VACUUM-BELT DRYING OF BLUEBERRY POWDERS

by

MICHELLE KIM

(Under the Direction of William L. Kerr)

ABSTRACT

Vacuum-belt drying (VBD) was performed on whole blueberry slurry (WBS) and blueberry pomace (BP). The effect of drying temperatures (80°C, 95°C, 110°C), and levels of maltodextrin (0.3, 0.45, 0.6 kg MD/Kg dry solids of blueberries) on physical properties of WBS powders were studied. Increasing MD levels and drying temperatures reduced hygroscopicty and improved powder flowability. Moisture sorption isotherms were well-fit with the GAB model and showed type Ⅲ behavior. WBS powders showed reddish-purple color having lowest L* value at 80°C, and highest C* value at 110°C. BP was dried using different temperatures (80°C, 100°C, 120°C) and methods (batch and continuous VBD). Higher temperatures and continuous drying reduced hygroscopicity of BP powders. BP powders showed good flowability compared to WBS powders. GAB model for BP powders showed type Ⅱ behavior. Higher drying temperatures resulted in lower C* and h°. Batch processed had higher C* and lower h° than continuously processed.

INDEX WORDS: Drying, Vacuum-belt drying, Blueberries, Blueberry pomace, Fruit powder, Maltodextrin, Hygroscopicity, Flowability

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DEDICATION

For God so loved the world that he gave his one and only Son, that whoever believes in him shall not perish but have eternal life. (JOHN 3:16)

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CHAPTER 1

INTRODUCTION

Blueberries produced in North America account for 90 % of world production (Shi and others 2008). Blueberry markets are expanding fast due to the perceived health benefits, nutritional values, and distinctive flavors of blueberries (Feng and others 1999; Diaz and others 2011). However, blueberries have limited growing seasons and fresh blueberries have a relatively short shelf life even under refrigerated conditions (Feng and others 1999). To extend the shelf life of blueberries, processing such as dehydration and freezing are necessary. It is reported that almost 99% of lowbush blueberries produced in North America are used for processing (Brazelton and Strik 2007).

Blueberries are included in the family Ericaceae, genus *Vaccinium* (Chakraborty and others 2010). Three main types of blueberries, namely rabbiteye (*Vaccinum ashei*), southern highbush (*V.corymbosum* and *V.darrowi* Camp), and northern highbush (*V.corymbosum* L.) are grown in Georgia. Rabbiteye is the easiest cultivar to grow in Georgia, and it has high productivity (Krewer and Nesmith 2006).

Berries are widely known for their antioxidant and anticarcinogenic properties (Zheng and Wang 2003), and blueberries in particular have high antioxidant capacity as compared to other fruits and vegetables (Prior and others 1998). Blueberries are rich in phenolic acids, flavanols (catechins), flavonols, anthocyanins, proanthocyanidins, and hydroxycinnamates, which have significant health benefits to human health (Kalt and McDonald 1996; Prior and others 1998). In particular, anthocyanins give the blue, violet, purple, and red color in

blueberries, and they have a variety of health benefits (Lohachoompol and others 2004). Blueberries have shown promise for positively impacting cognition and vision. They may also reduce the risk of cancer and cardiovascular disease (Zafra-Stone and others 2007).

A significant amount of blueberries are processed into juices. Pomace, or presscake, remains after juice processing, and accounts for up to 20% of the initial weight of the fruit (Khanal and others 2009). Blueberry pomace is a great source of health promoting compounds such as anthocyanins, polyphenolics (Lee and Wrolstad 2004), and proanthocyanidins (Khanal and others 2009). Thus, blueberry pomace can add value to products (Lee and Wrolstad 2004). In addition, the economic and environmental costs associated with pomace disposal can be lowered (Khanal and others 2010) .

Drying is one of the most extensively used techniques to preserve food products (Tunde-Akintunde and Afolabi 2010). Microbial growth and moisture-meditated deteriorative reactions in food products are minimized by moisture removal (Sacilik 2007). In particular, vacuum belt drying (VBD) is commonly used for fruit and fruit juice drying (Wang and others 2007). It is also a good method for drying hygroscopic, sticky and pasty food products (Liu and others 2011). During VBD, the material to be dried is introduced through an air-lock, as a result oxidative degradation is very low compared to other drying technologies (Jaya and Das 2009). High quality end products, short drying time, convenience of drying, and low costs are additional pros of VBD (Liu and others 2009; Wang and others 2007).

Fruit powders are convenient to use, also they add value to food products due to their nutritional and functional benefits. They are considered ideal ingredients for food products such as baby foods, extruded cereal products, soups, dips, marinades, sauces, snack fillings, and confections (Jakubczyk and others 2010; Grabowski and others 2008). On the other hand, drying

fruit juice is not easy due to high content of low molecular weight sugars (sucrose, fructose, glucose), which have low glass transition temperatures (Roos 1995; Jaya and Das 2004). Food materials are unstable, experience structural collapse above glass transition temperature (Phanindrakumar and others 2005). To prevent stickiness during drying, high molecular weight drying aids such as maltodextrin, gum Arabic, and isolated proteins are added. Glass transition temperatures are increased by these drying aids (Jaya and others 2006; Comunian and others 2011).

The objectives of this study were the following:

- To produce blueberry and blueberry pomace powders by vacuum-belt drying that have good physical properties
- To determine optimal drying temperatures and times for blueberry and blueberry pomace powders
- To investigate the physical properties of blueberry and blueberry pomace powders

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CHAPTER 2

REVIEW OF LITERATURE

Blueberries

Blueberries (Figure 2.1) are included in the family Ericaceae, genus *Vaccinium,* and they grow well in mild climates (Chakraborty and others 2010). They have a spherical shape, and the size ranges between 0.7 to 1.5 cm in diameter (Vega-Galvez and others 2009). The characteristic color of blueberries is due to the water-soluble anthocyanins they contain (Allan-Wojtas and others 2001). The red, blue, and purple colors in grapes and other berries are also due to anthocyanins, although the proportions of various anthocyanins varies amongst fruit (Camire and others 2002). For example, blueberries generally have higher content of malvidin 3-O-glusoide while cranberries have higher levels of pelargonidin-3-O-glucoside.

North America is the major blueberry producer, contributing to almost 90% of the world's production (Shi and others 2008). In 2005, 69% of highbush blueberries were planted in North America. In addition, 67% of fresh blueberries and 94% of processed blueberries were produced in North America. Highbush blueberries are grown in the east, west, and south parts of North America. Rabbiteye (*Vaccinum ashei*), southern highbush (*V. corymbosum* and *V. darrowi* Camp), and northern highbush (*V. corymbosum* L.) are included in highbush blueberries. Production of highbush blueberries has grown rapidly since 1975. In North America, the production acreage for highbush blueberries has increased approximately 50% over the past few years, while that for lowbush blueberries has increased by 33%. Almost 99% of lowbush blueberries produced in North America are used for processing (Brazelton and Strik 2007).

Rabbiteye, southern highbush, and northern highbush cultivars are the major types of blueberries grown in Georgia. Among these cultivars, rabbiteye (*Vaccinium ashei*) is the most important type, as it is the easiest and most productive cultivar that can be grown in Georgia (Krewer and Nesmith 2006). Rabbiteye blueberries have a sweet taste and also outstanding firmness and shelf life. Additionally, they grow well in high temperatures and low-organic-matter soils. Ripening starts in late May in southern Georgia (Fonsah and others 2008). Rabbiteye blueberries are larger in size than lowbush; furthermore, rabbiteye and highbush blueberries are distinguished by their organic acid composition profiles (Kalt and McDonald 1996).

The consumption of blueberries has been increasing since 1990 (Vega-Galvez and others 2009). The increase in blueberry production and markets are attributable to the marketing campaigns that started in 1997 which emphasized the health benefits of blueberries (Brazelton and Strik 2007). Sales have kept pace with production as consumers desire the health promotion, unique flavor, and nutritional value of blueberries (Feng and others 1999; Diaz and others 2011). Consumer demand for blueberries is increasing rapidly, and as a result, worldwide markets for fresh and processed blueberries will grow (Brazelton and Strik 2007). Blueberries are also used as ingredients in a variety of food products such as pastries, beverages, cereal, and dairy products (Kalt and others 2000).

Blueberry Pomace

It is reported that approximately 5.0 million tons of non-citrus fruits were used for juice and wine processing in the US, and this accounts for up to 52% of total fruit processing (Park and others 2010), with blueberries being a prominent example for juice processing (Lee and Wrolstad 2004). As a result, significant amounts of pomace, or presscake, are left after juice

processing, accounting for up to 20% of the initial weight of the fruits (Khanal and others 2009). By-products after juice processing include seeds, stems, and skins (Lee and Wrolstad 2004). Blueberry pomace is a great source of health promoting compounds such as anthocyanins, polyphenolics (Lee and Wrolstad 2004), and procyanidins (Khanal and others 2009). Blueberry pomace can be used in new value-added products, such as natural colorants and nutraceuticals (Lee and Wrolstad 2004). Thus, finding ways to process pomace so that it can be easily used in products can help lower the economic and environmental costs associated with disposal (Khanal and others 2010)

Phytochemicals

Phytochemicals are classified as secondary metabolites synthesized by plants (Naczk and Shahidi 2006) and are responsible for protecting against oxidative damage (Syamaladevi and others 2012). Phenolic compounds, l-ascorbic acid, and carotenoids are considered to act mainly as protective metabolites in vegetables and fruits (Naczk and Shahidi 2006). Fruits, particularly berries, are rich in phenolic compounds. Fruit phenolics, both flavonoids and non-flavonoids, influence qualities such as color and shelf stability. In particular, the flavonoid group is very important to human health due to its antioxidant and other biological properties (Kalt and others 2007).

Blueberries are high in phenolic acids, flavanols (catechins), flavonols, anthocyanins, proanthocyanidins, and hydroxycinnamates (Kalt and McDonald 1996; Prior and others 1998; Ayaz and others 2005). Gallic, caffeic, *p*-coumaric, ferulic, and ellagic acids are also found in blueberry crops (Sellappan and others 2002). Additionally, blueberry leaves are known to possess high level of phenolics (Naczk and others 2003).

Anthocyanins are an important group of flavonoids, which give color (blue, red, and purple) to fruits and vegetables (Lohachoompol and others 2008). Blueberries, strawberries, cranberries, and grapes are examples of fruits containing anthocyanins (Camire and others 2002). The anthocyanin content is high in fruit skins compared to the flesh, particularly in berry fruits (Basu and others 2010). The color of blueberries changes dramatically as they ripen, and this is a result of a significant increase in anthocyanin concentration (Kalt and others 2007). While anthocyanin concentration increases during ripening, the total phenolics contents and antioxidant capacity decreases (Kalt and others 2003).

Blueberries have complex anthocyanin patterns and high total anthocyanin content compared to other berries (Kalt and others 2007; Zheng and Wang 2003). The following anthocyanins are present in blueberries: malvidin 3-O-galactoside, delphinidin 3-O-galactoside, delphinidin 3-O-arabinoside, petunidin 3-O-galactoside, petunidine 3-O-arabinoside, malvidin 3-O-arabino-side, cyanidin 3-O-glucoside, cyanidin 3-O-galatoside, cyanidin 3-O-arabinoside, delphinidin 3-O-glucoside, malvidin 3-O-glucoside, peonidin 3-O-glucoside, peonidin 3-O-galactoside, peonidin 3-O-arabinoside, and peonidin 3-O-glucoside (Lohachoompol and others 2004). Different structures of anthocyanins will result in different colors of blueberries, with different antioxidant effects (Kalt and others 2003).

Proanthocyanidins are class of flavonoids that has unique bioactivity and structure. Only a small amount is found in blueberries. They compromise of oligomers and polymers of the flavonol monomers, cathechin and epicatechin (Kalt and others 2007).

Health Benefits

The antioxidants in fruits and vegetables became a hot topic for research in the mid-1990s, as studies began to show the positive effects of antioxidants on cardiovascular diseases,

cancer, and age-related macular degeneration. Phenolics, vitamin C, tocopherols, and carotenoids are the most important groups of fruit and vegetable antioxidants (Kalt and others 2007). Fruits, especially berries, have various biological effects, such as antioxidant and anticarcinogenic properties, because they are high in phenolic compounds, flavonoids, and phenolic acids (Zheng and Wang 2003). It is known that blueberries have higher antioxidant properties than other fruits and vegetables. The total phenolics contents and anthocyanins have a great effect on the antioxidant capacity of blueberry fruits (Prior and others 1998).

Blueberries provide benefits with regard to neurobiology. Cognitive problems due to aging and oxidative stress can be lessened by consumption of blueberries (Kalt and others 2007; Zafra-Stone and others 2007). In addition, compounds in blueberries improve intracellular communication in brain cells by enhancing dopamine release in the brain (Zafra-Stone and others 2007).

Many studies have reported that blueberries have an anti-cancer effect (Nichenametla and others 2006). In particular, it is known that pterostillbene in blueberries is effective in protecting against colon cancer. Furthermore, blueberry anthocyanins improve cardiovascular health by minimizing platelet aggregation and inflammatory responses, and protecting permeability of vasculature (Zafra-Stone and others 2007). Basu and others (2010) reported that blueberries, cranberries, chokeberries, and strawberries had positive effects on lipid peroxidation, LDL oxidation, dyslipidemia, total plasma antioxidant capacity, and glucose metabolism. Also, they recommended blueberries as an essential fruit group to maintain heart health.

Blueberry anthocyanins also provide benefits with regard to vision. They improve vision by enhancing retinal pigment generation, decreasing diabetic retinopathy, increasing circulation

in the retina capillaries, and preventing or reducing retinitis pigmentosa, glaucoma, and cataracts (Zafra-Stone and others 2007).

Procyanidins in blueberries have cardio-protective effects (Brownmiller and others 2009). They also can decrease adhesion of bacteria such as *E. coli* that are factors in urinary tract infection, as well as reducing the risk of stomach ulcers by interfering with the binding of specific pathogenic bacteria to tissues (Kalt and others 2007).

Effect of Processing on Antioxidant Activity

In recent times, consumers have become interested in fruit and vegetable consumption due to the health benefits. However, dried, frozen, and canned products are being selected as substitutes for fresh products because of their convenience and longer shelf life. Various types of processing are necessary for these products to maintain freshness and physical integrity (Schmidt and others 2005). Processing methods such as drying, juicing, and pureeing of blueberry fruits can cause anthocyanin and phenolic loss due to thermal treatment, enzymatic degradation, oxygenation, and pH effects (Chakraborty and others 2010). The antioxidant capacity of processed blueberry products is strongly related to phenolic components, particularly anthocyanin content (Prior and others 1998). The total phenolics content of blueberries is mainly influenced by anthocyanin flavonoids and hydroxycinnamic acids such as chlorogenic acid (Kalt and others 2000). Anthocyanins are sensitive to heat, pH, oxygen, and storage conditions (Francis 1989). In general, it is known that highly processed food products have a lower antioxidant capacity compared to non-processed and minimally processed foods (Kalt and others 2000).

Schmidt and others (2005) compared the total phenolics content of fresh blueberries; IQF (individually quick frozen) blueberries, freeze dried, spray dried, heat dried, and cooked

blueberries; juice concentrate; pie filling; and jam. They reported that products that were not heat treated had higher phenolic levels compared to heat processed blueberry products. In addition, they concluded that although processed blueberries maintain high levels of antioxidant activity and total phenolics content compared to fresh and frozen blueberries, there was a significant loss in bioactivity such as antiproliferative activity (Schmidt and others 2005). Kwok and others (2004) demonstrated that dehydration reduced the total phenolics and anthocyanin content, and antioxidant capacity of Saskatoon berries. Mejia-Meza and others (2010) also reported that drying resulted in loss of polyphenols and anthocyanins, and antioxidant capacity of raspberries.

Drying

Drying is the most widely used method for food preservation (Tunde-Akintunde and Afolabi 2010). It is also a very important technique used in food processing (Lin and others 1998). Moisture removal from the product is the most essential operation in dehydration, and through moisture removal, microbial growth and moisture-meditated deteriorative reactions are minimized (Sacilik 2007). Furthermore, enzymatic activities and chemical and physical changes during storage are reduced (Koc and others 2008). To ensure longer shelf life and less perishable food products, free water in food is reduced to such a level that these reactions are minimized (Kwok and others 2004). Dehydration significantly reduces the volume and weight of food products, and as a result, costs of packaging, storage, and transportation are minimized; furthermore, dehydrated foods can be stored under ambient temperature conditions (Sacilik 2007; Zomorodian and Dadashzadeh 2009).

Drying is a complex process that includes simultaneous heat and mass transfer, which can cause permanent changes in physical and/or chemical properties (Tunde-Akintunde and Afolabi

2010). Changes in physical properties (volume, area, size, and shape) of food products are results of moisture loss from the interior area to the surface and surrounding air during drying (Kerdpiboon and others 2007). Shrinkage and changes in microstructure, such as pore formation, are due to stresses applied to cellular structure by heating and moisture loss (Koc and others 2008).

Drying-Rate Curve

The drying process is comprised of a series of drying stages, each with different rates (Figure 2.2). The initial moisture removal starts as the material and water begin to heat up (AB in Figure 2.2). The surface of the material approaches the wet-bulb temperature at this stage. The next stage is the constant rate period (BC). In this stage, a significant amount of water is removed at a constant rate until the product reaches its critical moisture content (Xc). Typically, temperature stabilizes near the wet-bulb temperature. A falling rate period (CE) follows after the constant rate period as the moisture content of the material falls under the critical moisture content level. There are one or more falling rate periods after the constant rate period. Hygroscopic food products are known to have two or more falling rate periods. During the falling rate period, the moisture removal rate decreases as the material reaches equilibrium with the air. In addition, the product temperature increases and approaches the temperature of the drying medium (Kerr 2007).

Effect of Drying on Foods

Changes in food products occur during drying, and this may result in diminished quality compared to fresh products. Thus, it is important to minimize the changes during the dehydration process while maximizing efficiency of operation. Texture, flavor or aroma, color, and nutritional value are the main quality attributes that are affected by drying.

Particularly in solid foods, quality deterioration is mainly due to loss of texture. Food products become more viscous as they dry, and they may also go through a series of leathery and rubbery states until most of the moisture is removed. Changes in structural polymeric compounds, aggregation and denaturation of protein, and loss of water-holding capacity are the main reasons for severe changes in texture. Generally, foods dried at high temperatures by rapid drying experience greater changes than foods dried by moderate-rate drying and lower temperatures. Particularly when drying fruits, high air temperatures can cause case hardening. Case hardening is the formation of a hard, crusty, and impermeable outside layer on food products due to physical and chemical changes in solutes. It is known that case hardening reduces the extent of diffusion and hence the drying rate.

In general, fresh foods have more flavor than dried foods, as there is loss of volatile compounds in addition to moisture removal during drying. Temperature, moisture content of the product, and vapor pressure of volatiles are important factors influencing volatile loss. High economic value foods such as herbs and spices, which possess characteristic flavors, are dried at lower temperatures because volatiles are lost during the early stage of drying. Oxidative or hydrolytic enzymes change flavors in foods. Adding sulfur dioxide, ascorbic acid, or citric acid to dried fruits; blanching vegetables; and pasteurizing fruit juices and milk are methods used to prevent flavor changes.

The color of foods is important to their acceptability. During dehydration, color change in foods can be influenced by drying temperature, time, oxidation, and pretreatments. In particular, in vegetables and fruits, heat and oxidation can result in chemical changes to food pigments such as carotenoid, chlorophyll and anthocyanins. For example, vegetables lose their green color during drying due to conversion of chlorophyll to olive-colored pheophytin as a

result of losing some of the magnesium in the pigment molecules. Lower drying temperatures, shorter drying times, blanching, osmotic pre-treatment, sulfur-dioxide or ascorbic acid treatment, and vacuum or nitrogen packing reduce pigment losses during dehydration (Fellows 2000).

Type of Drying

Sun-drying is the most basic and traditional way of drying foods, which uses solar energy as the source of heat (Sacilik 2007). Solar energy is preferred as an alternative source of energy since it is inexpensive, abundant, reuseable, and environment friendly (Doymaz 2012). On the other hand, a long drying period is necessary for sun-drying, and it also is difficult to control the final moisture content of foods because solar energy is influenced by daily weather conditions (Lee and Kim 2009). Additionally, wind-borne dirt and dust and infestation by insects, rodents, and other animals can cause serious problems (El-Beltagy and others 2007). Sun-drying has lost some importance due to the development of newer technologies; nevertheless, it is still considered an effective method for food preservation in many places around the world. For example, in Portugal, sun-drying is used for drying regional pears because of the distinctive taste, texture, and color produced (Ferreira and others 2008).

Hot-air drying is one of the oldest methods used for drying food products. During air-drying, food products are introduced to continuously flowing hot air for moisture removal (Ratti 2001). Convection is the major mode for heat transfer in hot-air drying. The size and geometry of the food products; the physical flow path of the air; the physical properties of the air such as temperature, humidity, and velocity; and the design characteristics of the drying equipment affect the drying time and rate during hot-air drying (Beaudry and others 2004). Hot-air drying is less influenced by climate conditions, and hygienic conditions can be improved compared to traditional sun drying (Fang and others 2010). However, low energy efficiency and long drying

times during the falling rate period are the main downsides in hot-air drying. This is due to reduced heat and mass transfer caused by shrinkage and rapid drops in surface moisture. Long exposure to high drying temperatures may cause quality degradation in color, flavor, texture, and nutrients. In addition, bulk density and rehydration capacity is significantly affected by shrinkage during drying (Feng and Tang 1998).

The use of microwave drying has increased as its use can prevent the significant quality loss inherent in sun-drying and hot-air drying. Furthermore, it is fast and effective compared to hot-air drying (Askari and others 2009). Because drying time is substantially decreased, microwave drying has the potential to produce better quality products. Microwaves are electromagnetic energy at frequencies between 300 MHz and 300 GHz. During drying, there are direct interactions between microwaves, and heat is generated volumetrically (Feng and others 2001). During microwave drying, heat is generated by rotation of water molecules and ionic particles, as they absorb EMR (Li and others 2010a). Drying time, speed, and efficiency are mostly influenced by microwave power and drying temperature (Li and others 2010b).

Uneven heating, limited penetration of microwaves into the product, and textural damage are the main disadvantages of microwave drying. To overcome these problems, it has been combined with other types of drying, such as hot-air drying, vacuum drying, freeze drying, and spouted bed drying (Zhang and others 2006; Li and others 2010a). For example, Feng and Tang (1998) combined microwave drying with spouted bed drying to reduce the drawbacks of using microwave drying alone. Excessive microwave power will lead to loss of quality and textural change by puffing due to rapid mass transfer (Nijhuis and others 1998). Furthermore, when the microwave power exceeds 500 W, arcing can be a problem (Cohen and Yang 1995).

Overheating and irreversible drying out due to excessive heat at the corners and edges of the product can result in the development of off flavors (Zhang and others 2006).

Freeze drying is known as one of the most sophisticated methods of drying. During freeze drying, ice sublimates directly to vapor, bypassing the liquid state. As a result, a porous shell of dehydrated material remains, and the sublimation interface starting from outside of the product recedes (George and Datta 2002). Freeze dried products reconstitute with water very quickly and closely resemble their original form when reconstituted. In addition to their superior rehydration quality, minimized shrinkage and porous structure add quality to freeze-dried products. A relatively low drying temperature minimizes damage to biological compounds that are sensitive to heat. Better flavor, taste, and volatile retention compared to other dried products are additional advantages of freeze drying (Krokida and Philippopoulos 2006). Structure and shape of the products are protected by the solid state of water during freeze drying. Microbial growth and product deterioration are restricted since the drying is done in a moisture free and low temperature environment (Ratti 2001). The main drawback of freeze drying is the high cost due to relatively long drying time compared to other drying methods. The relatively low vapor pressure driving force is the main reason for the extended drying time (George and Datta 2002).

Spray drying is a widely used method for making dried food powders (Kajiyama and Park 2011). It is also commonly used by pharmaceutical industry. Limited types of foods can be spray-dried including liquids, purees, and low viscosity pastes that can be atomized into small particulates. Moisture is removed by contact with hot air in the spray dryer chamber (Sollohub and Cal 2010). Many studies have been done on spray drying of fruit powders such as tomato pulp, passion fruit juice, and pomegranate juice (Goula and Adamopoulos 2008; Angel and others 2009; Vardin and Yasar 2012).

Drum drying is one of the least expensive drying techniques and is particularly suited for heat resistant products. Thin layers of food products such as purees, pastes, mashes and liquids are continuously applied to heated surface of a rotating drum. Steam circulates in the drum interior and provides heat to the drum surface. When the temperature reaches the boiling point of water, the food product starts to dehydrate. Depending on the type of food, drying time usually takes 1min or less when the thickness of the product is less than 2mm. Typically, the temperature of the drum surface is above 100°C, and can often be as high as 150°C. Drum surface temperatures above 120°C are used for rapid drying, but does result in product with more cooked color and flavor compared to products dried at lower temperatures. Milk, heat tolerant purees such as tomato paste, potato mash, and animal feeds are the products mostly commonly used for drum drying. Dried fruits and juices are not suitable for drum drying due to their stickiness (Potter and Hotchkiss 1995).

Vacuum drying is another alternative to conventional air drying (Lee and Kim 2009). During vacuum drying, moisture is removed under a low pressure environment (Jaya and Das 2003). Thus, moisture starts to evaporate at a lower temperature compared to conventional drying methods. Consequently products are dried at lower temperature resulting in higher quality products (Sunjka and others 2004). The main modes for heat transfer in vacuum drying are conduction and radiation. Drying temperature and intensity of the vacuum significantly affect the drying rate and heat damage to the product (Potter and Hotchkiss 1995). Vacuum-dried products have a puffed and frothy structure because air and moisture inside the product expands due to vacuum. This allows high rate of heat and mass transfer. In addition, oxidation of the product is minimized since drying is accomplished under an air-deficient environment. Vacuum drying is an appropriate method for heat and oxygen sensitive foods such as fruits and

vegetables (Jaya and Das 2003). It has been applied to mango powder (Jaya and others 2006) and honey powder (Sahu 2008).

Vacuum-Belt Drying

Vacuum belt drying is a novel method which works continuously, and can be used for fruit or fruit juice drying. The material to be dried is introduced through an air-lock and is conveyed by belt over a series of plate heaters. Vacuum-belt dried products have high quality compared to other dried products, and also the drying time is relatively short (Wang and others 2007). It is considered as a good method for drying hygroscopic products, sticky and pasty foods. As the drying occurs under low oxygen pressure, oxidative degradation is very low compared to other drying methods (Jaya and Das 2009). Minimal quality loss, convenience of drying, and low costs are other advantages of vacuum belt drying (Liu and others 2009; Wang and others 2007). High quality foods such as instant tea, citrus juice, herbal extract, and other delicate liquid foods are produced by vacuum belt drying in the industry (Potter and Hotchkiss 1995).

Liu and others (2009) studied the mathematical model of vacuum belt dried natural herb extract (*Panex. notoginseng*) to find out the influences of drying temperature, belt speed, and feeding speed on rate of the drying. Three different drying temperatures (90°C, 100°C, 110°C), feeding speed (15 min/ml, 20 min/ml, 25 min/ml), and belt speed (4 cm/min, 7 cm/min, 10 cm/min) were used. They showed that the dehydration process was greatly affected by temperature and belt-speed.

Liu and others (2011) demonstrated that the quality of *Panax notoginseng* extract was significantly affected by different drying methods (spray drying, freeze drying, vacuum drying, and vacuum belt drying). They concluded that vacuum belt drying is the most suitable method

for drying *P. notoginseng* extract. Shorter drying time, lower moisture content of the end product, and higher hydrogen peroxide scavenging activity in final products were advantages of vacuum belt drying.

Vashisth and others (2011) investigated the effect of drying types (vacuum belt drying, freeze drying, hot-air drying) on muscadine pomace. They demonstrated that vacuum belt drying is a promising method for muscadine pomace drying, as it resulted in shorter drying time with proper moisture content, water activity, and high nutritional value.

Although vacuum belt drying has many advantages, volatile loss could be a problem. A study conducted by Wang and others (2007) compared volatiles of banana powder dried by vacuum belt drying, freeze drying, and air drying. They demonstrated that freeze drying is a suitable drying technique for banana puree drying with the optimum aroma followed by vacuum belt drying and then air drying. Damage of original volatile compounds and formation of new compounds occurs, because of the relative high dehydration temperature of vacuum belt drying and air drying.

Fruit Powder

Fruit powders have a variety of nutritional and functional benefits and they add value to food products (Grabowski and others 2008). Additionally, they are stable at ambient temperature, have long shelf life when properly packaged, and rehydrate well to their original juice form (Gabas and others 2007; Benedetti and others 2011). Fruit powders are widely used for baby foods, extruded cereal products, soups, dips, marinades, sauces, snack fillings, and confections (Jakubczyk and others 2010; Grabowski and others 2008).

On the other hand, drying of fruit pulps and juices into powder is difficult because of their stickiness and thermoplascity during drying (Vardin and Yasar 2012). The stickiness of

fruit powders is due to presence of low molecular weight sugars such as sucrose, glucose, fructose and acids which contribute to low glass transition temperature (T_g) and high molecular mobility at high temperatures. The glass transition temperature of sucrose is 62 °C, fructose is -5°C, and glucose is 32°C (Jaya and Das 2004; Roos 1995). In particular, the T_g of pure components in foods significantly affects the T_g of dried products. The T_g of a food system is a critical marker for several physical and chemical changes. As the temperature increases above T_g, a glassy food turns into a lower viscosity rubbery state, with an increase in molecular mobility. Foods are often stable at $T<T_g$, however at $T> T_g$, they often become sticky and undergo structural collapse and color change (Jakubczyk and others 2010; Phanindrakumar and others 2005). For this reason, fruit powders should be stored at temperatures below T_g, with an appropriate amount of water to act as a plasticizer (Bhandari and others 1993). It should be noted that the T_g of a food is quite dependent on moisture content; generally, the lower the moisture content the higher is the T_g.

Drying aids and anticaking agents can also be added to food materials to overcome hygroscopicity of fruit powders at high temperatures and humidities during dehydration. Drying aids have high T_g, so by adding these T_g of samples can be increased. High molecular weight drying aids such as maltodextrin, gum Arabic, cashew gum, isolated protein, carboxylmethyl celluolose, pectins, and calcium silicate are added to improve hygroscopicity and flowability of powders (Jaya and others 2006; Roos 1995; Gabas and others 2007; Jaya and Das 2009; Jakubczyk and others 2010; Jaya and Das 2004; Comunian and others 2011; Tonon and others 2011; Angel and others 2009; Goula and Adamopoulos 2008; de Oliveira and others 2009; Rodríguez-Hernández and others 2005). In addition, anti-caking agents such as tricalcium

phosphate, phosphates, silicon dioxide, silicates and modified carbohydrates are added to prevent caking and enhance the flow behavior of powders (Jaya and others 2006).

Maltodextrins

Among many high molecular weight additives maltodextrins are considered to be the most effective, particularly when considering their low cost. Maltodextrins are hydrolysates of corn starch, and come as a white powder that is a blend of polysaccharides, oligossachrides, and glucose. It consists of β-D-glucose units primarily connected by glycosidic (1-4) bonds (Figure 2.3). They are typically classified by their molecular weight, as measured by the dextrose equivalent (DE). The reducing capacity of maltodextrin is determined by the DE and there is an inverse relationship between DE and molecular weight. For example, a higher DE coincides with a shorter chain length (Gabas and others 2007). Table 2.1 shows glass transition temperatures and molecular weights of several mono- and di- saccharides (glucose, sucrose, fructose and maltose), along with those of maltodextrins. The T_g of the foods is affected directly by DE. Maltodextrins with lower DE values have higher glass transition temperatures. Thus, maltodextrins with lower DE values are more effective at preventing powder stickiness and promoting flowability (Gabas and others 2007). Maltodextrins are mostly used to improve product stability and hygroscopicity of fruit juices, flavorings, and sweeteners which are difficult to dry (Gabas and others 2007).

Some studies have shown that maltodextrins are good drying aids. Jaya and others (2006) showed that adding maltodextrin (drying aid) and tricalcium phosphate (anti-caking agent) improved the hygroscopicity and flowablility of mango powders. Jakubczyk and others (2010) showed that the hygroscopicity of apple puree powder was considerably reduced by

adding maltodextrin. Benedetti and others (2011) reported that persimmon pulp powder with maltodextrin absorbed less water compared to pure persimmon powder.

Figure 2.1 Blueberries (http://www.blueberrycouncil.org/blueberry-news/library/#photos)

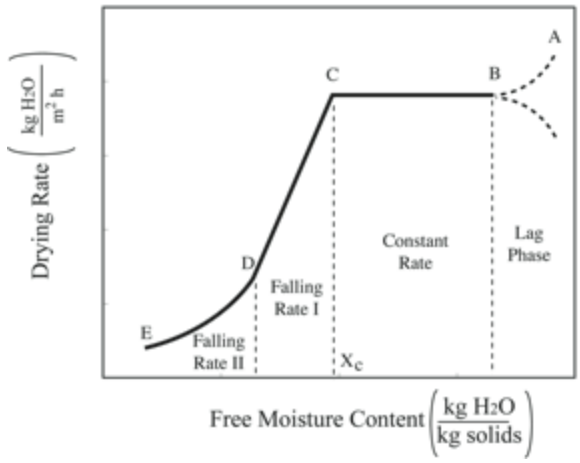

Figure 2.2 Drying rate curve

(Kerr WL. 2007. Food drying and evaporation processing operations. In: Kutz, M., (editor). Handbook of farm, dairy, and food machinery. Norwich, NY: William Andrew Publishing)

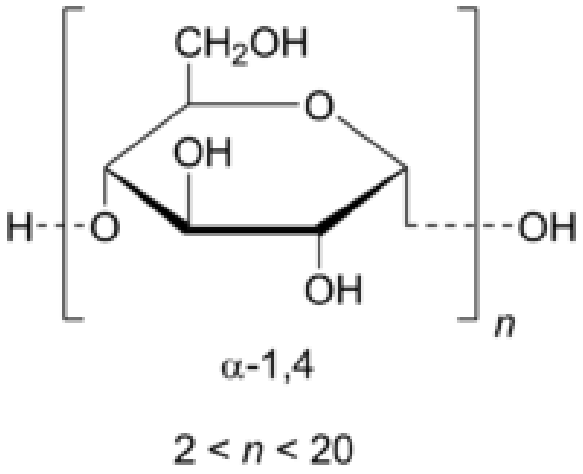

Figure 2.3 Maltodextrin (http://en.wikipedia.org/wiki/File:Maltodextrin.png)

Food Material	Molecular weight (g/mol)	T_g (°C)
Fructose	180	5
Glucose	180	31
Sucrose	342	62
Maltose	342	87
Matodextrins		
DE 36	500	100
DE 25	720	121
DE 20	900	141
DE 10	1800	160
DE 5	3600	188

Table 2.1 Glass transition temperature of sugars and maltodextrins

(Hebbar HU, Rastogi NK & Subramanian R. 2008. Properties of Dried and Intermediate Moisture Honey Products: A Review. Int. J. Food Prop. 11(4):804-819)

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CHAPTER 3

VACUUM-BELT DRYING OF RABBITEYE BLUEBERRY SLURRIES: INFLUENCE OF DRYING CONDITIONS ON PHYSICAL PROPERTIES OF BLUEBERRY POWDER[1]

[1] Kim, M. and Kerr, L.W. To be submitted to *Journal of Food Science*.

Abstract

This study investigates the influence of vacuum-belt drying conditions on the physical properties of a powdered product obtained by drying blueberry slurry. Drying was performed in a laboratory-scale vacuum belt dryer (Zwag, LKM-101, Zchokke Wartman Ltd. Bucher, Dottingen, Switzerland) at three different temperatures (80°C, 95°C, 110°C). Maltodexrtin (MD) was used as the drying agent at three levels (0.3 kg/kg dry blueberry solid, 0.45 kg/kg dry blueberry solid, and 0.6 kg/kg dry blueberry solid). Two-way ANOVA indicated that increasing the MD level and drying temperature decreased the hygroscopicty of the powders. Moisture adsorption isotherms were developed, showing Type Ⅲ behavior, and were fit with the Guggenheim-Anderson-de Boer (GAB) model. "Monolayer" moisture (m_o) decreased from 8.8 to 8.18 g H_2O/100g solids with increasing MD levels. Higher levels of MD also improved the flowability of the powders.

Introduction

The market for blueberry products is rapidly growing due to their unique flavor, nutritional value (Feng and others 1999) and various health benefits (Diaz and others 2011). Blueberries are known to possess high antioxidant capacity (Kalt and others 2007), which is related to the phenolic compounds and anthocyanins they contain (Prior and others 1998). Blueberry anthocyanins help lower the risk of heart disease, inhibit carcinogenesis and lower anti-inflammatory activity in the brain (Lohachoompol and others 2008). Unfortunately, blueberries have a short growing season and freshly picked fruit have limited shelf life even under refrigeration (Feng and others 1999). In addition, anthocyanins are sensitive to conditions of processing and storage (Pavón-García and others 2011). To mitigate these problems, further processing including freezing, canning, or drying is often used.

Drying is one of the most common and important methods of food preservation (Tunde-Akintunde and Afolabi 2010). By removing water from the food, microbial growth and moisture-meditated deteriorative reactions are minimized (Sacilik 2007). However, drying fruit juice is not easy due to its stickiness and thermoplasticity (Vardin and Yasar 2012). The presence of low molecular weight sugars (such as sucrose, glucose, and fructose) and organic acids that contribute to low glass transition temperatures (T_g) and high molecular mobility are the main reasons for the stickiness of fruit powders (Jaya and Das 2004).

Dried fruit powders have various nutritional and functional benefits and are considered as ingredients for adding value to food products (Grabowski and others 2008). They also have relatively long shelf life at ambient temperatures when packaged properly (Benedetti and others 2011). Fruit powders are ideal for use in extruded cereal products, baby foods, sauces, soups, dips, marinades, confections, and snack fillings due to their convenience, characteristic color,

flavor, nutrient value, and water binding properties (Grabowski and others 2008; Jakubczyk and others 2010).

High molecular drying aids such as maltodextrin, gum Arabic, starch, cashew gum, and isolated protein, as well as anticaking agents such as tricalcium phosphate, are often added to make non-sticky and free flowing fruit powders (Jaya and Das 2004; Rodríguez-Hernández and others 2005; Goula and Adamopoulos 2008; Vardin and Yasar 2012; de Oliveira and others 2009; Angel and others 2009; Adhikari and others 2004). Among these additives maltodextrins are considered to be the most promising, particularly when considering their low cost. They consist of partially hydrolyzed starches that have a bland flavor. They are typically classified by their molecular weight, as measured by the dextrose equivalent (Cai and Corke 2000).

Vacuum-belt drying (VBD) is a relatively new continuous drying method that has been used for fruits or fruit juices. The material to be dried is introduced through an air-lock and is conveyed by belt over a series of plate heaters. Vacuum-belt dried products have high quality, while the drying time is relatively short compared to other dying methods (Wang and others 2007). It is also suitable for drying sticky and hygroscopic food products (Liu and others 2011) and slurries (Vashisth and others 2011). As the drying occurs under low oxygen pressure, oxidative degradation is very low compared to other drying methods (Jaya and Das 2009). VBD has been applied to a limited number of products such as peach juice (Cesare and Nani 1995), banana powder (Wang and others 2007), *Panax notoginseng* extract (Liu and others 2011), and muscadine pomace (Vashisth and others 2011).

The objective of this study was to investigate the use of vacuum belt drying to produce dried powders from whole blueberries. One aspect of this was to determine optimal drying temperatures for producing powders with good properties. Preliminary studies indicated that

powders from pure berries were too sticky, thus we studied different levels of maltodextrin (MD) combined with tricalcium phosphate (TCP) to improve the physical properties of the powder. To assess end-quality, several powder properties were measured including moisture sorption isotherm behavior, hygroscopicity, flowability, and color.

Materials & Methods

Sample Preparation

Frozen rabbiteye blueberries (*Vaccinium ashei* 'Brightwell') were donated from the University of Georgia Research and Demonstration Farm (Alma, GA). Frozen blueberries were thawed in a walk-in cooler at 4°C for 24 h prior to processing. Thawed blueberries were ground in a food processor (Model KFP600WH, Kitchen Aid, St.Joseph, MI) for 2 min. Three different levels of maltodextrin (Star-Dri 10, Tate & Lyle, Decatur, IL) were added to the blueberry slurry: 0.3kg per kg dry blueberry solid, 0.45 kg/kg dry solid, and 0.6 kg/kg dry solid. The mixture was further blended for 5 min. Equal amounts of tricalcium phosphate (16.7g TCP per kg dry blueberry solid) was added to each sample and mixed with a food processor for 2 min. Samples were kept at 4°C prior to drying.

Vacuum-Belt Drying

A laboratory-scale vacuum belt dryer (Zwag, LKM-101, Zchokke Wartman Ltd. Bucher, Dottingen, Switzerland) was used to dry blueberry slurries. The dryer comprised an evacuated chamber with a 20.33 cm-wide Teflon-coated conveyor belt that passes over 3 independently controlled heating plates, and one cooling plate (Figure 3.1). A radiation plate 22.9 cm wide was located above the belt and spanned the length of the conduction heating plates. A DVT Aqua Seal 80 CFM vacuum pump (Dekker vacuum technologies, INC., Michigan City, IN) was used to pull a vacuum on the chamber. The pressure was held between 3.3 kPa-60 kPa (25 torr and

450 torr) throughout drying. An integrated touch screen panel and programmable logic controller were used to specify heater temperatures.

Three different temperatures (80°C, 95°C, or 110°C) were used to dry the blueberry slurry. The three conduction plates and the radiation plate were set to the specified temperature. Approximately 500 g of slurry were processed in each drying run. Samples were dried to reach a target water activity (a_w) of 0.2 ± 0.02. At 80°C samples were dried for 70 min, at 95°C for 60 min, and at 110°C for 50 min. In most cases, the full vacuum was not introduced immediately to limit over-expansion of the slurry and explosion of the sample onto the walls of the dryer. Thus, at 80°C, the pressure was held at 30 kPa (225 torr) for ~35 min before reducing it to 3.3 kPa (25 torr). At 95°C, the initial pressure was 50.7 kPa (380 torr) for 20 min before proceeding to 3.3 kPa (25 torr). At 110°C, a pressure of 60 kPa (450 torr) was maintained for 20 min, before proceeding to 3.3 kPa (25 torr).

Powder Grinding

Dried samples were cooled in desiccators at room temperature for 5 min. About 15~20 g of cooled samples were placed in a Model 111338 grinder (General Electric Co., Fairfield, CN) and ground with 5 to 7 g of dry ice for 15 s. Dry ice was added to prevent excessive heat build-up during grinding. Final products were sealed in PET/Al pouches and kept frozen at -20°C.

Moisture Content and Water Activity

AOAC Method 934.06 was used to determine the moisture content of the blueberry powders with some modifications (AOAC 1995). Approximately 1.5-2 g of blueberry powder samples were weighed in 5 cm diameter aluminum pans. Samples were dried in a vacuum oven (Model VWR 1430 MS, Optics Planet Inc., Northbrook, Il, USA) at 70°C and 40 kPa for 24 h, or

until a constant weight was attained. The moisture content was calculated both on the wet basis (M_{wb}) and the dry basis (M_{db}) from the initial (w_i) and final (w_f) sample weight:

$$M_{wb} = \frac{w_i - w_f}{w_i} \times 100 \quad (1)$$

$$M_{db} = \frac{w_i - w_f}{w_f} \times 100 \quad (2)$$

The water activity (a_w) of the blueberry powders was determined using an Aqualab water activity meter CX-2 (Decagon Devices Inc, Pullman,WA) at 22±2 °C. Nine samples were measured three times.

Dynamic Hygroscopicity

Dried samples were tested for their tendency to pick up moisture from the environment. Approximately 1g of blueberry powder was evenly spread on a 9 cm petri dish, so as to maximize the surface area between sample and humid air. Samples were prepared for each of the combinations of maltodextrin levels and drying temperatures: 80°C/0.3MD, 80°C/0.45MD, 80°C/0.6MD, 95°C/0.3MD, 95°C/0.45MD, 95°C/0.6MD, 110°C/0.3MD, 110°C/0.45MD, and 110°C/0.6MD. Each sample was placed on an analytical balance (Adventurer Pro AV 64, OHAUS Corporation, Pine Brook, NJ, USA) located in an environmental chamber (Model 435314, HOTPACK, PA) set to maintain constant temperature (23°C) and relative humidity (75%). As the dried samples adsorbed moisture, the mass change was monitored by a laptop computer (Model CF-74, Panasonic, Japan) connected to the balance. Weights were recorded every 5 min for 20 h, or until constant masses were attained. In general, weight changes were minimal after ~ 5 h. Measurements were performed in duplicate for each drying batch.

The data was fit to the moisture sorption equation:

$$ln\left(\frac{m_e - m_i}{m_e - m}\right) = Kt + b \quad (3)$$

The constant K describes how quickly the sample adsorbs moisture, while m_e describes the total moisture adsorbed. In addition, hygroscopicity was analyzed according to Jaya and Das (2004):

$$Hygroscopicity\ (\%) = \frac{\frac{b}{a} + m_i}{1 + \frac{b}{a}} \quad (4)$$

Where a is the initial sample weight, b the weight at a specified time (here take at 15 h), and m_i is the initial wet basis moisture content.

Moisture Isotherms

Saturated salt solutions were prepared to produce isopiestic environments with relative humidity in the range of 11 to 75%, leading to powders with a_w values between 0.11 and 0.75. The solutions included LiCl (0.11), CH_3CO_2K (0.22), $MgCl_2$ (0.33), $Mg(NO_3)_2$ (0.52) and NaCl (0.75). Triplicate samples of ~1.5 g of blueberry powder were weighed into small plastic pans and placed on plastic racks in desiccators containing the solutions. A slight vacuum was applied to the dessicator to ensure they were sealed. The chambers were stored for 4-5 weeks until the samples held at each relative humidity had attained a constant mass. The dry basis moisture content was determined by the change in mass, that is:

$$M_{db} = \frac{w_f - w_s}{w_s} \quad (5)$$

Where w_s is the mass of solids in each of the original samples and w_f is total mass of wet samples.

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

$$M_{db} = \frac{m_0 kca_w}{(1-ka_w)(1-ka_w+cka_w)} \quad (6)$$

Where m_o is the "monolayer" moisture content, c is a constant related to the surface enthalpy, and k is related to adsorption of multiple layers of moisture.

Flowability

Flowability was assessed using a rotating drum method (Jaya and others 2006; Liang and Langrish 2010). The tester was made from an aluminum cylinder 9 cm long and 12 cm in diameter, containing two removable covers. The cylinder had two 4 mm slots (70 mm long) located on opposite ends of the cylinder. A direct-drive DC motor was used to rotate the drum at 30 rpm. Before use, the drum was sprayed with 95% ethanol and dried to help limit electrostatic buildup. For each of the 9 sample trials, ~7 g of powder was introduced into the drum and the covers replaced. The drum was induced to rotate and the amount of material emerging from the slots was measured every 1 s on an HP 4200C analytical balance (Avery Weigh-Tronix, Fairmont, MN), and recorded on a laptop computer. Each sample was tested three times. Plots of the mass of powder versus time were prepared. As additional measures of flowability, the percent of powder appearing after 10 and 30 s were determined.

Color

The color of the blueberry powder was measured with a Chroma meter (Model CR-410, Konica Minolta, Ramsey, NJ). A white standard tile was used to calibrate the Chroma meter. The colorimeter was positioned from above the pertri dish filled with blueberry powder. Values of lightness (L*), chroma (C*) and hue (h) were measured three times for each treatment group. L* values, representing sample lightness, ranged from 100 (white) to 0 (black). Chroma measures color saturation and ranged from 0 to 100 (high saturation). Hue angle indicates the primary color value with associated angles of 0° (red), 90° (yellow), 180° (blue-green), and 270° (blue-red).

Statistical Analysis

The experimental design consisted of drying temperature at 3 levels (80, 95 and 110°C) and maltodextrins at 3 levels (0.3, 0.45, and 0.6 kg/kg dry blueberry solid). JMP®9.0.2 (SAS Institute Inc.) was used to analyze the data. Two-way ANOVA was performed at a confidence level of 95% ($p < 0.05$). Tukey's HSD was performed to investigate differences amongst the 9 treatments.

Results & Discussion

Hygroscopicity

Results of the powder hygroscopicity as measured by moisture uptake over time are shown in Figure 3.2. In general, powders adsorbed moisture rapidly and reached a steady moisture content after 5 to 6 h. Whole blueberry powder is expected to be hygroscopic due to its high sugar and acid content. It was observed that all samples turned a dark-brown purple color after 20 h of exposure to 75% RH. Derived constants for the sorption model (Equation 3) and the % hygroscopicity (Equation 4) are shown in Table 3.1.

Two-way ANOVA showed that the MD level had a significant effect on K values. At a given temperature, samples with higher MD levels had lower K values, suggesting that they adsorbed moisture more slowly. The differences were not large, however. For example, for samples dried at 80°C, K values ranged from 0.495 h^{-1} for samples with 0.3 kg MD/kg solids to 0.460 h^{-1} for samples with 0.6 kg MD/kg solids. Temperature was not a significant factor for absorption as measured by K. At equilibrium, moisture contents ranged from 21.3 to 24.6 gH_2O/100 g solids. Samples dried at lower temperatures and with lower MD levels absorbed more water.

Two-way ANOVA demonstrated that both MD and temperature were significant factors for hygroscopicity (%) and 'm_e' value. Tukey's test also showed that there is a significant difference amongst samples. Samples dried at the highest temperature (110°C) and with highest levels of MD (0.6 kg/kg solids) had the lowest hygroscopicity (17.5%), whereas samples with the lowest level of MD (0.3 kg/kg solids) had the highest hygroscopicity (19.8%). Other researchers have noted that the inclusion of maltodextrins decreases the hygroscopicity of fruit and vegetable powders. Goula and Adamopoulos (2008) found that the hygroscopicity of spray-dried tomato powder decreased with increasing drying temperature and maltodextrin levels. Tonon and others (2011) reported lower hygroscopicity values for spray-dried acai with a maltodextrin carrier. Jaya and Das (2004) showed that increasing levels of maltodextrin decreased hygroscopicity of mango powder. Vardin and Yasar (2012) showed that addition of maltodextrin reduced the stickiness and hygroscopicity of spray-dried pomegranate juice powder. Cai and Corke (2000) showed that the addition of maltodextrins helped reduce hygroscopicity of spray-dried betacyanin powder. Rodríguez-Hernández and others (2005) found out that the highest maltodextrin levels resulted in the least hygroscopic spray-dried cactus pear juice powders. Phanindrakumar and others (2005) reported that hygroscopicity of the freeze-dried pineapple juice powder was reduced by addition of high molecular weight additives.

Drying of fruit juices and purees can be difficult due to the presence of low molecular weight sugars and organic acids which contribute to lower glass transition temperatures (T_g). According to Roos (1995), physical changes in high sugar and low moisture dried powders, including hygroscopicity, can be related to the T_g of the powder. Adding high molecular weight polymers such as maltodextrin, gum Arabic and cashew gum can reduce T_g and problems

associated with hygroscopicity and stickiness (Silva and others 2006; de Oliveira and others 2009; Bhandari and others 1997).

Moisture Sorption Isotherm

Sorption isotherms for the samples produced at 95°C are shown in Figure 3.3. The equilibrated moisture content of the blueberry powder increased with water activity. The powders stored at different levels of water activity showed different physical characteristics. When stored at aw 0.11, powder showed free-flowing behavior, and no marked differences were observed compared to the original samples. When stored at higher water activities, samples showed signs of caking. At a_w 0.75, the powders turned into a dark and hard block due to caking. In general, at a given a_w, samples with lower MD absorbed slightly more water, and this is in concordance with the hygroscopicity results of Table 3.1.

The isotherms were most closely described by Type Ⅲ behavior according to Brunauer's classification. Type Ⅲ curves are often associated with crystalline substances such as sucrose, or those materials high in sugars. The isotherm initially shows a small amount of absorption as a_w increases from 0. At a critical a_w, water begins to dissolve crystalline components, and thereafter the hygroscopic material readily absorbs additional water (Sundaram and Durance 2008). Fruit powders often display Type Ⅲ isotherms. For example, vacuum dried pineapple powder with maltodextrin (Gabas and others 2007), freeze dried Chinese gooseberry (Wang and others 2008), and model fruit powders (Tsami and others 1998) also showed this type of isotherm. As noted, samples with lower levels of MD absorbed more water. This was most noticeable at higher water activity (a_w 0.75), suggesting that MD had a greater effect at higher a_w. It has been suggested that the addition of MD decreases hygroscopicity by decreasing the number of active sites, hydroxyl groups of small carbohydrates, that can bind with water.

Instead of associating with water, the hydroxyl groups can bind with the maltodextrins (Catelam and others 2011).

Results for the nonlinear regression fitting of the GAB model is shown in Table 3.2. All data was well-fit with the model, with $r^2 > 0.99$. So-called monolayer values (m_o) have been of interest as they relate to the amount of water that is strongly absorbed to specific sites on the surface of foods. It is often considered as an optimum value to assure food stability (Comunian and others 2011). The m_o values in this study were determined from the GAB equation and were found to range between 0.0818-0.0880 g H_2O/g solid. The m_o decreased somewhat as the amount of maltodextrin added to powder increased. Overall, m_o values were lower than those reported for fruit powders. The m_o values for pineapple powders ranged from 0.146-0.166 g H_2O/g solid at 20-50°C (Gabas and others 2007). For grapes, apricots and apples m_o values were between 0.095-0.220 g H_2O/g solid (Kaymak-Ertekin and Gedik 2004); for apples 0.107 g H_2O/g solid (Corey and others 2011); and for persimmon powder 0.144-0.166 g H_2O/g solid at 20-50°C (Benedetti-Damy and others 2010).

Values of c are most often related to the enthalpy of moisture adsorption on the powders (Pavón-García and others 2011). In this study, c values decreased as the level of MD increased. Others have found that increased MD decreased c values, such as for camu-camu pulp powder (Silva and others 2006), grapefruit juice powder (Telis and Martinez-Navarrete 2009) and persimmon pulp powder (Benedetti-Damy and others 2010).

The value of k measures the interactions between the molecules in multilayers adjacent to the adsorbent (Catelam and others 2011). In our studies, k did not vary with the level of maltodextrin. Benedetti-Damy and others (2010)also reported that k did not vary for persimmon powder vacuum-dried with a variety of encapsulating materials.

While the addition of maltodextrins did modify the sorption behavior of dried blueberry powders, resulting in less hygroscopic powders, the exact mechanisms for this action are uncertain. While the addition of biopolymers such as MD modify the balance of hydrophilic and hydrophobic sites, other structural changes may occur which effect properties such as density or porosity, which in turn effect the amount of water sorbed at a given relative humidity.

Flowability

Plots of the amount of powder flowing out the cylindrical tester over time are shown in Figure 3.4. Table 3.3 summarizes the amount of material emerging after 10 and 30 s for each of the 9 temperature/MD treatments. The two-way ANOVA analysis indicated that MD level was a significant factor, in that more powder flowed from the container in a set time with higher levels of MD. However, the differences amongst samples were not large. For example, 87.67-93.05% of the material emerged after 10 s for powders produced at 80°C; 84.68-92.16% for powders produced at 95°C; and 86.25-91.93% for powders produced at 110°C.

Wang and Zhou (2012) reported that the flowability of soy sauce powder produced with 20 and 40% of MD were not statistically different. Moreira and others (2009), however, found that drying aids (MD and cashew tree gum) enhanced the flowability of spray-dried acerola pomace powder. Jaya and Das (2004) showed that increasing amounts of MD and tricalciumphosphate decreased the flow time of mango powder. Sahu (2008) reported that flow time decreased by increasing the amount of MD and TCP in vacuum-dried honey powder.

Flowability and hygroscopicity are often correlated properties, as hygroscopic powders tend to have more resistance to flow related to absorbed water (de Oliveira and others 2009; Fitzpatrick and others 2005). Using model powders, Scoville and Peleg (1981) found that greater sorbed water leads to liquid bridges and capillary forces that increase cohesion and limit

flow. Flowability and hygroscopicity have also been related to powder glass transition temperatures. Higher T_g values are associated with less hygroscopic powders, resulting in more free flowing powder. Consequently, it can be considered that MD enhances flowability of the powder by increasing T_g. The powders also contained fixed amounts of TCP (tricalciumphosphate). TCP is an anticaking agent and it is known to improve flowability. Moy (1971) found that adding TCP to pineapple and guava juice powders enhanced the powder flowability.

Color

Color is a critical factor influencing quality of the products (Rodríguez-Hernández and others 2005). No noticeable signs of browning were detected, and all the powders displayed dark reddish-purple color. Lightness (L*) of whole blueberry powders ranged from 25.7 to 28.6, chroma (C*) from 12.8 to 14.5, and hue (h) from 354.2 to 356.6 (Table 3.4). In comparison, frozen-thawed whole blueberries had color values of L*=18.3, C*= 2.4 and h°= 325.7°. Dried blueberry powders displayed lighter, more saturated color with more reddish-purple hue compared to frozen-thawed blueberries.

The lightness (L*) of blueberries is mostly associated with their anthocyanin content, which gives the characteristic colors to blueberries. In general, drying will result in increased L*, due degradation of anthocyanins (Pallas 2011). Wang and others (2010) reported that significant degradation in anthocyanins occurred when blueberries were exposed to higher temperatures. In this study, samples dried at lowest temperature (80˚C) were darker (had lowest L*) at every maltodextrin level. In addition, there were also significant differences in L* due to maltodextrin levels. Samples with higher levels of maltodextrin resulted in lighter color

compared to samples with lower levels of maltodextrin. This may be due in part to the inherent white color of maltodextrins.

The degree of color saturation can be measured by the chroma (C*) (Abers and Wrolstad 1979). Both drying temperature and maltodextrin levels affected the chroma of the powders. However, the differences amongst samples were not large. At a maltodextrin level of 0.45 kgMD/kg dry solids and 0.6kg MD/kg dry solids, samples dried at higher temperature had higher chroma values. The change in chroma values during drying may be due to formation of browning compounds due to non-enzymatic browning (Lopez and others 2010), along with degradation of anthocyanins.

Hue is related to perceived color (Abers and Wrolstad 1979). Hue angles of samples were between 354.2˚ and 356.6˚, showing predominantly purple to red hue. Drying temperature did not significantly influence hue of the powders. Addition of maltodextrins, however, did affect powder hue. The differences in samples were relatively small. Interaction between other compounds and anthocyanins during dehydration, degradation of anthocyanin, change in composition and content of phenol are the reasons for change in hue angle (Gonçalves and others 2007).

Conclusion

Dried powders with good appearance and color were produced from blueberry slurry using a vacuum-belt drier. The addition of maltodextrin helped reduce hygroscopicity and improve flowability. While this study focused on the physical properties of the powders, subsequent studies will investigate changes in nutrient and phytochemicals due to processing and storage.

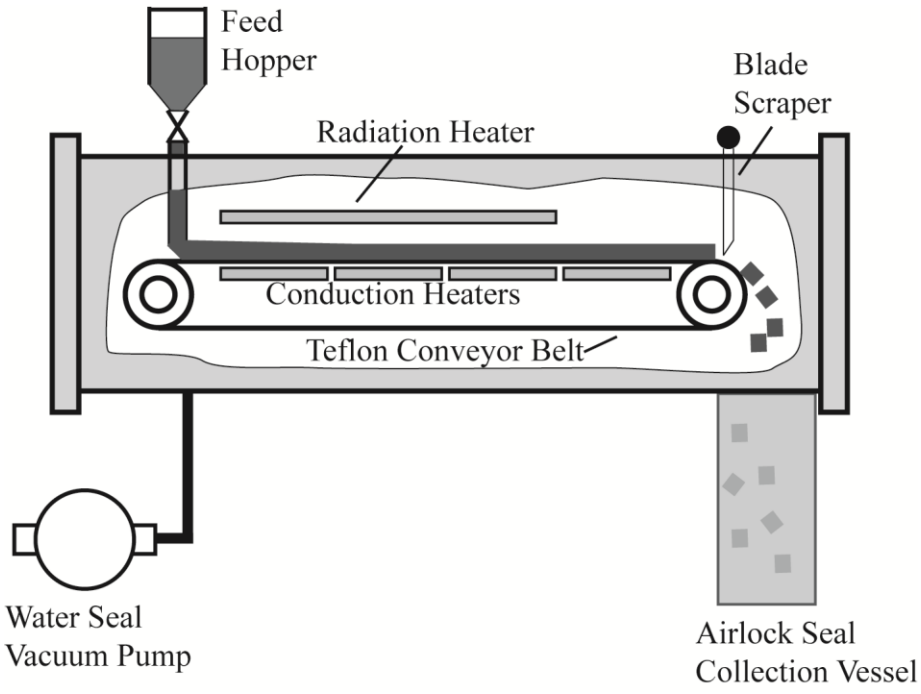

Figure 3.1 Vacuum-belt drier used to produce dried blueberry powders

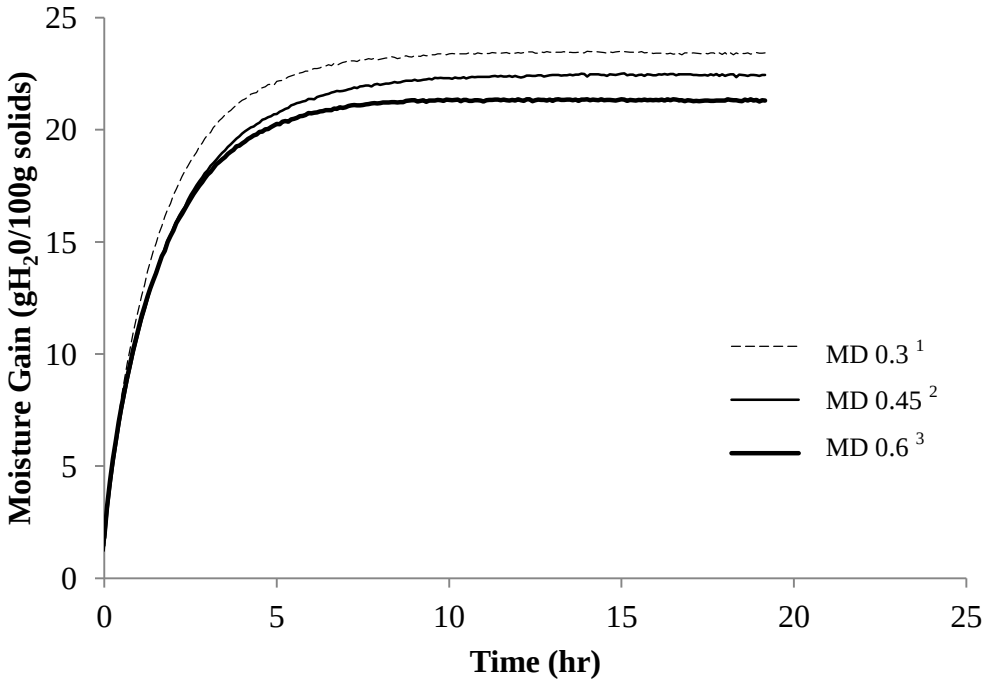

Figure 3.2 Hygroscopicity of blueberry powders dried at 95°C

[1] Blueberry powders with 0.3 kg MD/kg dry blueberry solids added

[2] Blueberry powders with 0.45 kg MD/kg dry blueberry solids added

[3] Blueberry powders with 0.6 kg MD/kg dry blueberry solids added

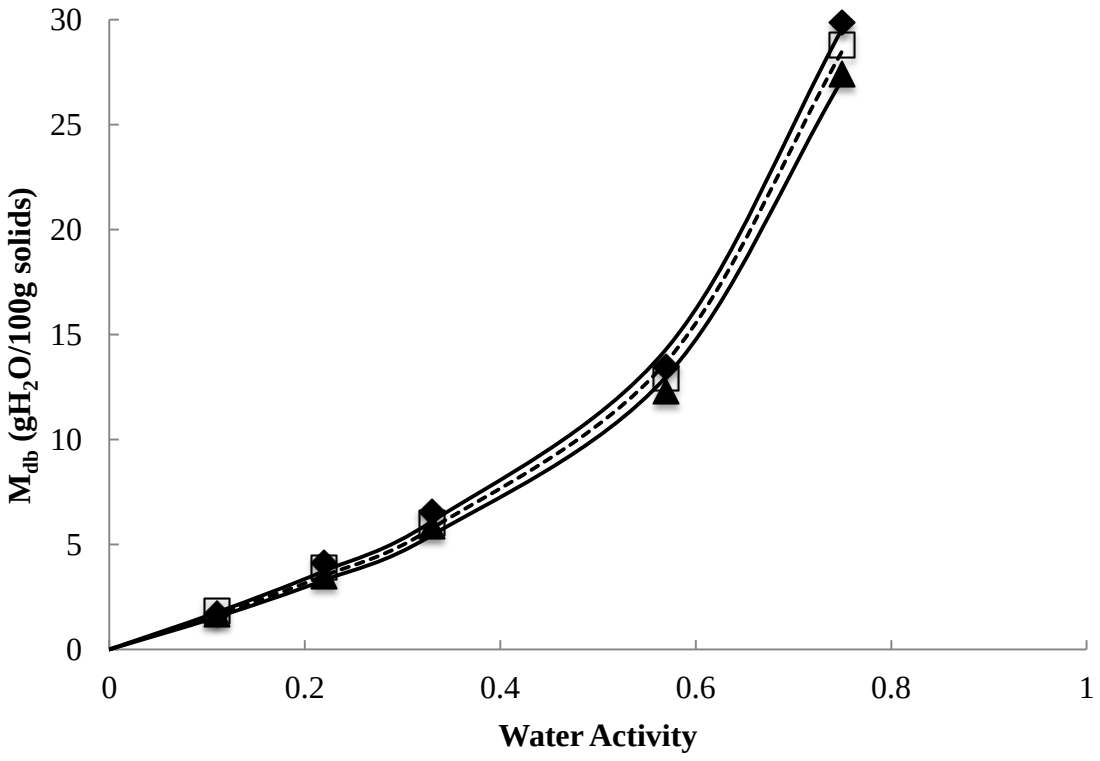

Figure 3.3 Moisture isotherm of blueberry powders dried at 95°C with different maltodextrin levels. ◆ - 0.3 kg MD/kg solids, □ - 0.45 kg MD/kg solids, ▲ - 0.6 kg MD/kg solids. Data fit by GAB equation.

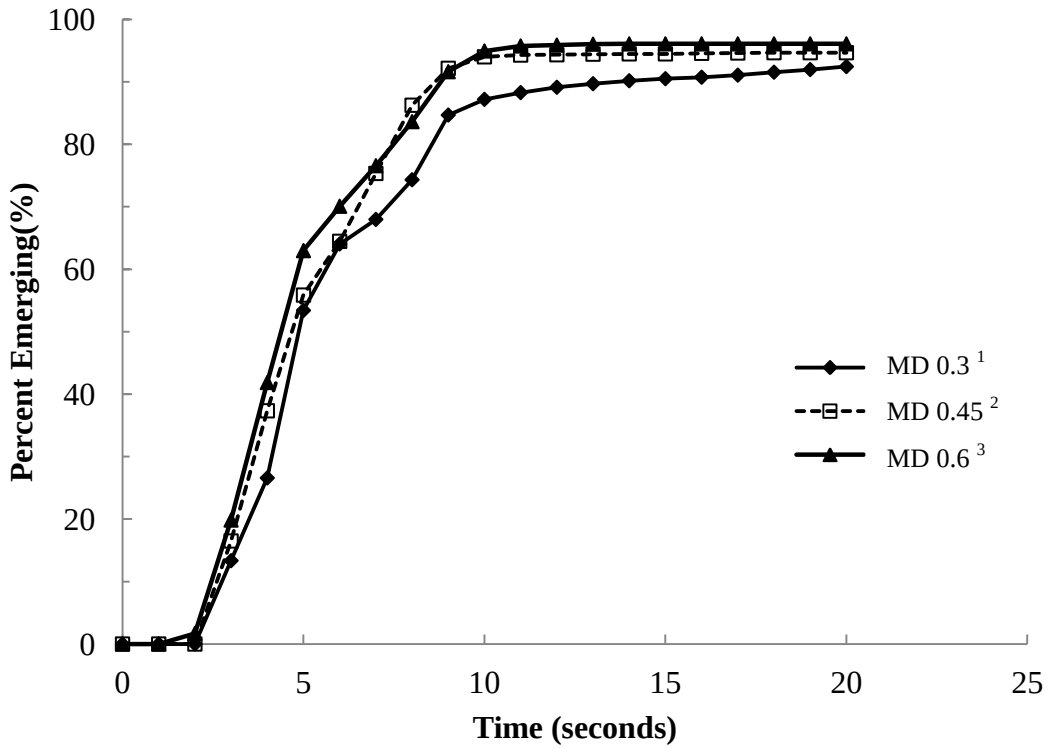

Figure 3.4 Flowability of blueberry powders dried at 95°C

[1] Blueberry powders with 0.3 kg MD/kg dry blueberry solids added

[2] Blueberry powders with 0.45 kg MD/kg dry blueberry solids added

[3] Blueberry powders with 0.6 kg MD/kg dry blueberry solids added

Table 3.1 Hygroscopicity model values of vacuum-belt dried blueberry powders as affected by temperature and maltodextrin (MD) level

	K[(2)]	**R^2**	**m_e[(3)] (gH_2O/100 g solids)**	**Hygroscopicity(%)[(4)]**
80°C MD[(1)] 0.3	0.4948[a]	0.9988	24.6[a]	19.85[a]
80°C MD 0.45	0.4848[a]	0.9969	22.7[abc]	18.71[bc]
80°C MD 0.6	0.4601[a]	0.9910	23[abc]	18.60[bc]
95°C MD 0.3	0.512[a]	0.996	23.5[ab]	18.99[ab]
95°C MD 0.45	0.495[a]	0.9984	22.4[bc]	18.33[bc]
95°C MD 0.6	0.4658[a]	0.9934	21.6[bc]	17.56[c]
110°C MD 0.3	0.4989[a]	0.9949	23.3[ab]	18.85[abc]
110°C MD 0.45	0.4627[a]	0.9969	22.3[bc]	18.23[bc]
110°C MD 0.6	0.4441[a]	0.9958	21.3[c]	17.55[c]

Mean values in the same column followed by same superscript are not significantly different ($p<0.05$)

(1) Level of maltodextrin added

(2) According to equation (3)

(3) Equilibrium moisture content

(4) According to equation (4)

Table 3.2 Parameters for moisture isotherm (GAB model) of vacuum-belt dried blueberry powders as affected by maltodextrin (MD) level

Parameters	**0.3 MD [1]**	**0.45 MD [2]**	**0.6 MD [3]**
Monolayer Value (H_2O/100 g solid)	8.80	8.55	8.18
c	1.7581	1.6733	1.6324
k	1.000	1.000	1.000
R^2	0.997	0.998	0.998

[1] Blueberry powders with 0.3 kg MD/kg dry blueberry solids added

[2] Blueberry powders with 0.45 kg MD/kg dry blueberry solids added

[3] Blueberry powders with 0.6 kg MD/kg dry blueberry solids added

Table 3.3 Flowability of vacuum-belt dried blueberry powders (Percentage mass emerging from cylinder at 10s, 30s) as affected by temperature and maltodextrin (MD) level

	% emerging at 10 s	% emerging at 30s
80°C 0.3MD [1]	87.67[a]	94.33[a]
80°C 0.45MD	93.05[a]	95.98[a]
80°C 0.6MD	92.85[a]	96.40[a]
95°C 0.3MD	84.68[a]	93.71[a]
95°C 0.45MD	92.16[a]	94.85[a]
95°C 0.6MD	91.62[a]	96.07[a]
110°C 0.3MD	86.25[a]	93.01[a]
110°C 0.45MD	89.66[a]	94.67[a]
110°C 0.6MD	91.93[a]	95.85[a]

Mean values in the same column followed by same superscript is not significantly different ($p<0.05$)

[1] Level of maltodextrin added

Table 3.4 Color parameters for vacuum-belt dried blueberry powders

	L*	C*	h°
80°C 0.3MD [1]	26.2^{c}	13.0^{de}	355.3^{cd}
80°C 0.45MD	25.7^{c,d}	13.6^{bc}	356.0^{b}
80°C 0.6MD	28.4^{a}	13.4^{cd}	354.2^{e}
95°C 0.3MD	25.3^{d}	13.6^{bc}	356.6^{a}
95°C 0.45MD	28.4^{a}	12.8^{e}	354.2^{e}
95°C 0.6MD	28.6^{a}	13.5^{bcd}	354.9^{d}
110°C 0.3MD	27.1^{b}	14.0^{ab}	355.7^{bc}
110°C 0.45MD	27.2^{b}	13.2^{cde}	355.5^{bc}
110°C 0.6MD	28.5^{a}	14.5^{a}	355.9^{b}

Mean values in the same column followed by same superscript are not significantly different ($p<0.05$). Frozen-thawed Brightwell: L* 18.3, c 2.4 and h 325.7.

[1] Level of maltodextrin added

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CHAPTER 4

VACUUM-BELT DRYING OF RABBITEYE BLUEBERRY POMACE: INFLUENCE OF DRYING CONDITIONS ON PHYSICAL PROPERTIES OF THE POWDER[1]

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Abstract

Blueberry pomace was vacuum-belt dried using three different temperatures (80°C, 100°C, 120°C) and two different drying methods (continuous and batch vacuum-belt drying), and analyzed for moisture content, water activity, moisture sorption isotherm, hygroscopicity, flowability, and color. Vacuum dried pomace was ground into blue powders with 1.7-2.7 g H_2O/100 g dry matter, and a_w 0.19-0.22. GAB modeling of the sorption isotherms showed monolayer moisture content of 0.047-0.051 gH_2O/g solids and Type II isotherm behavior. Hygroscopicity ranged from 18.97 to 21.09% at 7 days. Both drying temperature and method affected hygroscopicity, with batched processed powders having slightly higher values than continuous processed powders. All samples had good flowability, and no differences were found due to drying temperature or process. Drying temperature and method had a small but significant influence on chroma (C*) and hue (h) of the blueberry pomace powder. High drying temperatures resulted in lower chroma values due to anthocyanin loss and formation of brown compounds related to non-enzymatic browning. Batch process powders had slightly different hue (h=357.5-359.4°) as compared to continuously processed powders (h=359.1-0.3°), and higher chroma (C=14.5-15.4 as compared to C=12.6-13.2).

Introduction

Blueberries are an excellent source of antioxidants that provide health benefits to humans. Levels of phenolic compounds in blueberries are nearly three times higher than in other berries (Vega-Galvez and others 2009). Furthermore, blueberries are best recognized for their relatively high levels of anthocyanins and other flavonoids (Ehlenfeldt and Prior 2000). The anthocyanins are pigments that give the blue, violet, purple, and red color in berry crops, and they provide various health benefits (Lohachoompol and others 2004). The consumption of blueberries and the flavonoids they contain has shown promises for treating or lowering the risk of memory loss, cancer, heart disease, urinary disease, vision problems, and other problems related to aging (Wang and others 2011; Shi and others 2008; Kalt and others 2000).

Blueberries are included in the family Ericaceae, genus *Vaccinium* (Chakraborty and others 2010). They have spherical shape and range in size between 0.7 and 1.5 cm in diameter (Vega-Galvez and others 2009). The fruit size of rabbiteye (*Vaccinium ashei*) and highbush (*Vaccinium corymbosum*) fruits are bigger than that of lowbush cultivars (Kalt and McDonald 1996). North America produces the most blueberries in the world, contributing almost 90% of world production (Shi and others 2008). The consumption of blueberry has been increasing since 1990 (Vega-Galvez and others 2009), and developing new processing technologies is necessary to improve post-harvest shelf life of fruits. Current further processing technologies include canning, freezing and hot-air drying (Moraga and others 2006).

A significant amount of blueberry corps are used for juice processing (Lee and Wrolstad 2004). Pomace or presscake left after juice processing comprise approximatey 20% of the initial fruit weight (Khanal and others 2009). By-products after juice processing include seed, stems, and skins of fruits. Blueberry pomace is a great source of health promoting compounds such as

anthocyanins, polyphenolics (Lee and Wrolstad 2004) and procyanidins (Khanal and others 2009). Blueberry pomace can be used for new value-added products, such as natural colorants and nutraceuticals (Lee and Wrolstad 2004). Thus, finding ways to process pomace so that it can be easily used in products can help lower economic and environmental costs associated with disposal (Khanal and others 2010).

Drying is one of the most commonly used methods of fruit preservation. Dehydration can diminish physical and chemical changes in foods, and limit microbial growth by reducing water activity (Koc and others 2008). Most commonly, osmodehydration, sun drying or hot-air drying have been used to dry fruits. Osmotic drying involving high-concentration syrups may lead to leaching of desirable phytochemicals, high sugar content of the fruit, and not applicable to fruit pomace. Methods involving exposure to high temperature air can degrade anthocyanins (Lohachoompol and others 2004). An interesting alternative is vacuum belt drying (VBD), in which the material is introduced through an airlock onto a conveyor belt, and rides over several conduction heaters and supplemented by radiative heat. The product dries at relatively low temperatures and at relatively short drying times, particularly when compared to freeze drying. In addition, it has relatively low operating costs compared to spray and freeze-drying (Liu and others 2009). As a result, VBD is a promising method for producing high-quality food and pharmaceuticals (Liu and others 2011). Only a few studies have been reported on vacuum-belt dried foods. Wang and others (2007) produced VBD banana powder that had good retention of volatile aroma compounds. Liu and others (2011) produced VBD *Panax notginseng* extract that had better retention of bioactive compounds than spray or freeze-dried material. Pallas (2012) studied vacuum-belt drying of whole blueberries, and found that it provided excellent retention of anthocyanins and total phenolics.

A particularly promising use for blueberry pomace is as a source for dried powders. Dried fruit powders can add nutritional value to a variety of food products (Grabowski and others 2008). For example, fruit powders including blueberry, cranberry, concord grape, and raspberry were added to corn cereal extrusion process to increase functionality of it (Camire and others 2007). There have been several studies done on fruit pomace powders, such as muscadine pomace (Vashisth and others 2011), lime and cabbage pomace (Jongaroontaprangsee and others 2007), and apple pomace (Carson and others 1994).

The aim of this study was to evaluate blueberry pomace powder produced by vacuum belt drying. This involved determining optimal drying temperature and times to produce sufficiently low-moisture fruit powder. Key properties were evaluated including moisture sorption isotherm, hygroscopicity, flowability and color of the blueberry pomace powders.

Materials & Methods

Sample Preparation (Blueberry Pomace)

Frozen rabbiteye blueberry (*Vaccinium ashei 'Brightwell' and 'Tift Blue'*) varieties were collected from the University of Georgia Research and Demonstration Farm in Alma, GA. Frozen blueberries were crushed in a grinder (Model 98, Hobart corporation), and then placed in a 10 gallon jacketed steam kettle. The material was heated to 40°C, and samples were stirred until defrosted. To increase juice yield, 0.26 ml/kg mash of Pectinex Ultra SP-L (Novozymes, Bagsvaerd, Denmark) was added and the mixture held for 1.5 h. Juice was pressed from the mash using a Bucher HP 14 Laboratory Press (Bucher Industries AG, Murzlenstrasse 80, CH-8166 Niederweningen), giving ~70% yield of juice. The remaining pomace was collected and kept in a cooler at 4°C.

Vacuum-Belt Drying of Blueberry Pomace

A laboratory-scale vacuum belt dryer (Zwag, LKM-101, Zchokke Wartman Ltd. Bucher, Dottingen, Switzerland) was used to dry blueberry pomace. Pomace was introduced onto a 21 cm Teflon conveying belt inside the drying chamber. The belt allowed the product to pass over three conduction heater plates (Zones 1, 2 and 3) and one cooling plate (Zone 4). A radiation plate 22.9 cm wide was located above the three conduction heating plates (Figure 4.1). A DVT Aqua seal 80 CFM vacuum pump (Dekker vacuum technologies, INC., Michigan City, IN) was used to pull vacuum and the vacuum was held at 2.7-3.3 kPa (20~25 torr) during drying. An integrated touch screen panel and programmable logic controller were used to set up the temperature of the plates.

Three different temperatures (80°C, 100°C, and 120°C) and two types of drying method (Batch, Continuous) were used to dry samples. During batch processing, Zones 1-3 were set to the same temperature. Each drying cycle included two 350g batches of blueberry pomace. The pomace was evenly spread on the Teflon-coated fiberglass belt to a thickness of 0.5 cm. Samples were dried for either 40 min at 120°C, 70 min at 100°C or 100 min at 80°C. All the dried samples were targeted to be in the water activity (a_w) range of 0.15±0.02.

For continuous drying, the heating plates were set to the same temperature and blueberry pomace was fed into the vacuum-belt dryer using a solids feeder incorporating an airlock (Figure 4.1). A total of 350 g of pomace was dried at each temperature, with approximately 20 g of blueberry pomace introduced in increments. At 80°C, the belt speed was set to give a dwell time of 130 min on the heating plates. At 100°C, the dwell time was 100 min, while at 120°C the dwell time was 70 min. To maintain adequate vacuum, the pressure was held at 2.7-3.3 kPa (20~25 torr).

Powder Grinding

The dried product was cooled to room temperature, and then ground in a food processor (Model KFP600, Kitchen Aid, St.Joseph, MI) for one minute to form a fine powder. The ground samples were placed in a No. 70 (0.212 mm) sieve (Newark Wire Cloth Co., Newark, NJ) and shaken for 5 min. This process was triplicated. Final products were sealed in Pet/Al/PE laminate pouches and kept in a freezer. The weight of the powder obtained in a single experiment varied between 40 g and 60 g.

Moisture Content and Water Activity

The moisture content of blueberry pomace powders was determined by vacuum oven method 934.06 (AOAC 1995) with slight modification. Approximately 1.5-2g of blueberry pomace powder samples were added to 5 cm diameter aluminum pans (Fisher Scientific) and placed into vacuum oven (Model VWR 1430 MS, Optics Planet Inc., Northbrook, Il, USA). Samples were dried in the oven at 70°C and 40 kPa for 24 h or until samples reached constant mass. An Aqualab water activity meter CX-2 (Decagon Devices Inc, Pullman,WA) was used to determine water activity of samples at 22±2 °C. Values were measured in triplicate.

Moisture Sorption Isotherms

A series of plastic dessicators were used to hold samples at a relative humidity between 11% and 75%. The following saturated salt solutions were place in the bottom of each chamber (with corresponding a_w): LiCl (0.11), CH_3CO_2K, (0.22), $MgCl_2$ (0.33), $Mg(NO_3)_2$ (0.53), NaCl (0.75). Triplicate samples of ~1.5 g of blueberry powder were weighed into small plastic pans and placed on a platform in each chamber. A slight vacuum was pulled to seal the chamber. Samples were stored at 22°C for 4-5 weeks or until the samples reached constant weight. The

equilibrium moisture content was determined as described previously, in a vacuum oven at 70˚C and 58 KPa for 24 h (AOAC 1995).

Isotherms were plotted as the dry-basis moisture (M_{db}) content versus a_w. The data were fit to the Guggenheim-Anderson-de Boer (GAB) equation :

$$M_{db} = \frac{m_0 kca_w}{(1-ka_w)(1-ka_w+cka_w)} \quad (1)$$

Where m_o is the "monolayer" moisture content , and c and k are constants related to adsorption processes.

Hygroscopicity

Hygroscopocity was determined by the method of Cai and Corke (2000) with slight modifications. Approximately 1.5 g of each sample was weighed into a plastic pan and placed on a shelf inside a glass desiccator. The dessicator contained a saturated solution of KCl to establish a relative humidity of 86%. Samples were stored at 20˚C for 7 days. After one week, the hygroscopic moisture (%) was calculated as (Jaya and Das 2004):

$$\text{Hygroscopicity (\%)} = \frac{\frac{b}{a}+W_i}{1+\frac{b}{a}} \quad (2)$$

Where a (in g) is the weight of the initial sample, b (g) is the powder weight after one week, and W_i (%,wb) is the moisture content before the measurement. Samples were measured three times.

Flowability

Flowability was determined based on the method of Jaya and others (2006) and Liang and Langrish (2010). The flowability tester consisted of an aluminum cylinder 120 mm in diameter and 90 mm in width. At opposite ends of the curved surface, two slots were cut 4 mm wide and 70 mm in length. Sample was introduced through a removable side cap. The cylinder was held with an axle and caused to turn by small 24 V DC motor. To initiate the test, ~7 g of powder was added to the drum. The cylinder was connected to the motor drive and caused to

turn at 30 RPM. As sample emerged from the slots it was collected on a pan sitting top of an analytical balance. Sample weight was recorded every second by a laptop computer interfaced with the balance.

Color

A chromameter (Model CR-410, Konica Minolta, Ramsey, NJ) was used to measure sample color. A white standard tile was used to calibrate the chromameter. The colorimeter was located above a pertri dish filled with blueberry pomace powder. Values of lightness (L*), chroma (C*) and hue (h) were measured three times for each treatment group.

Statistical Analysis

Drying experiments were conducted at 3 different temperatures (80, 100 and 120°C) and two different drying processes (continuous versus batch). Results were analyzed using JMP®9.0.2 (SAS Institute Inc.). Two-way ANOVA was performed at confidence level of 95% ($p < 0.05$). Tukey's HSD was performed to investigate differences amongst the treatments.

Results & Discussion

Moisture Sorption Isotherms

The initial moisture content of dried blueberry pomace powders were in the range of 1.7-2.7 g H_2O/100 g dry matter. As the initial moisture content of the blueberry pomace powder was low, adsorption of water was the process of primary concern. As expected, the equilibrium moisture content of the blueberry pomace powder increased with increasing a_w. Overall, there were not large differences in the isotherm shapes or moisture depending on drying conditions. In general, powders produced at higher drying temperatures had slightly lower moisture content at a given a_w, for both batch and continuously processed powders. Powders produced by continuous drying had slightly lower moisture than those produced in batches. For example, at a_w=0.22

powders produced in batches at 120, 100, and 80°C had moisture contents of 3.539, 3.952, and 4.183 $gH_2O/100g$ solids. In comparison samples produced continuously at 80°C had a moisture content of 3.976 $gH_2O/100g$ solids.

Some have found that the processing method of juice powders can make some difference in the shape of the sorption isotherms, as for vacuum-dried, spray-dried, fluidized-bed dried, and freeze dried passion fruit pulp powder (Gabas and others 2007). Inchuen and others (2009), however, reported that no difference in sorption behavior was found between hot air and microwave dried red curry (Inchuen and others 2009). Debnath and others (2002) also showed that different drying methods (freeze drying, vacuum shelf drying, and through flow drying) did not significantly affect adsorption capacity of onions powders. We observed little difference in the isotherm shapes for blueberry pomace, likely as in our case there were not major differences in the type of drying mechanism occurring. The sorption isotherm for the pomace powder was most similar to the Type Ⅱ isotherm as described by Brunauer. The Type Ⅱ curve is sigmoidal in shape, and many foods follow this classification. The shape reflects that several mechanisms influence the relationship between moisture content and a_w, including colligative effects, capillary effects, and surface-water interactions in the food (Bell and Labuza 2000). It is worth noting that in our previous work on whole blueberry powders, the sorption isotherms were of a Type Ⅲ classification, a shape most often associated with crystalline materials. This is likely because the whole berries contained substantially more sucrose, fructose and other sugars, while much of the sugary juice and pulp were missing from the pomace.

Figure 4.2 shows sorption isotherms for blueberry pomace powder dried at different temperatures and by different drying methods. There was no marked difference among samples. It is known that processing methods have influence on shape of isotherms (Sundaram and

Durance 2008), however the effect of drying method on pomace powder was not evident. This means that the states of absorbed moisture of blueberry pomace powder during sorption process were not greatly influenced by the drying method.

The isotherm data were fit by the GAB model (Equation 1) and the curves also shown in Figure 4.2. Derived constants (m_o, c and k) for each fit are shown in Table 4.1. In general, the data were well-fit by this model and it has been found to successfully describe isotherms for many fruit products (Gabas and others 2000; Telis and others 2000; Lomauro and others 1985). The monolayer value (m_o) has been described as the amount of water strongly bound or immobilized on specific sites of the food surface, and has been considered as a target moisture content to limit deterioration in many dehydrated foods (Pavón-García and others 2011). The m_o values for the pomace powder ranged from 0.047-0.051 gH_2O/g solids. There was not a significant difference amongst m_o values of the differently treated samples, nor was drying method or drying temperature a significant factor.

The parameter c is related to adsorption processes (Inchuen and others 2009). C values decreased somewhat as the drying temperature increased, ranging from 7.24 at 80°C drying temperatures to 4.96 at 120°C. K value in GAB model describes adsorption of multiple layers of moisture (Benedetti-Damy and others 2010). In this study, k values were not affected by different drying type or temperature.

Hygroscopicity

The results for blueberry pomace powder hygroscopicity are presented in Table 4.2. ANOVA analysis showed that both drying temperature and method had significant effects on hygroscopicity. The least hygroscopic powders were obtained by continuous drying at 120˚C (18.97% after 7 days), and powders dried at 100˚C by batch drying were most hygroscopic

(20.9%). These results are consistent with the moisture isotherm data, which also showed that powders dried at the highest temperature had lower equilibrium moisture content, and continuously dried samples slightly less moisture than batch dried powder. While there were some small differences in hygroscopicity, it should be noted that the samples did not readily absorb moisture or shown signs of clumping. In addition, pomace powders were much less hygroscopic than powders made from whole blueberries (from chapter 3). While pomace powders reached 18.97-20.9% hygroscopicity after 7 days, whole berry powders (at 75% humidity) reached 17.5-19.8% hygroscopicity within 5-6 h.

Hygroscopicity is an essential powder property that measures the capacity of adsorption, absorption, and entrap of moisture (Rodríguez-Hernández and others 2005). In general, a powder that has the hygroscopicity value less than 10% is known as good quality, and non-hygroscopic powder (Pisecky 1985). In this study, the hygroscopicity ranged from18.97-21.09%, therefore it can be characterized as a hygroscopic powder.

Glass transition temperature (T_g) is the one of the main factor that affects hygroscopicity of the powder (Roos 1995). Foods are stable under the glass transition temperature ($T_{g)}$, however most foods become sticky and undergo structural collapse above T_g (Phanindrakumar and others 2005) . The difference of hygroscopicity among samples can be explained by different T_g temperature of each samples.

Flowability

To assess flowability, the mass of powder that had emerged from the rotating slotted cylinder was measured (Figure 4.3). The percent mass emerged after 10 and 30 s was also measured (Table 4.3). As seen in Figure 4.3, much of the powder had emerged within the first 8 s, and in general the powders showed good flowability. Further analysis of the percent of the

powder that had exited the cylinder at 10 and 30 s showed that continuously processed powders had 86.08-88.03% emergence at 10 s, while batch processed powders had 81.09-85.82% at 10 s. By 30 s, 94.82-95.25% of the continuously processed powder had emerged and 94.91-95.62%. Statistical analysis showed, however, that there were no significant differences in flowability linked to either processing method or drying temperature.

Powder flowability is an important and complex property controlled by particle size distribution and shape, surface interactions due to internal friction and cohesion, moisture content, and degree of caking (Fitzpatrick 2005). Interestingly, previous work on whole blueberry powder showed similar if not better flowability to pomace powder. For those powders, 87.0-94.3% of the powder had emerged within 10 s. However, those powders had 0.3-0.6 kg/kg of maltodextrins incorporated to decrease stickiness. While whole berry powders had good initial flowability, they were much more hygroscopic and lost their free-flow behavior within a few hours once removed from sealed packages. Blueberry pomace powders maintained reasonable flowability, however, even when stored without packaging for several days.

Color

Visually, all powders appeared dark blue-purple and had no obvious signs of browning. Lightness (L*) of the blueberry pomace powder varied from 25.2 to 25.7, chroma (c) from 12.6 to 15.4, and hue (h) from 357.5 to 359.4 (Table 4.4). In comparison, color values for frozen-thawed whole blueberries were L*=18.3, c = 2.4 and h = 325.7°. Thus, drying and grinding resulted in lighter, more saturated color, with a more red than purple-blue hue. Most of the red, blue and purple color of blueberries arises from the anthocyanins, contained primarily in the peel, and these are expected to be more concentrated in the dried powder. No differences in L* were observed due to drying temperature. Also, there was no significant difference between

batch drying and continuous drying. The L* values of blueberry are related to anthocyanis that give characteristic color to blueberries. Increase in L* means lighter color due to anthocyanin loss (Pallas 2011). In general, color of dired fruit and powder can change due to dehydration. Larrauri and others (1997) reported that L* of hot-air dried red grape pomace peels were significantly lower than freeze dried ones, however there was no significant difference amongst samples dried in hot-air at different temperatures (60˚C, 100˚C, 140˚C). Yang and Atallah (1985) reported that vacuum oven, freeze, forced air, and micro-convection dried blueberry samples had higher L* value than frozen blueberries.

Chroma (c) is related to color saturation (Voss 1992). Chroma of the powders ranged from 12.6 to 15.4 in this study. Powder dried at 80˚C had the highest c values followed by powders dried at 100˚C and 120˚C. There was also a significant difference in c values due to drying method. Samples dried by the batch process had higher chroma compared to those dried by the continuous processing. This may be due to increased drying time during continuous process. Lopez and others (2010) reported changes in blueberry color during hot-air drying due to increase of browning compounds related to non-enzymatic browning. Wang and others (2010) showed that considerable amount of anthocyanins degraded after exposure to high temperatures over 80˚C for 2 h. More likely, however, is that changes in chroma and hue due to higher temperatures may be related to changes in anthocyanin content. Blueberries dried by the same vacuum-belt drying system had losses in total anthocyanins depending on temperature and drying time (Pallas 2011). For example, they reported 13.9 mg C3G/g dry matter at 90°C and 9.9 C3G/g dry matter at 130°C, when berries were dried for 90 min. When dried for 105 min, the corresponding values were 12.2 and 5.7 C3G/g dry matter. Interestingly, there were no differences in total phenolics content due to drying temperature.

Hue is perhaps most immediately associated with visual color perception (Abers and Wrolstad 1979). In this study, the hue angle of the powders varied from 357.5˚ to 0.3˚ (that is 360.3°), with blueberries dried at highest temperature (120˚C) by batch process having the lowest hue angle, and blueberries dried at lowest temperature (80˚C) by continuous process having the greatest hue angle. The samples displayed more blueish-purple hue as the drying temperature increased, and continuous drying was showed more reddish-purple compared to batch dried samples. The differenes amongst samples, however, were relatively small. As with chroma, changes in the hue angle can be explained by degradation of anthocyanin, interaction between anthocyanin and other compounds, and changes in phenolic composition (Gonçalves and others 2007).

Conclusion

Dried blueberry pomace powder was produced by vacuum belt drying, by both batch and continuous methods. The powders had good apperance and color, with no signs of browning, although there was a slight diminishment of color saturation at higher drying temperatures. Moisture sorption behavior was not significantly affected by drying temperature or drying method. Higher drying temperatures and continuous drying resulted in slightly lower hygroscopicity of the powder. This study showed that continuous operation could produce pomace powders with properties similar to batch-processed material. Future studies will focus on incorporating pomace powder into value-added products and functional foods.

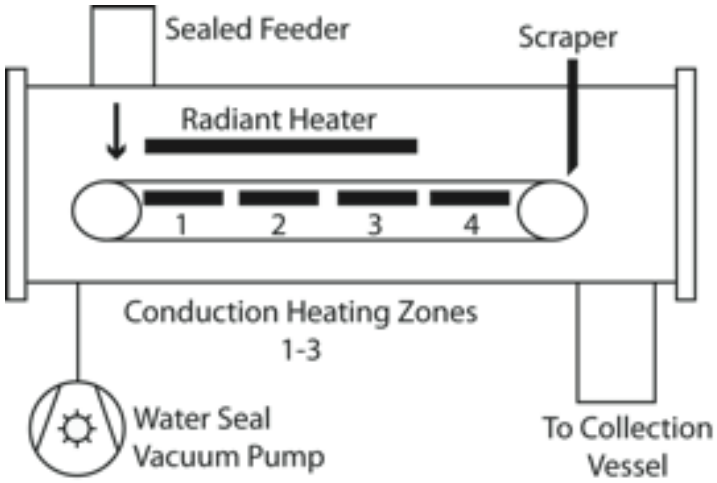

Figure 4.1 Vacuum-belt drier used to produce dried blueberry pomace powders

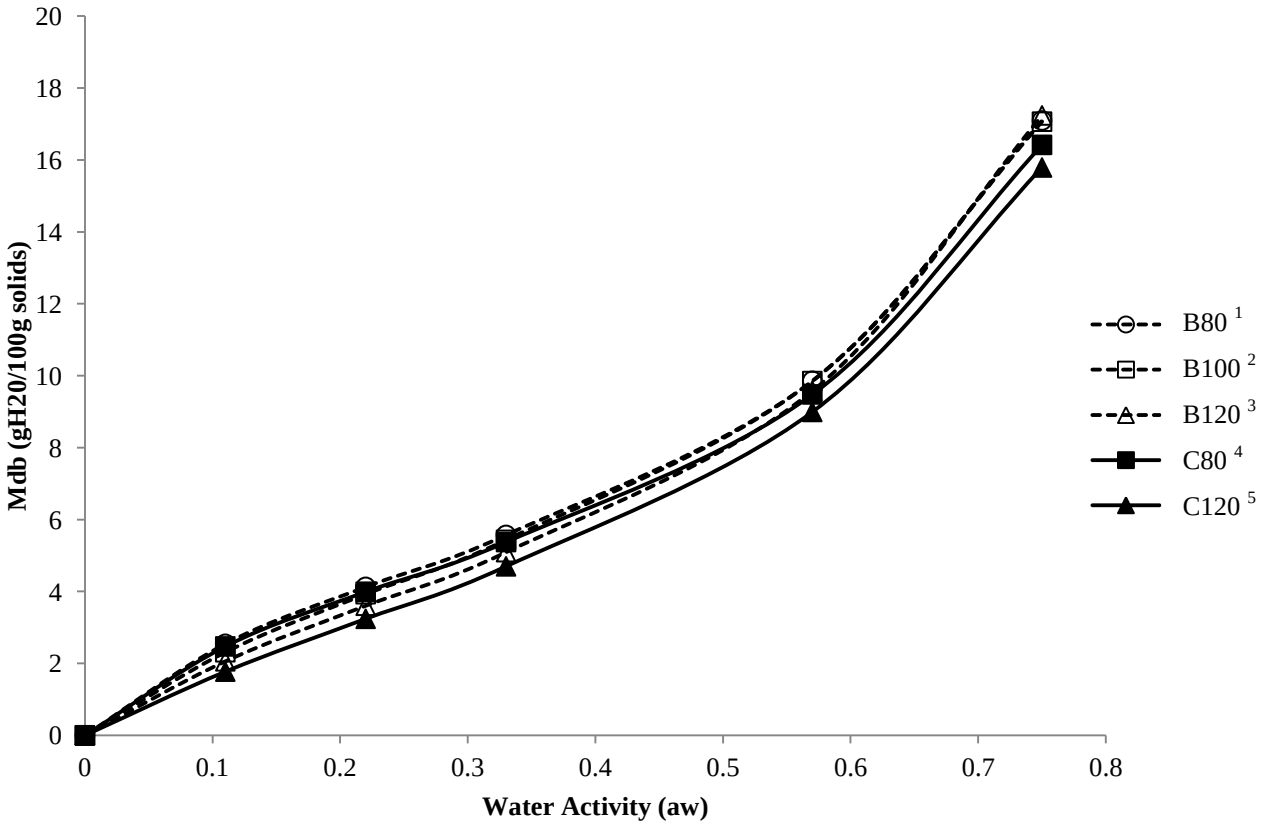

Figure 4.2 Moisture sorption isotherm of blueberry pomace powders prepared by vacuum-belt drying

([1] Batch processed at 80°C, [2] Batch processed at 100°C, [3] Batch processed at 120°C, [4] Continuously processed at 80°C, [5] Continuously processed at 120°C)

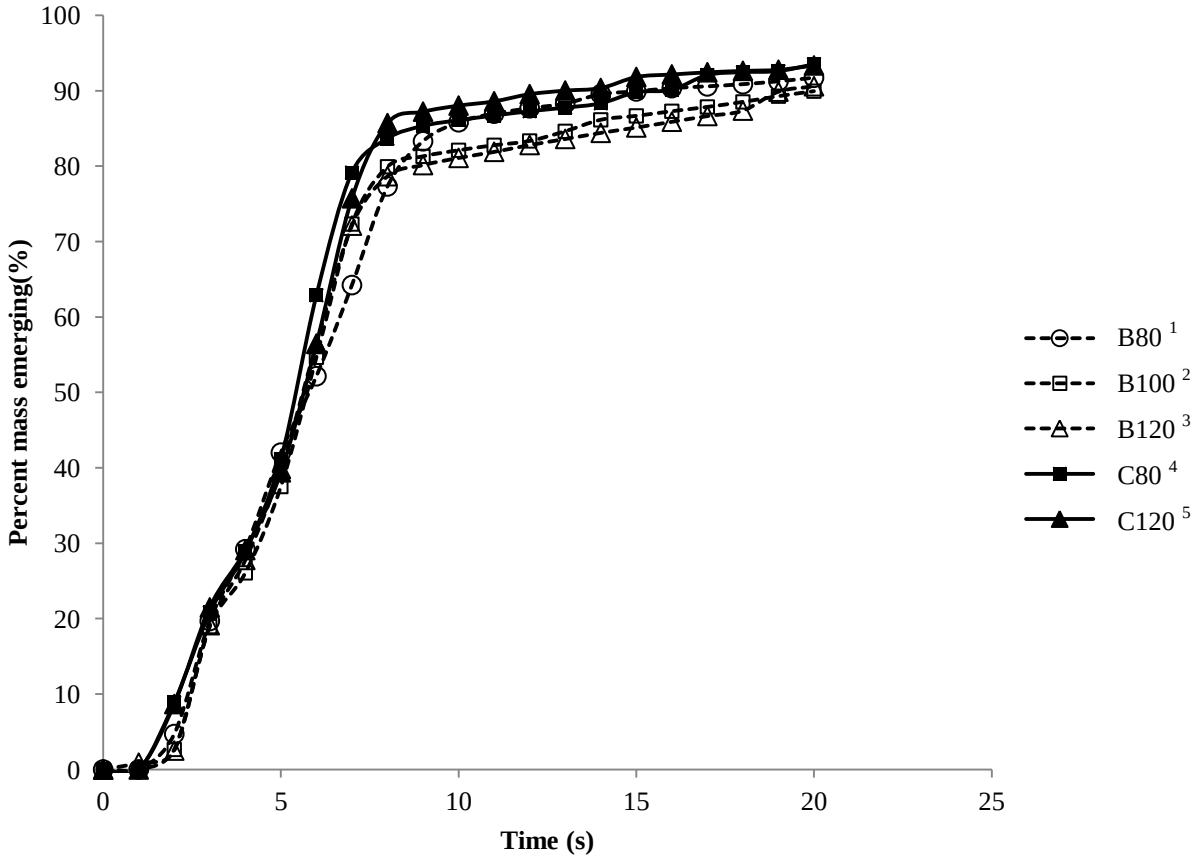

Figure 4.3 Flowablility of vacuum-belt dried blueberry pomace powders

([1] Batch processed at 80°C, [2] Batch processed at 100°C, [3] Batch processed at 120°C, [4] Continuously processed at 80°C, [5] Continuously processed at 120°C)

Table 4.1 Parameters for moisture isotherm (GAB model) of vacuum-belt dried blueberry pomace powders

Parameters	**B80** [1]	**B100** [2]	**B120** [3]	**C80** [4]	**C120** [5]
m_o (H_2O/g solid)	0.049	0.051	0.049	0.047	0.050
c	7.24	5.77	4.96	7.42	3.98
k	0.97	0.96	0.98	0.97	0.95
R^2	0.999	0.999	0.999	0.999	0.999

([1] Batch processed at 80°C, [2] Batch processed at 100°C, [3] Batch processed at 120°C, [4] Continuously processed at 80°C, [5] Continuously processed at 120°C)

Table 4.2 Hygroscopicity of vacuum-belt dried blueberry pomace powders

	Hygroscopicity(%)
B80 [1]	20.90[a]
B100 [2]	21.09[a]
B120 [3]	20.40[b]
C80 [4]	19.47[c]
C120 [5]	18.97[d]

Mean values in the same column followed by same superscript is not significantly different ($p<0.05$)

([1] Batch processed at 80°C, [2] Batch processed at 100°C, [3] Batch processed at 120°C, [4] Continuously processed at 80°C, [5] Continuously processed at 120°C)

Table 4.3 Flowability of vacuum-belt dried blueberry pomace powders (Perncetage mass emerging from cylinder at 10s, 30s)

	%emerging at 10s	**%emerging at 30s**
B80 [1]	85.82[a]	94.91[a]
B100 [2]	82.09[a]	94.73[a]
B120 [3]	81.09[a]	95.62[a]
C80 [4]	86.08[a]	94.82[a]
C120 [5]	88.03[a]	95.25[a]

Mean values in the same column followed by same superscript is not significantly different ($p<0.05$)

([1] Batch processed at 80°C, [2] Batch processed at 100°C, [3] Batch processed at 120°C, [4] Continuously processed at 80°C, [5] Continuously processed at 120°C)

Table 4.4 Color Parameters for vacuum-belt dried blueberry pomace powders

Color Parameters	**B80 [1]**	**B100 [2]**	**B120 [3]**	**C80 [4]**	**C120 [5]**
L*	25.7[a]	25.3[a]	25.3[a]	25.2[a]	25.4[a]
C*	15.4[a]	15.0[a,b]	14.5[b]	13.2[c]	12.6[d]
h°	359.4[a]	358.4[b]	357.5[b]	0.3[c]	359.1[d]

Mean values in the same row followed by same superscript is not significantly different ($p<0.05$). Frozen-thawed Brightwell: L* 18.3, c 2.4 and h 325.7.

([1] Batch processed at 80°C, [2] Batch processed at 100°C, [3] Batch processed at 120°C, [4] Continuously processed at 80°C, [5] Continuously processed at 120°C)

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CHAPTER 5

SUMMARY AND CONCLUSION

Vacuum-belt drying is a relatively novel technique that can produce high quality products. Vacuum-belt drying was used to produce whole blueberry and blueberry pomace powders. Different drying temperatures and times were used. Dried powders had good appearance and color. Three different levels of maltodextrin were added to blueberry slurry to produce whole berry powders with improved hygroscopicity and flowability. Addition of maltodextrin significantly affected hygroscopicity of the powders. Powders with higher levels of maltodextrin showed less hygroscopic behavior compared to powders with lower level of maltodextrins. Additionally, higher levels of MD also improved the flowability of the powders. Drying temperature was another significant factor influencing hygroscopicity of powders. Moisture sorption behavior was not significantly affected by drying temperatures or levels of matodextrin added. As whole blueberries contain high level of sugars, the drying operation was not easy due to product stickiness. However, adding maltodextrin improved problems related to stickiness and thermoplascity. Although stickiness was improved during drying, when whole berry powders were exposed to air, they readily absorbed moisture and color change was observed in short period of time. Storage conditions are very important to whole berry powders.

Blueberry pomace powder was produced by vacuum belt drying, by both batch and continuous methods. The powders showed good appearance and color, and no signs of browning were detected. However, slight diminishment of color saturation occurred at higher temperatures. Higher drying temperatures and continuous drying showed slightly lower

hygroscopicity of the powder. Drying temperature or drying method did not significantly influence moisture sorption behavior. This study proved that continuous drying could produce pomace powders with properties similar to batch-dried material.

It can be concluded that vacuum-belt drying is a suitable method for producing blueberry powders with good appearance. While this study focused on the physical properties of the blueberry powders, subsequent studies will investigate changes in nutrient and phytochemicals due to processing and storage conditions.