1.74 <sup>a</sup>	26.0 <sup>b</sup>	28.3 <sup>c</sup>	356.9 <sup>ab</sup>	0.67 <sup>ab</sup>
VD-0.6	0.29 <sup>a</sup>	1.78 <sup>a</sup>	30.0 <sup>b</sup>	33.4 <sup>ab</sup>	360.0 <sup>ab</sup>	0.73 <sup>a</sup>

<sup>a</sup> Values within a column with same letters indicates values are not statistically different (p<0.05).

<sup>1</sup> DD= Drum-dried, FD= freeze-dried, SD= spray-dried, TD= tray-dried, VD-0= vacuum belt-dried without addition of maltodextrin, VD-6= vacuum belt-dried with 0.6 g maltodextrin/ g beet solid

Table 4.2: Flowability of beet powders prepared with different drying methods

Drying Method <sup>1</sup>	% emerged at 4 s	% emerged at 20 s
DD	50.7 <sup>b</sup>	91.6 <sup>a</sup>
FD	37.8 <sup>b</sup>	85.6 <sup>a</sup>
SD	35.6 <sup>b</sup>	88.8 <sup>a</sup>
TD	78.2 <sup>a</sup>	92.1 <sup>a</sup>
VD-0 MD	76.4 <sup>a</sup>	85.2 <sup>a</sup>
VD-0.6 MD	78.7 <sup>a</sup>	93.8 <sup>a</sup>

<sup>a</sup> Values within a column with same letters indicates values are not statistically different (p<0.05).

<sup>1</sup> DD= Drum-dried, FD= freeze-dried, SD= spray-dried, TD= tray-dried, VD-0= vacuum belt-dried without addition of maltodextrin, VD-6= vacuum belt-dried with 0.6 g maltodextrin/ g beet solid

Table 4.3: Percent hygroscopicity and moisture sorption model of beet powders prepared with different drying methods

Drying Method <sup>1</sup>	k (min <sup>-1</sup> )	R <sup>2</sup>	m <sub>e</sub> (g water/ 100 g powder)	% hygroscopicity
DD	0.0167	.998	22.0	17.9
FD	0.0222	.994	23.0	18.7
SD	0.0149	.973	16.5	13.6
TD	0.0154	.998	22.4	18.3
VD-0	0.0147	.998	22.0	18.0
VD-0.6	0.0139	.998	19.4	16.2

<sup>1</sup> DD= Drum-dried, FD= freeze-dried, SD= spray-dried, TD= tray-dried, VD-0= vacuum belt-dried without addition of maltodextrin, VD-6= vacuum belt-dried with 0.6 g maltodextrin/ g beet solid

Table 4.4: Guggenheim-Anderson-de Boer (GAB) model of moisture isotherms of beet powders prepared with different drying methods

Drying Method <sup>1</sup>	$m_0$ (g water/ 100 g powder)	c	$R^2$
DD	4.41	5.45	0.960
FD	8.10	1.70	0.994
SD	5.40	4.61	0.994
TD	8.46	0.89	0.998
VD-0	8.88	0.65	0.989
VD-0.6	6.42	2.71	0.998

<sup>a</sup> Values within a column with same letters indicates values are not statistically different (p<0.05).

<sup>1</sup> DD= Drum-dried, FD= freeze-dried, SD= spray-dried, TD= tray-dried, VD-0= vacuum belt-dried without addition of maltodextrin, VD-6= vacuum belt-dried with 0.6 g maltodextrin/ g beet solid

Table 4.5: Total betalain quantification of beet powders prepared with different drying methods

Drying Method	mg betanin/ g dry powder	mg vulgaxanthin/ g dry powder	mg betanin/ g dry beet solids	mg vulgaxanthin/ g dry beet solids
DD	0.31 <sup>c</sup>	0.14 <sup>bc</sup>	0.31 <sup>d</sup>	0.14 <sup>d</sup>
FD	0.61 <sup>a</sup>	0.32 <sup>a</sup>	0.61 <sup>a</sup>	0.32 <sup>a</sup>
SD	0.18 <sup>d</sup>	0.073 <sup>c</sup>	0.57 <sup>ab</sup>	0.23 <sup>bc</sup>
TD	0.37 <sup>c</sup>	0.15 <sup>b</sup>	0.37 <sup>d</sup>	0.15 <sup>d</sup>
VD-0	0.52 <sup>b</sup>	0.26 <sup>a</sup>	0.52 <sup>bc</sup>	0.26 <sup>ab</sup>
VD-0.6	0.29 <sup>c</sup>	0.11 <sup>bc</sup>	0.47 <sup>c</sup>	0.18 <sup>cd</sup>

<sup>a</sup> Values within a column with same letters indicates values are not statistically different (p<0.05).

<sup>1</sup> DD= Drum-dried, FD= freeze-dried, SD= spray-dried, TD= tray-dried, VD-0= vacuum belt-dried without addition of maltodextrin, VD-6= vacuum belt-dried with 0.6 g maltodextrin/ g beet solid

## CHAPTER 5

### SUMMARY AND CONCLUSION

This research focused on the dehydration of crushed beetroot using different drying methods. The first objective of the study was to optimize dehydration with a vacuum belt dryer based on betalain content and physical properties. To better understand the drying process, the dehydration of beets with the vacuum belt dryer at two thicknesses and three temperatures was modeled. This provided information about the drying rate, productivity, and drying ratio of beet dehydration. Drying rate increased with temperature and decreased with sample thickness.

To optimize the dehydration of beets with a vacuum belt dryer, beet samples were then dehydrated at three temperatures (75, 85, 95°C) and three different levels of maltodextrin (0, 0.3, 0.6 g maltodextrin/ g beet solids) using a vacuum belt dryer. Important properties of the resulting powders included betalain content as well as physical properties such as bulk density, flowability, hygroscopicity, and moisture isotherms. Betalain content did not differ between powders on a per g dry beet solid basis, thus neither temperature nor maltodextrin content led to degradation of betalains; however, maltodextrin had a dilution effect as the betalain content per g powder was lower with increasing maltodextrin. There was no difference in the amount of powder which emerged from the drum after 4 s but more total powder emerged from the drum with powders which contained increased maltodextrin content. Bulk density also increased with increasing maltodextrin content of the powders. Hygroscopicity and monolayer moisture content decreased with increasing maltodextrin content. Overall, due to a faster drying rate and

conservation of betalains, the powders dried at 95°C are recommended for creating value-added beet powders.

The beet powders dried at 95°C with 0 and 0.6 g maltodextrin/ g beet solids were compared against beet powders which were drum-dried, freeze-dried, spray-dried, and tray-dried. Important properties of the resulting powders also included betalain content as well as physical properties such as bulk density, flowability, hygroscopicity, and moisture isotherms. Freeze-dried and vacuum belt-dried powders had the highest betalain contents due to the least amount of thermal processing. Bulk densities of vacuum belt dried powders and tray-dried powders were the highest while bulk densities of drum-dried, freeze-dried, and spray-dried powders had the lowest bulk densities. The most powder emerged from the drum at 4 s from the tray-dried and vacuum belt-dried powders; however the same total amount of powder flowed out of the drum. The spray-dried powder and vacuum belt-dried powder with maltodextrin were less hygroscopic than the other powders due to the presence of maltodextrin. These two powders, along with the drum-dried powder, had the lowest monolayer moisture contents.

Overall, vacuum belt-drying is a method for creating high quality beet powders. It takes much less time to dehydrate beets with a vacuum belt-dryer than a freeze dryer without degradation of betalains.

## REFERENCES

- Abadio, F.D.B., Domingues, A.M., Borges, S.V., & Oliviera, V.M. (2004). Physical properties of powdered pineapple (*Ananas comsus*) juice effect of maltodextrin concentration and atomization speed. *Journal of Food Engineering*, 64(3), 285-287.
- Agrawal, A. (2013). Scope of betalains as a food colorant. *International Journal of Advanced Scientific and Technical Research*, 3(3), 22-36.
- Ardsel, W. B. van, Copley, M. J., Morgan, A. I. Jr. (Eds.). (1973). *Food Dehydration, Vol. 1. Drying methods and phenomena*. Westport, CT: The AVI Publishing Company, Inc.
- Attoe, E. L., & Von Elbe, J. H. (1982). Degradation kinetics of betanin in solutions as influenced by oxygen. *Journal of Agricultural and Food Chemistry*, 30(4), 708-712.
- Azeredo, H.M.C. (2009). Betalains: properties, sources, applications, and stability- a review. *International Journal of Food Science & Technology*, 44, 2365-2376.
- Bailey, S. J., Winyard, P, Vanhatalo, A., Blackwell, J. R., DiMenna, F. J., Wilkerson, ... Jones, A.M. (2009). Dietary nitrate supplementation reduces the O<sub>2</sub> cost of low-intensity exercise and enhances tolerance to high-intensity exercise in humans. *Journal of Applied Physiology*, 107, 1144-1155.
- Barbosa-Cánovas, G. V., Vega-Mercado, H.V. (1996). *Dehydration of foods*. New York, NY: Chapman & Hall.
- Barbosa-Canovas, G.V., Ortega-Rivas, E., Juliano, P., & Yan, H. (2005). *Food powders: Physical properties, processing, and functionality*. New York, NY: Springer.

- Bhagwat, S., Haytowitz, D. B., & Holden, J.M. (2013). USDA Database for the flavonoid content of selected foods. USDA Nutrient Data Laboratory, Release 3.1.
- Bobek, P., Galbavy, S., & Mariasssova, M. (2000). The effect of red beet (*Beta vulgaris var. rubra*) fiber on alimentary hypocholesterolemia and chemically induced colon carcinogenesis in rats. *Nahrung*, 44(3), 184-187.
- Cai, Y.Z., & Corke, H. (2000). Production and properties of spray dried amaranthus betacyanin pigments, *Journal of Food Science*, 65(6), 1248-1252.
- Catelam, K.T., Trindade, C.S.F., & Romero, J.T. (2011). Water adsorption isotherms and isosteric sorption heat of spray-dried and freeze-dried dehydrated passion fruit pulp with additives and skimmed milk. *Ciencias Agroteca*, 35, 1196-1203.
- Cermak, N.M., Res, P., Stinkens, R., Lundberg, J.O., Gibala, M.J., & van Loon, L.J.C. (2012). No improvement in endurance performance after a single dose of beetroot juice. *International Journal of Sport Nutrition and Exercise Metabolism*, 22(6), 470-478.
- Cohen, J.S., Yang, T.C.S. (1995). Progress in food dehydration. *Trends in Food Science and Technology*, 6(1), 20-25.
- Delgado-Vargas, F., Jiménez, A.R. & Paredes-López, O. (2000). Natural pigments: carotenoids, anthocyanins, and betalains—characteristics, biosynthesis, processing, and stability. *Critical Reviews in Food Science and Nutrition*, 40(3), 173-289.
- Dongowski, G. (2001). Enzymatic degradation studies of pectin and cellulose from red beets. *Nahrung*, 45(5), 324-331.
- Doulia, D., Tzia, K., & Gekas, V. (2000). A knowledge base for the apparent mass diffusion coefficient (Deff) of foods. *International Journal of Food Properties*, 3(1), 1-14.

- Drdak, M. & Vallova, M. (1990). Kinetics of the thermal degradation of betanine. *Nahrung*, 34(4), 307-310.
- Food and Agriculture Organization of the United States, FAOSTAT database (Sept. 2011).
- Francis, F.J. (1999). Anthocyanins and betalains. In F.J. Francis (Ed.), *Colorants*, 280-309. St. Paul, MN: Eagen Press.
- Fitzpatrick, J.J., & Ahrne, L (2005). Food powder handling and processing: Industry problems, knowledge barriers and research opportunities. *Chemical Engineering and Processing: Process Intensification*, 44(2), 209-214.
- Gentile, C., Tesoriere, L., Allegra, M., Livrea, M.A., & D'Alessio, P. (2004). Antioxidant betalains from cactus pear (*Opuntia ficus-indica*) inhibit endothelial ICAM-1 expression. *Signal Transduction and Communication in Cancer Cells*, 1028, 481-486.
- Gharsallaoui, A., Roudaut, G., Chambin, O., Voilley, A., & Saurel, R. (2007). Applications of spray-drying in microencapsulation of food ingredients: An overview. *Food Research International*, 40, 1107-1121.
- Gilchrist, M., Winyard, P.G., Benjamin, N. (2010). Dietary nitrate-Good or bad?. *Nitric Oxide*, 22(2), 104-109.
- Gliszczynska-Swiglo, A., Szymusiak, H., & Malinowska, P. (2006). Betanin, the main pigment of red beet: Molecular origin of its exceptionally high free radical-scavenging activity. *Food Additives and Contaminants*, 23, 1079-1087.
- Gokhale, S.V. & Lele, S.S. (2011). Dehydration of red beet root (*Beta vulgaris*) by hot air drying: process optimization and mathematical modeling. *Food Science and Biotechnology*, 20(4), 955-964.

- Gokhale, S.V. & Lele, S.S. (2012). Optimization of convective dehydration of beta vulgaris for color retention. *Food and Bioprocess Technology*, 5(3), 868-878.
- Goldman, I., & Navazio, J. (2008). Table Beet. In J. Prohens & F. Nuez (Eds.), *Vegetables I*, vol. 1 (pp. 219-238): Springer New York.
- Goula, A.M., Adamopoulos, K.G., & Kazakis, N.A. (2004). Influence of spray drying conditions on tomato powder properties. *Drying Technology*, 22, 1129-1151.
- Grabowski, J.A., Truong, V.-D., & Daubert, C.R. (2006). Spray-drying of amylase hydrolyzed sweetpotato puree and physicochemical properties of powder. *Journal of Food Science*, 71(5), E209-E217.
- Hawlder, M. N. A., Perera, C.O., Tian, M., & Yeo, K. L. (2008). Drying of guava and papaya: Impact of different drying methods. *Drying Technology*, 24(1), 77-87.
- Hayashi, H., Kumazawa, E., Saeki, Y., & Ishioka, Y. (1983). Continuous vacuum dryer for energy saving. *Drying Technology*, 1 (2), 275-283.
- Herbach, K.M., Stinzing, F.C., & Carle, R. (2004a). Impact of thermal treatment on color and pigment pattern of red beet (*Beta vulgaris L.*) preparations. *Journal of Food Science*, 69, C491-498.
- Herbach, K.M., Stintzing, F.C. & Carle, R. (2004b). Thermal degradation of betacyanins in juices from purple pitaya (*Hylocereus polyrhizus [Weber] Britton and Rose*) monitored by high-performance liquid chromatography-tandem mass spectrometric analyses. *European Food Research and Technology*, 219, 377–385.
- Hogekamp, S., & Schubert, H. (2003). Rehydration of food powders. *Food Science and Technology International*, 9(3), 223-225.

- Huang, A.S. & von Elbe, J.H. (1985). Kinetics of the degradation and regeneration of betanine. *Journal of Food Science*, 50(4), 1115-1120.
- Jackman, R.L. & Smith, J.L. (1996). Anthocyanins and betalains. In G.F. Hendry & J.D. Houghton (Eds.) *Natural food colorants* (244-309). London, Great Britain: Blackie Academic & Professional.
- Janiszewska, E. & Włodarczyk, J. (2013). Influence of spray drying conditions on beetroot pigments retention after microencapsulation process. *Acta Agrophysica*, 20(2), 343-356.
- Jaya, S. & Das, H. (2004). Effect of maltodextrin, glycerol monostearate and tricalcium phosphate on vacuum dried mango powder properties. *Journal of Food Engineering*, 63(2), 125-134.
- Jiratanan, T., & Liu, R.H. (2004). Antioxidant activity of processed table beets (*Beta vulgaris* var, *conditiva*) and green beans (*Phaseolus vulgaris* L.). *Journal of Agricultural and Food Chemistry*, 52, 2659-2670.
- Kanner, J., Harel, S., & Granit, R. (2001). Betalains- a new class of dietary catonized antioxidants. *Journal of Agricultural and Food Chemistry*, 49, 5178-5185.
- Kapadia, G.J., Tokuda, H., Konoshima, T., & Nishino, H. (1996). Chemoprevention of lung and skin cancer by Beta vulgaris (beet) root extract. *Cancer Letters*, 100 (1-2), 211-214.
- Kim, M., & Kerr, W.L. (2012). Vacuum-belt drying of rabbiteye blueberry slurries: influence of drying conditionson physical and quality properties of blueberry powder. *Food and Bioprocess Technology*, 2013, 3227-3237.
- Konoshima, T., & Takasake, M. (2003). Anti-carcinogenic activities of natural pigments from beet root and saffron. *Foods and Food Ingredients Journal of Japan*, 208(8), 615-622.

- Krokida, M.K., Karathanos, V.T., & Maroulis, Z.B. (1998). Effect of freeze-drying conditions on shrinkage and porosity of dehydrated agricultural products. *Journal of Food Engineering*, 35(4), 369-380.
- Kujala, T. S., Loponen, J. M., Klika, K. D., & Pihlaja, K. (2000). Phenolics and betacyanins in red beetroot (*Beta vulgaris*) root: distribution and effect of cold storage on the content of total phenolics and three individual compounds. *Journal of Agricultural and Food Chemistry*, 48, 5338-5342.
- Liu, X., Qiu, Z., Wang, L., & Chen, Y. (2011). Quality evaluation of *Panax notoginseng* extract dried by different drying methods. *Foods and Bioproducts Processing*, 89 (2011), 10-14.
- Liu, X., Qiu, Z., Wang, L., Cheng, Y., Qu, H., & Chen, Y. (2009). Mathematical modeling for thin layer vacuum belt drying of *Panax notoginseng* extract. *Energy Conversion and Management*, 50, 928-932.
- Marmion, D. M. (1991). Handbook of US colorants: foods, drugs, cosmetics, and medical devices: Wiley.
- Mathlouthi, M., & Roge, B. (2003). Water vapor sorption isotherms and the caking of food powders. *Food Chemistry*, 82 (2003), 61-71.
- Mintel (2013). Food Colours-International- February 2013. Number 1475918. Mintel Group Ltd.
- Monzini, A. & Maltini, E. (1990). Production of juice powders without additives by vacuum belt drying. *Flussiges Obst*, 57(2), 89-90.
- Moraga, G., Martinez-Navarrete, N., & Chiralt, A. (2006). Water sorption isotherms and phase transitions in kiwifruit. *Journal of Food Engineering*, 72(2), 147-156.

- Moreno, D.A., Garcia-Viguera, C., Gil, J.I., & Gil-Izquierdo, A. (2008). Betalains in the era of global agri-food science, technology, and nutritional health. *Phytochemistry Reviews*, 7(2), 261-280.
- Muggeridge, D.J., Howe, C.C., Spendiff, O., Pedlar, C., James, P.E., & Easton, C. (2014). A single dose of beetroot juice enhances cycling performance in simulated altitude. *Medicine and Science in Sports and Exercise*, 46(1), 143-150.
- Murphy, M., Eliot, K., Heuertz, R.M., Weiss, E. (2012). Whole beetroot consumption acutely improves running performance. *Journal of the Academy of Nutrition and Dietetics*, 112(4), 548-552.
- Neelwarne, B. (2012). *Red Beet Biotechnology: Food and Pharmaceutical Applications*: Springer.
- Nemzer, B., Pietrkowski, Z., Sporna, A., Stalica, P., Thresher, W., Michalowski, T., & Wybraniec, S. (2011) Betalainic and nutritional profiles of pigment-enriched red beet root (*Beta vulgaris L.*) dried extracts. *Food Chemistry*, 127(1), 42-53.
- Ozsoy-Sacan, O., Karabulut-Bulann, O., Bolkent, S., Yanardag, R., & Ozgey, Y. (2004). Effects of chard (*Beta vulgaris L. var. cicla*) on the liver of the diabetic rats: a morphological and biochemical study. *Bioscience, Biotechnology, and Biochemistry*, 68, 1640-1648.
- Pedreno, M.A., & Escribano, J. (2001). Correlation between antiradical activity and stability of betanine from *Beta Vulgaris L* roots under different pH, temperature and light conditions. *Journal of the Science of Food and Agriculture*, 81(7), 627-631.
- Peleg, M. (1983). *Physical Properties of Food*. Westport, CT: AVI Publishing Company, Inc.

- Piatelli, M. (1981). The betalains: structure, biosynthesis, and chemical taxonomy. In E.E. Conn, The biochemistry of plants: A comprehensive treatise (vol. 7), 557-575. New York, NY: Academic Press.
- Rahman, M. S. (2007). Handbook of food preservation: CRC press.
- Research, T. M. (2013). Food Colors Market - Global Industry Size, Market Share, Trends, Analysis, And Forecasts 2012 - 2018. In).
- Ravichandran, K., Saw, N.M.M.T., Mohdaly, A. A. A., Gabr, A.M.M., Kastell, A., Riedel, H., ... Smetanska, I. (2013). Impact of processing of red beet on betalain content and antioxidant activity. *Food Research International*, 50(2013), 670-675.
- Rodriquez-Hernandez, G.R., Gonzalez-Garcia, R., Grajales-Lagunes, A., Ruiz-Cabrera, M.A., & Abud-Archila, M. (2005). Spray-drying of cactus pear juice (*Opuntia streptacantha*): Effect on the physiochemical properties of powder and reconstituted product. *Drying Technology*, 23(4), 955-973.
- Roos, Y.H. (1995). *Phase Transitions in Foods*. New York, NY: Academic Press.
- Sagar, V.R., Kumar, S. (2010). Recent advances in drying and dehydration of fruits and vegetables: a review. *Journal of Food Science and Technology*, 47(1), 15-26.
- Saguy, I., Kopelman, I.J. & Mizrahi, S. (1978). Thermal kinetic degradation of betanin and betalamic acid. *Journal of Agricultural and Food Chemistry*, 26(2), 360-362.
- Strack, D., Vogt, T., & Schliemann, W. (2003). Recent advances in betalain research. *Phytochemistry*, 62(3), 247-269.
- Tang, J., Feng, H., & Shen, G.-Q. (2003). Drum drying. In Encyclopedia of Agricultural and Biological Engineering (pp. 211-214): Dekker: New York.

- Teunou, E., Fitzpatrick, J., & Synnott, E. (1999). Characterization of food powder flowability. *Journal of Food Engineering*, 39(1), 31-37.
- Tesoriere, L., Allegra, M., Butera, D. & Livrea, M.A. (2004). Absorption, excretion, and distribution of dietary antioxidant betalains in LDLs: Potential health effects of betalains in humans. *American Journal of Clinical Nutrition*, 80(4), 941–945.
- Tonon, R.V., Brabet, C., & Hubinger, M. D. (2008). Influence of process conditions on the physiochemical properties of acai (*Euterpe oleraceae* Mart.) powder produced by spray drying. *Journal of Food Engineering*, 88 (3), 411-418.
- U.S. Department of Agriculture (2011) USDA National Nutrient Database for Standard Reference, Release 26. <http://ndb.nal.usda.gov/> (accessed Jan. 15 2014).
- U.S. Department of Agriculture (2013). Vegetables 2012 Summary. <http://usda01.library.cornell.edu/usda/current/VegeSumm/VegeSumm-01-29-2013.pdf> (accessed Jan. 15 2014)
- von Elbe, J.H. (2001). Unit F3.1: Betalains. *Current Protocols in Analytical Chemistry*. F.3.1.1-F.3.1.7.
- Wang, J., Li, Y.Z., Chen, R.R., Bao, J.Y., Yang, G.M. (2007). Comparison of volatiles of banana powder dehydrated by vacuum belt drying, freeze-drying, and air-drying. *Food Chemistry*, 104(2007), 1516-1521.
- Wang, W. & Zhou, W. (2013). Water adsorption and glass transition of spray-dried soy sauce powders using maltodextrin as carrier. *Food and Bioprocess Technology*, 6, 2791-2799.
- Webb, A.J., Patel, N., Loukogeorgakis, S., Okorie, M., Aboud, Z., Misra, S., ... & Ahluwalia, A. (2008). Acute blood pressure lowering, vasoprotective, and antiplatelet properties of dietary nitrate via bioconversion to nitrite. *Hypertension*, 51(3), 784-790.

- Wilkerson, D.P., Hayward, G.M., Bailey, S.J., Banhatalo, A., Blackwell, J.R., & Jones, A.M. (2012). Influence of acute dietary nitrate supplementation on 50 mile time trial performance in well-trained cyclists. *European Journal of Applied Physiology*, 112, 4127-4134.
- Wu, L., Hsu, H.W., Chen, Y.C., Chiu, C.C., Lin, Y.I. & Ho, J.A. (2006). Antioxidant and antiproliferative activities of red pitaya. *Food Chemistry*, 95(2), 319–327.
- Wyanbriec, S. (2007). A method for identification of diastereomers of 2-decarboxy-betacyanins and 2,17-bidecarboxy-betacyanins in reversed-phase HPLC. *Analytical and Bioanalytical Chemistry*, 389(5), 1611-1621.
- Yamasita, T., & Sato, K. (2008). Effects of beet red inhibiting experimental diabetes in vivo-studies using ALS strain mice. *Foods and Food Ingredients Journal of Japan*, 213(2), 122-130.
- Yan, H. (2012). Vacuum belt dried apple pomace powder as a value-added ingredient. MS Thesis. Department of Food Science and Technology, University of Georgia, Athens, GA.