ABSTRACT

Three temperatures (80, 95, and 110°C) were employed to dry apple pomace using vacuum belt dryer. Color, hygroscopicity, flowability and particle size were compared between three ground powder samples. Moisture isotherms were developed, and glass transition temperatures were measured at different water activities. In addition, total phenolics, anthocyanin and fiber content were quantified, and the results were compared to those of fresh apple and raw apple pomace. There was no significant difference in terms of color, hygroscopicity, and total phenolics content between the pomace dried at 80 °C and 95°C, while the pomace dried at 110 °C had significant differences. When compared to fresh apple pomace, no significant differences were found between the samples and the apple pomace in terms of total phenolics and anthocyanin contents.

Key words: apple pomace, dietary fiber, powder, total phenolic content, vacuum belt drying
VACUUM BELT DRIED APPLE POMACE POWDER AS A VALUE-ADDED FOOD INGREDIENT

by

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VACUUM BELT DRIED APPLE POMACE POWDER AS A VALUE-ADDED FOOD INGREDIENT

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DEDICATION

For my family.
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CHAPTER 1

INTRODUCTION

In the process of making apple juice or apple cider, apple pomace becomes a main by-product with it consisting of 25-35% of the dry mass of apple (Gullón and others 2007). Large quantities of apple pomace are generated worldwide. Apple pomace has high moisture content (70%-75%), making it bulky and susceptible to microbial decomposition (Bhushan and others 2008). Once deteriorated, it generates a foul smell and can be hazardous to public sanitation.

General intake of dietary fiber reduces the risk for developing coronary heart disease, stroke, hypertension, diabetes, obesity, and certain gastrointestinal disorders. An increased consumption of dietary fiber improves serum lipid concentrations, lowers blood pressure, improves blood glucose control in diabetes, promote regularity, aids in weight loss, and improve immune function (Anderson and others 2009). The recommended dietary fiber intake is > 18 g per day for healthy adults in the UK and 25 – 30 g per day in the USA (Figuerola and others 2005). However, total fiber intake in adults in the United States appeared to be less than half the acceptable intake (Anderson and others 2009). Along with the sharp growth in all kinds of diseases, the fiber intake deficiency is the question which anxiously needs to be solved.

Apples have a well-balanced proportion of soluble and insoluble parts. In whole fresh apples, there is about 0.43 g of insoluble fiber and 0.36 g of soluble fiber in 100 g of fresh fruit (Gorinstein and others 2001). It also has better quality than other dietary fibers due to the presence of associated bioactive compounds, such as flavonoids, polyphenols and carotenes, water- and fat- holding capacities, low energy value and phytic acid content (Figuerola and others 2005; Bhushan and others 2008).
In the past, apple pomace was dried and used as animal feed (Singh and Narang 1992). It lacks digestible protein and is high in pectin and pentosans (Pirmohammadi and others 2006), so it is not suitable for direct use as animal feed. It can be also used as a source for pectin, xyloglucan, pigment, aroma compounds, press aid, natural colors, biofuel etc. (Bhushan and others 2008; Almosnino and others 1996; Thakur and others 1997; Lin and Demain 1991; Atri and Joshi 2005; Roberts and others 2004).

Vacuum belt drying is a new alternative method for dehydrating fruit and natural herb extract (Liu and others 2009), and this semi-continuous drying method is applied in the production of pure fruit juices (Maltini and others 1992). Dried products produced by vacuum and continuous belt are of high quality while the process is short, convenient and low in cost (Wang and others 2007a).

Apple pomace powders provide convenient products for the consumers that have extended shelf-life at ambient temperature or ingredients that are easily handled by food processors. To optimize powder processes and powder functionality while reducing costs during manufacturing is the ultimate goal of studying the properties of powders. (Fellows 2009).

The objective of this study is to evaluate the physical chemical properties and nutritional quality of apple pomace powders. Color, hygroscopicity, flowability and particle size were compared between the samples dried at different temperatures. Moisture isotherms were developed for these samples, and glass transition data studies were conducted with different samples at different water activities. Total phenolic content, anthocyanin content and fiber content were quantified, and the results were compared to those of fresh apple and raw apple pomace.
CHAPTER 2

REVIEW OF LITERATURE

Apple pomace

In the process of making apple juice or apple cider, apple pomace becomes a main by-product with it consisting of 25-35% of the dry mass of apple. 95% (by weight) of apple pomace is apple skin/flesh, 2%-4% seeds and 1% is the stem. (Bhushan and others 2008).

America is the second contributor to 46.1 million tons of apple production worldwide in 2006-2007. About 25-30% of the total production was processed into juice, cider, as well as frozen and dried processed products. About 1.4 million metric tons of apple juice was produced worldwide during 2004-2005 (Bhushan and others 2008). Large quantities of apple pomace are generated each year. According to the U.S. Department of Agriculture economic research service, the supply of apple cider and apple juice for 2008 in U.S. was 651.8 million gallons. Based on 75% juice extraction efficiency, about 1 billion kg of apple pomace are generated annually as the primary waste product to meet the needs for U.S. supply. Apple pomace has high moisture content (70%-75%), making it bulky and susceptible to microbial decomposition. It also has a high biodegradable organic load. The chemical oxygen demand can reach 300 g/kg. Transporting bulky apple pomace is very difficult. With more than $10 million dollars spent on the disposal of apple pomace, along with the foul smell due to biodegradation, direct dumping becomes a problem (Kaushal 2002; Bhushan and others 2008).
**Apple pomace nutrients**

“Dietary fibers are located in the plant cell wall compounds (non-starch polysaccharides), which are resistant to hydrolysis by digestive enzymes in humans. They are intrinsic and intact in plants and consist of mainly cellulose, hemicelluloses, pectic substances and lignins. Diets with inadequate dietary fiber content are generally associated with constipation, diverticulosis, cardiovascular diseases and cancer” (Bhushan and others 2008). General intake of dietary fiber reduces the risk for developing coronary heart disease, stroke, hypertension, diabetes, obesity, and certain gastrointestinal disorders. An increased consumption of dietary fiber improves serum lipid concentrations, lowers blood pressure, improves blood glucose control in diabetes, promotes regularity, aids in weight loss, and improves immune function (Anderson and others 2009). The recommended dietary fiber intake is 25 – 30 g per day for healthy adult in the USA (Figuerola and others 2005). However, the total fiber intake for adults in the United States is less than half the recommended intake (Anderson and others 2009).

In dietary fiber, the soluble fraction is associated with the reduction of LDL cholesterol by influencing the lipid metabolism, and the insoluble fraction is associated with water absorption and intestinal regulation. A 7:3 to 1:1 ratio of insoluble fiber and soluble fiber is considered a well-balanced fiber source. In this sense, apples have a well-balanced proportion of soluble and insoluble fraction with 0.43 g of insoluble fiber and 0.36 g of soluble fiber in 100 g of fresh fruit (Gorinstein and others 2001).

Due to the presence of associated bioactive compounds, such as flavonoids, polyphenols, carotenes, phytic acid, apple fibers have better quality compared with other dietary fibers (Figuerola and others 2005; Bhushan and others 2008). The nutritive values of apple pomace are
depended on the variety of apple, fruit maturity and also the juice extraction process
(Pirmohammadi and others 2006). Dried apple pomace has a 48-62% total carbohydrate content
with a high fermentable sugar ratio (Bhushan and others 2008).

A range of polyphenolics compounds have been isolated from apple pomace, such as
epicatechin, caffeic acid, phloridzin, phloretin-2’-xyloglucoside, 3-hydroxyphloridzin,
avicularin, reynoutrin, hyperin, sioquercitrin and quercitrin. With a 4.24g kg$^{-1}$ DM, quercetin
glycosides accounting for more than half of the total polyphenols content (7.24g kg$^{-1}$ DM),
apple pomace could be commercially exploited (Lu and Foo 1997). Several studies showed that
the polyphenols responsible for the antioxidant activity in apple are still present in the pomace.
Thus, apple pomace can provide an inexpensive source for food fortification. (Yeap Foo and Lu
1999; Schieber and others 2002; Lu and Foo 1997).

Polyphenols are important secondary plant metabolites which in fruits and vegetables are
both desirable and undesirable food qualities. People used to believe that these chemicals had
adverse effects in human metabolites because of the existence of tannins. However, recent
studies showed the antioxidative properties of these phenolics proved otherwise (Kaur and
Kapoor 2001). Antioxidant properties such as reactive oxygen species (ROS) and electrophile
scavenging, metal chelatin in plant phenolics have potential in health promotion.
Epidemiological studies support a relationship between the consumption of phenolic rich food
products and a low incidence of coronary heart disease, myocardial infarction (Kris-Etherton and
others 2002). Randhir, Lin et al. (2004) summarized that the phenolics in plants also have
antitumor, antiviral, antimicrobial, anti-inflammatory, and hypotensive properties.
Another component of apple pomace is the apple seed. Lu and Yeap Foo found that 80.9% of its volatile fraction is fatty acid and linoleic acid is the most dominant one, followed by 10.5% of palmitic, 5.6% of linolenic, 4.3% of stearic and 4.1% of oleic acids. The seed pomace was further extracted, and amygdalin and phloridzin were identified using NMR spectroscopy. Chlorogenic acid, p-coumarylquinic acid, 3-hydroxyphloridzin, phloretin-2’-xyloglucoside and quercetin glycoside were also identified using HPLC/DAD (Lu and Yeap Foo 1998; Lu and Yeap Foo 2000).

Amygdalin, which is a food source of cyanide, can also be found in apple seeds. Cyanide is well known for its high degree of lethality. It is estimated that the lethal dose in adults is 50-200 mg (Gracia and Shepherd 2004). According to Holzbecher et al. (1984), the HCN concentration in apple seeds was 0.61mg/g. Depending on the amount of usage, toxicity should be considered accordingly.

**Apple pomace utilization**

In the past, apple pomace was dried and used as animal feed. Singh and Narang (1992) concluded that feeding calves apple pomace with urea as the only supplement to a straw diet is feasible. Several investigators have substituted ensiled apple pomace in the diet of sheep (Toyokawa and others 1977). They observed that it was deficient in digestible protein and high in pectin and pentosans (Pirmohammadi and others 2006). According to Gullón and others (Gullón and others 2007), the ideal use for apple pomace has yet to be found.

Pectin manufacture from apple pomace is the only utilization currently carried out at an industrial level. Pectins consist of a backbone, in which ramified rhamnogalacturonan regions
interrupt the “smooth” α-D-(1-4)-galacturonan regions, highly substituted by neutral sugar-rich side chains (Schols and Voragen 1996; Innjerdingen and others 2005). For the past two centuries, pectins have been used as a gelling agent (Oakenfull and Scott 1984). Pectins have always been a natural constituent of human foods. Its use is allowed in all countries of the world. The joint FAO/WHO committee on food additives recommended pectin as a safe additive. There are no limits on acceptable daily intake unless dictated by good manufacturing practice.

Thankur, Singh et al. (1997) summarized the use of pectin in food industry such as gelling agent, thickener, texturizer, emulsifier, and stabilizer, fat or sugar replacer in low-calorie foods. Pectin accounts for approximately 10-15 % as a dry weight basis in apple pomace (Wang and others 2007b). Apple pomace needs to be first dried and stored for a period of time; otherwise it is hard to extract the pectin from it. Pomace is therefore usually brought from over a wide area from a number of drying plants (Thakur and others 1997).

In addition to pectin, apple pomace is also used for extraction of xyloglucan. The extraction of xyloglucan can use a commercial source apple pomace either with or without pectinase pretreatment. Enzymatic treatment of the apple pomace resulted in the production of lower molecular weight xyloglucan. The yield of xyloglucan is influenced by the alkali to apple pomace ratio, time of extraction, concentration of alkali, type of pomace and process temperature. The thickening agents and texture modifiers that are converted from xyloglucan have medicinal properties which can provide a new means for the development of high-value biomolecules (Bhushan and others 2008).

Aroma compounds can also be obtained during the bioconversion of polyunsaturated fatty acids by the endogenous enzyme system in apple pomace. Almosnino et al. (1996) reported
that aldehyde production by apple pomace enzymes can be oriented by controlling the pH conditions of the reaction medium to produce hexanal or 2, 4-decadienal. Their results also showed that simultaneous the double enzyme system, lipoxygenase and hydroperoxide lyase considerably improved aldehyde production.

Besides enzyme systems, apple pomace also contains micro-elements such as iron, zinc, copper, calcium and magnesium which can act as cofactors in many biosynthetic processes. They can be investigated for new applications either in biotransformations or bioconversions (Bhushan and others 2008).

Natural colors have been added to food for many years, but with increase in demand, synthetic colors dominated the market with the advantage of price and properties. A study showed ingestion of synthesis colors in children’s diet had an increase in both the duration and frequency of hyperactive behaviors (Rose 1978). The growing public concern over synthesis of colors due to the adverse effects make the food industry become more interested in the use of microbial technology to produce colors (Lin and Demain 1991; Atri and Joshi 2005). Apple pomace is a rich source of carbohydrates, dietary fibers, minerals, vitamin C and also has high moisture content. Thus, it has potential to support the growth of microorganisms. Atri and Joshi (2005) reported the effect of carbon and nitrogen sources on yield and carotenoids production by Micrococcus sp. Apple pomace medium showed the maximum yield of biomass and carotenoids.

Phloridzin is a phenolic compound particularly concentrated in apple pomace. Its oxidized product is a yellow pigment which could be an alternative to tartrazin for use as a food dye. The highly water-soluble yellow pigment can be used in both food and cosmetic industries.
However, significant research efforts would still be required to make this a viable process on an industrial scale (Nicolas and others 1994; Bhushan and others 2008; Guyot and others 2007).

Juice industry processors often use press aids in their extraction operation to maximize juice yield. Materials such as rice hulls provide added channels from which juice can drain while being pressed. Roberts et al. (2004) analyzed the effects of press aids on strawberry, raspberry, and blueberry juice quality and evaluated the effectiveness of dried apple pomace as an alternative press aid. Juice yields with apple pomace were similar to the yields from using rice hulls and paper. Triangle difference tests showed that berry juices pressed with apple pomace were preferred. Although off-flavors in the berry juice such as indole and 4-vinylguaical from rice hulls, (Z)-2-octenal and 2-nonenal from paper can be detected by gas chromatography-olfactometry.

Compounds that differ in physical and chemical properties in apple pomace dietary fiber display important roles in the organism. Dietary fiber has the ability to absorb many harmful substances by reducing their concentrations such as cholesterol or bind to heavy metals, and as well as to bind to mineral components. When used for the filtering of heavy metals, dietary fiber from pomace may noticeably upgrade the health-promoting properties of a food (Nawirska and Kwasniewska 2005).

Lantto, Plathin et al. (2006) reported that the gel formation during heating in battered pork meat was improved by the protein-modifying enzyme (other than proteinase) in apple pomace powder. A commercial microbial transglutaminase and the apple pomace powder acted the same way in pork meat homogenate, indicating the presence of similar enzymatic activities in both preparations.
In recent years, interest in using food processing wastes as a substrate for enzyme production has increased. Hang and Woodams (1995) concluded that there is potential for using apple pomace as a substrate for the production of fungal β-fructofuranosidase (EC 3.2.1.26) which catalyses the enzymatic hydrolysis of a fructofuranoside to an alcohol and D-fructose. It is commercially used in the conversion of sucrose to glucose and fructose and in the manufacture of chocolate-coated soft cream candies.

Apple pomace can be also used to produce L-lactic acid, oligomeric compounds, citric acid and bio-fuels (Hang and Woodams 1995; Gullón and others 2007). The utilization of apple pomace can not only solve the pollution issues caused by foul smells, but also increase efficiency in the apple industry by using the food processing residues to generate value-added ingredients. More efforts are needed to be made to find more possibilities and to scale up more current utilizations to industrial levels.

**Apple pomace dehydration**

As mentioned before, apple pomace is highly perishable due to its high moisture content, and most proposed uses require dehydration as a pretreatment. In addition, dehydration is an approach to extend shelf-life and minimize handling costs for further processing. However, drying causes physical, chemical, and biological changes, especially to natural nutrients and bioactive compounds that are unstable to heat (Chang and others 2006; Ratti 2001). On the other hand, recent studies have showed that thermally processed foods, especially fruits and vegetables, have a higher biological activity due to their various chemical changes during heat
treatment when compared to food at its freshest state (Henriquez and others 2010; Chang and others 2006; Mrkč and others 2006).

Drying requires substantial thermal energy, and this is often obtained from the combustion of fossil fuels. Thus drying can have both an economic and environmental impact. Thermal dehydration industries contribute 12-25% of energy consumption in industrial nations (Cil and Topuz 2010). Two methods of water removal are usually employed to produce dry products, separation by crystallization and subsequent sublimation (freeze drying) and water removal by vaporization (various drying techniques) (Bhandari 2005). The selection of a particular method for apple pomace drying depends on its energy cost, change in nutritional profile and purpose of use. In one of the earliest processes, sun energy was used for drying apple pomace in the open to reduce bulk. This method results in rather dark apple pomace (because of enzymatic or oxidative browning) and may render it unfit as an additive, especially for human food fortification (Bhushan and others 2008).

Air-drying, in particular, has been used for many years to preserve food by exposing the food to a continuous stream of hot air to evaporate moisture. The phenomena underlying this process is a complex problem involving simultaneous mass and energy transport in a hygroscopic, shrinking system. Air-drying results in dehydrated products with much extended shelf-life, however, the quality of a conventionally dried product is usually drastically reduced from that of the original foodstuff (Ratti 2001).

Freeze drying, a process developed in the second half of the 20th century, minimizes damage during drying of biological products, is as a good dehydration method for foods, pharmaceuticals, and other products. Before drying, the wet solid material has to be frozen;
during the primary drying stage, sublimation of frozen free water occurs. Lastly, the bound water is eliminated at the secondary drying stage by desorption (Reyes and others 2010).

Freeze-drying occurs under high vacuum which allows sublimation of water from a frozen product. The final products obtained from freeze drying have excellent quality due to the absence of liquid water and low temperatures required for the process that most deterioration and microbiological reactions are stopped. The solid state of water in the products during freeze-drying protects the primary structure and the shape with minimal volume changes. Despite the advantages mentioned above, freeze-drying has always been recognized as the most expensive process for dehydrating a product (Ratti 2001).

Very low temperature coils are used in the conventional freeze-dryers to condense water vapor, which adds substantially to the cost. Instead of refrigeration coils, the modified process uses a desiccant to remove the water vapor. The adsorbent replaces the condenser, and reduces by 50% as compared to traditional freeze-drying. Despite the many advantages as compared to conventional freeze-drying, the quality of adsorption freeze-dried foods is not as good as the conventional freeze-drying (Bell and Mellor 1990; Ratti 2001).

Freeze-drying is not widely used in the food industry due to its high operation cost. In order to reduce cost of freeze-drying, new improvements such as adsorption, fluidization, and microwaves have been researched in the last decade, vacuum freeze-drying is only used on an industrial scale to dry some coffees, spices, meats, food ingredients and other high-value foods. However, this process could be considered a valuable alternative to preserve other foods with increasing concerns about food quality (Ratti 2001).
Spray-drying is a unit operation by which the atomized liquid product passes through hot gas currents to instantaneously become a powder. Air or inert gas such as nitrogen is usually used for gas. A solution, an emulsion or a suspension can be used for the sprayer. Either a very fine powder or large particle size granules can be produced from spray dryer, depending on the starting feed material and operating conditions. (Gharsallaoui and others 2007).

Fluidized bed drying is considered as one of the most successful dehydration techniques. The main distinctive advantages of fluidized bed drying are high heat and mass transfer, good temperature control, uniform temperature and high drying capacity. Although the drying rate is dependent on the drying temperature, during the fluidized bed drying process, the drying rate should not be too high because high temperature can cause the particles to spoil. Fluidized beds are commonly used commercially for drying materials such as granular materials, cereals, polymers, chemicals, pharmaceuticals, fertilizers, crystalline products and minerals due to their rapid drying characteristics and drying cost. (Cil and Topuz 2010).

In general, a complete microwave drying process consists of three drying periods. In the first period which is a heating-up process, while the temperature of the product increases with time, microwave energy is converted into thermal energy within moist materials. Once the moisture vapor pressure in food is above that of the in environment the material starts to lose moisture. The second period has a rapid drying rate when thermal energy converted from microwave energy is used for the vaporization of moisture. In porous food structures, dehydration rate at different locations in the food depend largely upon the local rates of thermal energy conversion from microwave. The third period is associated with a reduced drying rate; the energy needed for dehydration is less than the thermal energy converted from microwaves.
With this local temperatures may then rise above the boiling temperature of water. (Zhang and others 2006).

Vacuum drying has some distinctive characteristics compared with atmospheric drying. The high drying rate, relatively low drying temperature and oxygen deficient environment can help improve the quality and nutritive value of dried products, especially for those food materials with high nutrient values but are prone to be oxidized (Wu and others 2007). Vacuum belt drying is a semi-continuous drying method for dehydrating the fruit. It has been applied in the production of pure fruit juices (Maltini and others 1992). Shortened drying time with high quality drying products is an advantage of vacuum belt dryers. Wang, Li et al. (2007a) did research on banana powder dehydrated by vacuum belt drying, freeze-drying and air-drying. They found that the major volatiles of the three products were the same, but some components existed only in freeze dried product and some only in vacuum belt dried product. They concluded that the best aroma value was achieved in products dried by freeze-drying, followed by vacuum belt drying and finally air drying. Liu, Qiu et al. (2008) also compared vacuum belt drying of Panax notoginseng extract with vacuum freeze drying and spray drying. Compared with other two drying methods vacuum belt drying had a higher recovery of active ingredients, less moisture content of dried product and better overall yield.

**Apple pomace powder properties and uses**

As mentioned, apple pomace has substantial phenolic compounds, dietary fiber, protein, minerals and vitamins. When apple pomace is dried and ground into powder, it reduces off-odors as well as transportation and storage costs. It also provides convenient products that have
extended shelf-life at ambient temperatures or ingredients that are more easily handled by food processors (Fellows 2009).

Food powder properties can be classified as physical or chemical. Physical properties include the particle’s shape, size, distribution, density and porosity, surface characteristics, hardness and diameter. The chemical properties of a food material are related to the food’s composition and its interaction with other substances like solvents or components within the food structure. Physical properties provide a way to quantify the output particulate product in certain dehydration, grinding, or size enlargement processes, and to compare the effects of composition, structural conformation, material origin, types of processing equipments, or handling. Chemical reactions in food powders mainly occur due to the presence of free water within the particle or bulk. However, the most relevant chemistry-related properties in food powders are linked the powder solubility during processing and consumption, and its caking capacity during storage (Barbosa-Canovas and Juliano 2005). In addition, to investigate the process of dehydration and the stability of apple pomace powder in storage, a knowledge of moisture sorption isotherms is required (Aviara and others 2006). The degree of fit to the experimental data and the simplicity of the model was a widely accepted criteria used to select the most appropriate sorption model (Simal and others 2007).

Color is important to many foods, and when combined together with flavor and texture, it plays an important role in food acceptability. In addition, color may provide an indication of chemical changes in a food, such as browning and caramelization (deMan 1999). The color of apple pomace can be used as an indication of chemical changes during drying and provide information for incorporating apple pomace powder in other products.
The flow behavior of powders is very significant throughout powder production, storage transport and processing. For instance, the manufactured powder must flow out of the dryer after the drying process; likewise particulate material must flow in and out of comminution equipment. There are four factors that affect the flowability of food powders: particle size, shape and distribution, surface interactions, moisture content, and storage conditions. The particle size and bulk density of a powder are important considerations in many applications as well (Fitzpatrick 2004). High bulk densities containing a range of both small and large particles are required for many food powders to be used as ingredients. The relative movement of a bulk of particles among neighboring particles or along the container wall surface is the flow of the powder. Powders with both small particles and large particles have a better flow and a longer storage life with the reduced amount of air in the powders (Fellows 2009; Fitzpatrick and others 2007).

Apple pomace powders were used in pie filling and oatmeal cookies. Two different amounts of pomace powders in pie filling or three differing amounts in oatmeal cookies did not affect color, cookie size or sensory scores. Both food products were rated as liked moderately (Carson and others 1994). Masoodi et al. (1998) also used apple pomace powder as a source of dietary fiber in wheat bread, and the sensory evaluation of the product indicated that breads containing up to 5% pomace powder were acceptable.

One problem for apple pomace powder usage is the toxicity of apple seeds. Apple seeds are one of the most well-known food sources of cyanide. The concentration of cyanide in apple is around 690-790 mg HCN equivalents/kg apple (Rezaul Haque and Howard Bradbury 2002). It
is estimated that the lethal dose in adults is 50-200 mg (Gracia and Shepherd 2004). Due to the unpredictability of usage for apple pomace powder, apple seeds should be taken out prior to use.

With more researches on drying methods, physical and chemical properties of apple pomace powder, more applications will be discovered and developed.
CHAPTER 3

PHYSICAL PROPERTIES OF APPLE POMACE POWDERS PRODUCED BY VACUUM BELT DRYING

Abstract

Apple pomace dried by vacuum belt dryer at 80 °C, 95 °C and 110 °C was ground into powders. Physical properties of the three samples were evaluated. There was no significant difference between the samples dried at 80 °C and 95 °C in terms of color and hygroscopicity. When compared between those two and the sample dried at 110 °C, significant differences were found. Moisture isotherms were developed for each sample. Flowability and particle size were also measured by standard flowability tester and image analyzer respectively. The glass transition temperature was measured by DSC at different water activities.

Key words: apple pomace, powder, vacuum belt dryer, color, hygroscopicity, flowability, moisture isotherm, GAB model, particle size, glass transition
Introduction

In the process of making apple juice or apple cider, apple pomace becomes a main by-product with it consisting of 25-35% of the dry mass of apple (Gullón and others 2007). Large quantities of apple pomace are generated worldwide. According to Đilas (2009), the estimated annual waste of apple was 3.0-4.2 million metric tons. Uses for animal feed or as a food ingredient have been investigated, but serving as a starting source for pectin manufacture is the only utilization currently carried out at an industrial level. Gullón and others (2007) suggested that the ideal use for apple pomace has yet to be found.

Apples have a well-balanced proportion of soluble and insoluble fiber fractions (Gorinstein and others 2001), and apple pomace has better quality than other dietary fiber sources due to the presence of associated bioactive compounds, such as flavonoids, polyphenols and carotenes, as well as good water- and fat- holding capacities, low energy value and phytic acid content.

Due to its moisture content, apple pomace must be dried to extend the shelf-life. The basic objective is to remove water to a level where microbial spoilage is reduced and shelf-stable and less perishable product is ensured (Kwok and others 2004). The selection of a particular method for apple pomace drying depends upon its energy cost, change in nutritional profile and intended purpose. Studies have shown that a drying temperature, which contributed to discoloration during the process, have effects on the chromatic parameters, non-enzymatic browning compounds and extractable color (Vega-Gálvez and others 2009). Interestingly, the radical scavenging activity was higher at high temperatures rather than low temperatures. Compared with conventional atmospheric drying, vacuum drying has some distinct advantages
such as higher drying rate, lower drying temperatures and an oxygen-deficient processing environment. These characteristics may help to improve the quality and nutritive value of the dried products (Wu and others 2007).

Vacuum-belt drying is a semi-continuous process in which material can be fed into the dryer through an airlock, without needing to disrupt the vacuum as more material enters the system. The material is conveyed over conduction heaters, and heating may be supplemented by radiation. Relatively short drying times can be attained, and the low oxygen environment helps products that are prone to browning or oxidation. The shortened drying time may reduce energy consumption as well. Vacuum-belt dried, freeze dried and air dried banana powder was compared by Wang, Li et al. (2007). They found that freeze dried and vacuum belt dried products have distinct volatile compounds, and that the best aroma was achieved in freeze dried and followed by vacuum belt dried products. Freeze drying is more expensive than vacuum drying, however, and it is not usually economical for fruit products (Drouzas and others 1999). Liu, Qiu et al. (2008) also compared vacuum belt drying of drying Panax notoginseng extract with freeze drying and spray drying. Vacuum belt drying had higher recovery of active ingredients, lower moisture content of dried product and better overall yield.

A convenient form for apple pomace and other fruit materials is as a powder. The powder physical properties are quite important as they affect the powder behavior during storage, handling and processing. One important aspect is the relationship between moisture content and water activity, as shown by moisture isotherms, and how the material absorbs moisture when exposed to humid environments (Aviara and others 2006). Water activity and moisture content are also the major factors that influence stability of the product, while hygroscopicity of the
powder determines how it performs in many applications (Fitzpatrick 2004; Liang and Langrish 2010; Bhandari 2005). The distribution of particle sizes in the powder is also critical as it can figure into the taste, color, texture, smell, rehydratability, and smell of the final product (Hagan 2005). Flowability is another crucial property that influences processing, transportation, handling and storage. While the physical properties of powders are somewhat independent, modification of particle size distribution or moisture content can result in a simultaneous change in bulk density, flowability, and appearance. The glass transition ($T_g$) of dried powders has also been found to be a useful marker, as it determines the temperature below which the product exists as a true amorphous solid. Maintaining temperature below $T_g$ is useful for preventing chemical deterioration as well as stickiness or caking (Bhandari 2005).

The objective of this study was to produce powders from apple pomace through vacuum drying, in a system that would allow continuous production, and result in powders with good color, nutrition, and handling properties. The influence of temperature was studied as it determines the overall drying time of product. Several physical properties of the pomace powders were determined including color, hygroscopicity, flowability and particle size. Moisture isotherms were also developed for each sample. Glass transition temperatures were also determined by differential scanning calorimetry (DSC) as they relate to stability and handling properties of the powder.
Materials and methods

Chemicals and reagents

Citric acid, ascorbic acid, sodium hexametaphosphate (NaHMP), lithium chloride, potassium acetate, magnesium chloride, sodium bromide, sodium chloride and potassium chloride were purchased from J.T. Baker (Phillipsburg, NJ).

Determination of pretreatment method

The browning inhibitor solutions were prepared from the different combination of citric acid ascorbic acid and sodium hexametaphosphate with tap water. The formulations compared in this study are listed in Table 3.1. To minimize browning, the fresh apple pomace was immersed in solutions for 3 min immediately after pressing. The dipped apple pomace was placed in a colander to be de-watered and then transferred into polyethylene bags. The treated samples were stored at 4 °C for up to 6 weeks. Because of the irregular surface and small size of discolored areas relative to the aperture size of the instrumentation, the samples were evaluated visually for the onset of browning at intervals during storage (Pilizota and Sapers 2004).

Samples

Apple pomace was obtained directly from the belt press at Mercier Orchards (Blue Ridge, GA) after pressing the juice from a mix of Rome Beauty, Fuji, Red Delicious and Golden Delicious apples. After chopping, the apples were mixed with rice hulls (Riceland®, Riceland Foods, Stuttgart, AR, 1% by weight of juice slurries) as a pressing aid to maximize juice yield. The pomace was immediately immersed into a browning inhibitor containing 2% ascorbic acid, 2% citric acid and 1% sodium hexametaphosphate (Na HMP) in water. The treated apple pomace was frozen at -20 °C in polyethylene buckets until needed.
Vacuum belt drying process and powder making

A laboratory scale vacuum belt dryer (Zwag, CH-5312 Zchokke Wartman Ltd. Bucher, Dottingen, Switzerland) was used to dry the apple pomace samples. Product can be advanced in the dryer by a Teflon coated fiberglass conveyor belt which passes over three conduction heating zones. A fourth zone is used for cooling prior to product being scraped from the belt into a collection vessel. A radiation plate is situated ~8 mm above the sample on the belt, and spans the length of the 3 conduction heaters. For the trials, the three heating zones were set at the same temperature as the radiation plate, either 80 °C, 95 °C, or 110 °C. The chamber vacuum pressure was 2.5kPa- 3.1kPa (absolute pressure). The apple pomace was thawed at 4 °C over night prior to drying, and rinsed with distilled water before use. The thickness of apple pomace on the vacuum dryer belt was maintained at 5mm. Dried apple pomace was removed from the dryer after the water activity reached 0.2 ± 0.04.

The dried material was then processed to make powders. A food processor (Kitchen Aid KFP350WH Food Processor, St. Joseph, MI) was used for 40 seconds to form chopped pieces of the material. The apple seeds were then separated by a sieve (American standard No.6, 3.35mm opening). A Model 111338 grinder (General Electric, Fairfield, CT) was used for 1 min to grind the samples into powders.

Moisture content and water activity

Moisture content in the dried apple pomace powders was determined using Association of Official Analytical Chemists (AOAC) Method 934.06 (AOAC 1990) with some modification. The moisture content \( X \) was expressed as the ratio of the amount of water in the food to the amount of dry solids:
$X = \frac{W_t - W_s}{W_s}$  \hspace{1cm} (1)

where $W_t$ is the total weight of the wet material and $W_s$ is the weight of the dry solids. Powder samples were weighed in a pre-dried aluminum pan and placed in a vacuum oven (model VWR 1430 MS, Optics Planet Inc., Northbrook, Il) set at 70 °C and 58 KPa for 24 h. Measurements of the sample weights were taken using an Ohaus Analytical Plus AP210 (Parsippany, NJ) analytical balance. The water activity ($a_w$) of samples was determined using an Aqualab water activity meter (Decagon Devices, WA).

**Color measurement of the apple pomace powders**

The Lightness ($L^*$), Chroma ($C^*$) and Hue ($H^*$) values were measured in triplicate for each sample using a Model CR-410 Chroma meter (Minolta. Co., Ltd., Osaka, Japan). The instrument was calibrated against a white calibration plate. The $L^*$ value ranges from 0 (no lightness) to 100 (maximum lightness). The $C^*$ value ranges from 0 (a neutral grey, black or white) to 100 for very high Chroma or “color purity”. Hue angle represents the perceived color where $0^\circ$ corresponds to red, $90^\circ$ for yellow, $180^\circ$ for green and $270^\circ$ for blue. Apple pomace powders were filled into aluminum pans that were larger than the aperture of the colorimeter, and the glass face of the instrument brought into direct contact with the powder.

**Hygroscopicity**

To measure the tendency for the powders to pick up moisture from the environment, dried powders were subject to a specified relative humidity environment. About 2 g of each powder was spread on a 90 mm diameter Petri plate, and placed on a shelf in a glass desiccator. The desiccator containing a saturated solution of KCl to establish a relative humidity of 86%.
After sealing the chambers, powders were held at 86% humidity at 20 °C for up to a week. The hygroscopicity was calculated by (Jaya and Das 2004):

\[
\text{Hygroscopicity} \; (\%) = \frac{b + W_i}{a + \frac{b}{a}}
\]

Where \( a \) (g) was the initial weight of the sample, \( W_i \) the initial moisture content and \( b \) (g) was the powder weight after one week at 86% relative humidity. All the measurements were done in triplicate.

**Particle size**

An Camsizer image analyzer (Horiba Scientific, Inc. CA, USA) equipped with two digital cameras was used to measure average particle size and size distribution using a standard procedure (ASABE 2008). Apple pomace powder (20 g) was loaded onto the hopper of the instrument, and the sample introduced into the unit through a vibrating feeder. The size and shape of the powders were recorded and analyzed using the Camsizer software. The equations used for particle size analysis were taken from Standard ANSI/ASAE S319.4 (ASABE 2008). The geometric mean diameter \( (d_{gw}) \), geometric mean diameter standard deviation \( (S_{gw}) \), sphericity (SHPT) and the specific surface area \( (S_v) \) were analyzed. The geometric mean diameter was taken as the size at 50% cumulative distribution. The geometric mean diameter standard deviation is the measurement of the variation in the particle sizes of apple pomace powders and was analyzed with Equation 3. The SHPT was analyzed using Equation 4. Roundness values ranged from 0 to 1, with 1 representing a perfect sphere. The surface area \( (S_v) \) was calculated with Equation 5. The relevant equations include:
where $d_{84}$ and $d_{16}$ are particle diameters obtained from the cumulative distribution data at 84% and 16% respectively, $a$ is the measured area covered by a particle projection, $p$ is the measured circumference of a particle projection, $A$ is surface of all particles ($\text{mm}^2$), $V$ is volume of a particle distribution (Phanphanich and Mani 2011).

The particle size distribution curves were also plotted. The results of the particle size distributions were indicated by the percentage of frequency as a function of particle size (mm). Tests were performed in triplicate for each sample.

**Differential scanning calorimetry**

Differential scanning calorimetry was used to determine the glass transition temperatures for apple pomace powders at different moisture/water activity levels. Powder samples were stored in a series of chambers of defined relative humidity between 11 and 75%. The powders were spread on plastic weigh pans and placed on a shelf in a plastic desiccator chamber. Each chamber contained a saturated salt solution that defined the humidity and therefore the equilibrated $a_w$ of the sample. Various saturated salt solutions of known water activity were placed in different desiccators: lithium chloride (0.11), potassium acetate (0.22), magnesium chloride (0.32), sodium bromide (0.57) and sodium chloride (0.75). After reaching equilibrium,
10-15mg of each sample was weighed and hermetically sealed in a DSC crucible with an aluminum lid, and analyzed in a DSC1 differential scanning calorimeter (Mettler-Toledo, Inc., Columbus, OH, United States) with StarE software. The dynamic scans were set from -60 °C to 60 °C at a rate of 10 °C / min. Nitrogen gas was used to flush the area during the scans. All DSC scans were done in triplicate.

The empirical Gordon and Taylor equation was used to model the dependence of glass transition temperature on moisture content:

\[ T_{go} = \frac{X_s T_{gs} + K X_w T_{gw}}{X_s + K X_w} \]  

(6)

or

\[ T_{go} = T_{gs} + K \left( \frac{X_w}{X_s} \right) (T_{gw} - T_{go}) \]  

(7)

where \( T_{go} \) is the onset glass transition temperature (°C) of the apple pomace powders, \( T_{gs} \) is the glass transition temperatures of amorphous dry solid, and \( T_{gw} \) is the glass transition temperature of amorphous water (-135 °C); \( X_s \) and \( X_w \) are the weight fraction of amorphous anhydrous solid matter and water, respectively; and \( K \) is an empirical parameter (Welti-Chanes and others 1999).

Flowability

Powders treated produced at different drying temperatures were tested by a standard Flowability tester (Liang and Langrish 2010). The Flowability tester was comprised of an aluminum cylinder that was rotated by a small electric motor. The inside diameter of the cylinder was 120 mm and its width was 100 mm. The cylindrical chamber contained two slots 70 mm × 4 mm located on opposite ends through which powder can flow as the cylinder is rotated. Prior to
operation, the inside of the cylinder was sprayed with 90% ethanol to diminish electrostatic effects. Approximate 7.0 g of powder was placed in the chamber, and metal endcaps attached to contain the sample. The cylinder was coupled to the motor and caused to turn at 34 RPM. The amount of powder emerging from the slots was collected in a pan below the cylinder and continuously weighed on an analytical balance [Mettler Toledo PL6001-S (± 0.1 g)] attached to a laptop computer. Plots of the percent of total powder that had emerged over time were prepared. In addition, the percent emerged at 10, 20 and 30 s was calculated. Tests were performed in triplicate for each condition.

**Moisture sorption isotherms**

As described above, various saturated salt solutions of known water activity were placed in a series of sealable chambers. The saturated salt solutions and corresponding $a_w$ were: lithium chloride (0.11), potassium acetate (0.22), magnesium chloride (0.32), sodium bromide (0.57) and sodium chloride (0.75). After samples had reached constant weight, the final weights were used to determine the new moisture content based on the initial moisture content. Average moisture content was obtained from three equilibrated samples. Plots of dry basis moisture versus $a_w$ were prepared.

The sorption data were fit to the Guggenheim-Anderson-de Boer (GAB) model. The Water Analyser software (Webb Tech Pty Ltd., Australia) was used to estimate the GAB parameters from experimental data of sorption isotherms at 23 °C. The GAB model can be written as (Nogueira and others 2007):

$$X = \frac{X_m C K a_w}{(1-K a_w)(1-K a_w + C K a_w)}$$

(8)
where \( X \) is the equilibrium moisture content of the apple pomace powders, \( X_m \) is the monolayer moisture content, \( C \) is a factor associated with surface enthalpy, and \( K \) represents a multilayered moisture component.

**Statistical analysis**

The evaluations between samples were performed by one-way analysis of variance and statistical significance by Tukey HSD (Honestly Significant Difference) test. All statistical analysis and correlations were performed using JMP® 9.0.2 (SAS Institute Inc., San Antonio, TX). Differences at \( P<0.05 \) were considered to be significant.

**Results and discussion**

**Pretreatment**

Different formulations were used to treat fresh apple pomace and the samples were evaluated visually for the onset of browning at intervals during storage. Solutions containing only ascorbic acid or citric acid alone were least effective. For example, pomace immersed in either 2% ascorbic acid or 2% citric acid had begun to brown within 2 days when stored at 4°C. For the combination of ascorbic acid and citric acid, the higher ratio of the ascorbic acid content (the other two components keep at the same concentration), the better inhibition effect it had. Solutions without sodium hexametaphosphate were not as effective at controlling browning. At best, browning could be forestalled for 4 days without Na HMP. The solution containing a combination of 2% ascorbic acid, 1% citric acid and 1% sodium hexametaphosphate showed the best inhibition of browning. Samples pretreated with this combination still had not shown signs of browning after 28 days.
Characterization of apple pomace powders

The water activity, moisture content, drying time and color parameters of pomace powder prepared at the three temperatures are listed in Table 3.2. The drying time for apple pomace (to reach $a_w = 0.2\pm0.04$) at 110 °C, 95 °C and 80 °C were 96 min, 130 min and 170 min, respectively. The moisture content ranged from 0.76 to 1.93 g/100g, and samples prepared at 80°C had slight but significantly higher moisture content.

As shown in Table 3.2, there was no significant difference in hue amongst the powders dried at 80 °C, 95 °C and 110 °C. Hue angles ranged from 73.7 to 76.8°, indicative of a red-to-yellow color. Chroma values indicated low color saturation and varied from 22.2 to 27.5. Samples prepared at 80°C had slightly lower c* values. L* ranged from 71.5 to 75.7 and samples prepared at 80°C were somewhat lighter than the others.

Krokida, Maroulis et al. (2001) suggested that L* can c* may be indicative of browning reactions that occur during drying. A decrease in lightness value (L*) and shifts to greater redness come with browning reactions that occur at higher temperatures. Several researchers have observed this phenomenon during drying and other processes involving high temperatures (Larrauri and others 1997; Leadley and others 2008). In our studies, H° of the powders did not vary with drying temperature, but samples were slightly darker (lower L*) when prepared at higher temperature, suggesting that some small amount of browning may have occurred.

The hygroscopicity of powders were 25.75%, 23.93% and 22.09%, for samples dried at 80 °C, 95 °C and 110 °C, respectively. Samples dried at successively higher temperatures were slightly less hygroscopic than those dried at lower temperatures. Because of the presence of low molecular weight sugars and organic acids still remaining in apple pomace after juicing, the
powders are hydroscopic (De Oliveira and others 2009), although the powders are less hygroscopic than those produced from full fruit pulp. In apple pomace, there is approximately 20% glucose, 48% of fructose and other acids. These contribute to a lower glass transition, and therefore greater mobility at $T>T_g$, and also present sites at which water molecules can bind.

**Particle size distribution**

The geometric mean diameter ($d_{gw}$), geometric mean diameter standard deviation ($S_{gw}$), sphericity (SHPT) and specific surface area ($S_v$) of pomace powders are listed in Table 3.3. The $d_{gw}$ and $S_{gw}$ values of samples decreased somewhat with temperature, with $d_{gw}$ ranging from 0.514 to 0.362 mm, and $S_{gw}$ from 0.493 to 0.321 mm. The sphericity increased slightly with temperature, ranging from 0.538 to 0.606. $S_v$, which measure the total surface area as compared to the volume, also increased with drying temperature and ranged from 18.802 to 25.385 m$^{-1}$. This indicates that while drying temperature did not make a large difference in particle size properties, the differences were significant. Particles dried at higher temperatures were smaller and more rounded. As the powders were prepared after drying, this suggests that the pomace dried at higher temperature was more fragile and could be more easily fractured during the grinding process.

**Glass transition temperature**

The $a_w$, moisture content and glass transition onset temperature ($T_{go}$) of apple pomace powders dried at 80°C, 95°C and 110°C are listed in Table 3.4. $T_{go}$ varied from 41.8 to 49.7°C for samples at $a_w$ 0.11, and from -25.2 to -30.4°C for samples held at $a_w$ 0.75. There was no significant difference between $T_{go}$ for samples dried at 95 °C and 110 °C at any given $a_w$. For samples at low water activity ($a_w = 0.11-0.32$), samples dried at 80 °C had slightly higher $T_{go}$
than those dried at 95°C and 110°C. Decreasing $T_{go}$ is often associated with increasing water content as the water serves to increase the relative free volume for motions of food molecules (Corey and others 2011). $T_{go}$ values as a function of moisture content were fit by the Gordon-Taylor equation (Eqn. 6). The $K$ values and glass transition temperatures of amorphous dry solid are reported in Table 3.5. The $T_g$ of amorphous dry solid decreased with drying temperature, ranging from 60.82°C for samples dried at 80°C to 44.05°C for samples dried at 110°C.

Roos and Karel (1991) reported that the critical viscosity of stickiness is reached at temperatures 10-20 °C higher than the measured $T_g$ of the material. Conventionally, the glass transition temperature was thought to be the threshold for stability of the dried product. In the conditions of storage, a small amount of water will depress the glass transition temperature significantly to influence the molecular mobility of the matrix as water is a strong plasticizer. The major factor that influences stability is moisture content. The increase in internal mobility of reactants and diffusivity of oxygen may cause accelerated chemical changes in dried products if stored at $T>T_g$. (Bhandari 2005). For ambient temperatures (~20°C), samples at 0.11 and 0.22 $a_w$ had $T_{go}$ above this value and should remain relatively stable and exhibit minimal stickiness. At 0.32 $a_w$, $T_{go}$ values were on the order of 9.89-16.57°C, indicating greater risk for clumping and chemical changes.

Flowability

Figure 3.1 showed the percentage of powder emerging from the Flowability tester as a function of time. Table 3.7 lists the percent of mass that had flowed out of the cylinder at 10, 20 and 30s. Overall, pomace powder dried at lower temperature had greater flowability. As can be seen, a total of 96.57% of sample dried at 80°C, 94.67% of sample dried at 95°C and 74.38% of
sample dried at 110°C emerged even after the cylinder had been rotated for several minutes. At 10 s, similar amounts had emerged (50.33 and 51.19%) for samples dried at 80 and 90°C, while only 74.38% of the material dried at 110°C had emerged. After 20 s, most of the powder (93.48%) dried at 80°C had emerged, while only 71.48% and 38.29% of powders dried at 95°C and 110°C had came out. At 50 s, 96.43, 91.29 and 65.71% of powders prepared at 80, 95 and 110°C had emerged.

Particle size is a major factor that influences powder flowability. Fitzpartrick reported that significant changes in flowability may not be noticed as size is reduced from 80 to 60 µm, however if the powder size is reduced by an order of magnitude changes in flowability may be apparent. When the surface area per unit mass of powder is increased, there is more surface area with more surface contacts available for cohesive forces (such as van der Waals attractions), specifically the frictional forces that resist flow. Particle size distribution, as depicted as a mass or volume fraction size is also crucial. If the amount of fines in the distribution is the majority, then the fines may have a dominant influence on flowability, then the mean particle size powder may be worse from the flowability (Fitzpartrick 2005; Fitzpatrick 2004). This may help explain the differences in flowability amongst the three powders tested. We found that the powders that had been dried at higher temperatures were smaller, more round, and had a greater surface area/volume. Thus, the particles may pack more densely, and be subject to greater cohesive forces and proximity as they slide past each other.

**Moisture isotherms**

Sorption isotherm also known as the inherent relationship between the equilibrium moisture content and the water activity in foodstuffs, is dependent on structure, composition of
the food material, and parameters such as pressure or temperatures (Kaymak-Ertekin and Gedik 2004). The moisture isotherms for the apple pomace powders dried at different temperatures are shown in Figure 3.3. The parameters and regression coefficients of GAB models are presented in Table 3.6. In general, powders prepared from pomace dried at 110°C had lower moisture content at a given \( a_w \) than powders prepared at lower temperatures.

The shape of the sorption isotherms were somewhere between Type 2 and Type 3, the former more commonly associated with amorphous materials and the latter associated with crystalline materials. Food powders can contain complex structures, and may have crystalline materials including sugars, and which can change state upon absorption of water (Mathlouthi and Rogé 2003). Moisture levels increase modestly as \( a_w \) increases from 0 to 0.5 indicating relatively low hygroscopicity in this region. This behavior is typically seen for materials with high crystallinity or substances that are readily dissolved with added moisture. At low \( a_w \), the moisture resides mostly on the solid surfaces and possibly exists in a single layer (deMan 1999). At intermediate moisture, the influence of insoluble solids and entrapment of water in porous spaces becomes more important. At \( a_w > 0.6 \), the influence of soluble solids on water becomes more prevalent.

As seen in Table 3.6, the monolayer moisture ranged from 0.058 to 0.067 g H\(_2\)O/g solid, with pomace prepared at 110°C having slight higher \( m_0 \) values. The value of \( c \) was also lower for materials formed at 110°C. The constant \( c \) has been denoted the energy constant, as it relates to the enthalpy difference between bulk water and water absorbed in the first monolayer.
Conclusion

This study showed that apple pomace could be dried in a continuous vacuum-belt dryer, then ground into powders with good color and flow properties, typically within 96-170 min. The drying temperature had a small effect on powder properties. Drying at 110°C resulted in smaller particles and some slight additional browning. However, these particles did not flow nearly as well as those produced at 80 and 95°C drying temperatures. Properties of powders produced at 80 and 95°C were most similar. Thus, considering the faster drying time (96 versus 130 min) drying at 95°C would seem advisable. Subsequent work will focus on understanding the effects of vacuum drying on phytochemicals and other chemical constituents, and determining drying curves needed to better understand the stages and mechanisms of drying.
Table 3.1 – Effect of browning inhibitor formulations on control of browning in fresh apple pomace

<table>
<thead>
<tr>
<th>Formulation</th>
<th>Ascorbic Acid (%)</th>
<th>Citric Acid (%)</th>
<th>Sodium Hexametaphosphate (%)</th>
<th>Onset of browning at 4 °C (days)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2%</td>
<td>1%</td>
<td>1%</td>
<td>&gt;28</td>
</tr>
<tr>
<td>2</td>
<td>2%</td>
<td>0%</td>
<td>1%</td>
<td>2</td>
</tr>
<tr>
<td>3</td>
<td>0%</td>
<td>2%</td>
<td>1%</td>
<td>2</td>
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<td>1%</td>
<td>2%</td>
<td>1%</td>
<td>15</td>
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<td>0%</td>
<td>0%</td>
<td>3</td>
</tr>
<tr>
<td>6</td>
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<td>1%</td>
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<td>&lt;1</td>
</tr>
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<td>7</td>
<td>2%</td>
<td>1%</td>
<td>0%</td>
<td>4</td>
</tr>
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<td>8</td>
<td>1%</td>
<td>2%</td>
<td>0%</td>
<td>3</td>
</tr>
<tr>
<td>9</td>
<td>0%</td>
<td>2%</td>
<td>0%</td>
<td>&lt;2</td>
</tr>
</tbody>
</table>

*Formulations of the browning inhibitors
Table 3. 2– Characterization of vacuum belt dried apple pomace powders

<table>
<thead>
<tr>
<th>Parameters</th>
<th>80 °C</th>
<th>95 °C</th>
<th>110 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water Activity</td>
<td>0.20 ± 0.04</td>
<td>0.20 ± 0.04</td>
<td>0.20 ± 0.04</td>
</tr>
<tr>
<td>Moisture Content (%)</td>
<td>1.93 ± 0.59&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.76 ± 0.47&lt;sup&gt;b&lt;/sup&gt;</td>
<td>0.85 ± 0.51&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>Drying Time (min)</td>
<td>170 ± 5</td>
<td>130 ± 5</td>
<td>96 ± 5</td>
</tr>
<tr>
<td>Color Parameters</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>L&lt;sup&gt;*&lt;/sup&gt;</td>
<td>75.7 ± 0.2&lt;sup&gt;a&lt;/sup&gt;</td>
<td>74.3 ± 1.2&lt;sup&gt;a,b&lt;/sup&gt;</td>
<td>71.5 ± 1.9&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>C&lt;sup&gt;*&lt;/sup&gt;</td>
<td>22.2 ± 0.1&lt;sup&gt;b&lt;/sup&gt;</td>
<td>23.8 ± 0.9&lt;sup&gt;b&lt;/sup&gt;</td>
<td>27.5 ± 2.1&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>H&lt;sup&gt;+&lt;/sup&gt;</td>
<td>73.8&lt;sup&gt;a&lt;/sup&gt; ± 0.9&lt;sup&gt;aa&lt;/sup&gt;</td>
<td>73.7&lt;sup&gt;a&lt;/sup&gt; ± 0.5&lt;sup&gt;aa&lt;/sup&gt;</td>
<td>76.8&lt;sup&gt;a&lt;/sup&gt; ± 1.5&lt;sup&gt;aa&lt;/sup&gt;</td>
</tr>
<tr>
<td>Hygroscopicity</td>
<td>25.75 ± 0.34&lt;sup&gt;a&lt;/sup&gt;%</td>
<td>23.93 ± 0.06&lt;sup&gt;b&lt;/sup&gt;%</td>
<td>22.09 ± 0.11&lt;sup&gt;c&lt;/sup&gt;%</td>
</tr>
</tbody>
</table>

Values are means of triplicate determinations ± standard deviation.

<sup>a,b,c</sup> Different letters in the same property denote significant differences (P < 0.05).

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Table 3.3 – Particle size parameters

<table>
<thead>
<tr>
<th>Drying Temperature</th>
<th>$d_{gw}$ (mm)$^1$</th>
<th>$S_{gw}$ (mm)$^2$</th>
<th>SPHT$^3$</th>
<th>$S_v$ (mm$^{-1}$)$^4$</th>
</tr>
</thead>
<tbody>
<tr>
<td>80°C</td>
<td>0.514</td>
<td>0.4935</td>
<td>0.538</td>
<td>18.802</td>
</tr>
<tr>
<td>95°C</td>
<td>0.468</td>
<td>0.410</td>
<td>0.604</td>
<td>21.537</td>
</tr>
<tr>
<td>110°C</td>
<td>0.362</td>
<td>0.321</td>
<td>0.606</td>
<td>25.385</td>
</tr>
</tbody>
</table>

$^1d_{gw}$: Geometric mean diameter

$^2S_{gw}$: Geometric mean diameter standard deviation

$^3$SPHT: Sphericity

$^4S_v$: Specific surface area
Table 3.4—$a_w$, moisture content and glass transition temperature of apple pomace powders at different $a_w$ levels

<table>
<thead>
<tr>
<th>$a_w$</th>
<th>Drying Temperature</th>
<th>Moisture content (g H$_2$O/100g solid)</th>
<th>Onset $T_g$ $^1$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.11</td>
<td>80 °C</td>
<td>1.75 ± 0.04$^a$</td>
<td>49.71 ± 4.18$^a$</td>
</tr>
<tr>
<td></td>
<td>95 °C</td>
<td>1.75 ± 0.75$^a$</td>
<td>43.04 ± 2.97$^{a,b}$</td>
</tr>
<tr>
<td></td>
<td>110 °C</td>
<td>0.86 ± 0.07$^a$</td>
<td>41.80 ± 1.66$^b$</td>
</tr>
<tr>
<td></td>
<td>80 °C</td>
<td>3.35 ± 0.06$^a$</td>
<td>38.13 ± 0.51$^a$</td>
</tr>
<tr>
<td>0.22</td>
<td>95 °C</td>
<td>2.61 ± 0.04$^b$</td>
<td>32.51 ± 1.25$^b$</td>
</tr>
<tr>
<td></td>
<td>110 °C</td>
<td>2.15 ± 0.06$^c$</td>
<td>32.06 ± 2.57$^b$</td>
</tr>
<tr>
<td></td>
<td>80 °C</td>
<td>5.17 ± 0.08$^a$</td>
<td>30.57 ± 1.47$^a$</td>
</tr>
<tr>
<td>0.32</td>
<td>95 °C</td>
<td>5.17 ± 0.39$^a$</td>
<td>16.57 ± 5.16$^b$</td>
</tr>
<tr>
<td></td>
<td>110 °C</td>
<td>3.39 ± 0.09$^b$</td>
<td>18.94 ± 1.62$^b$</td>
</tr>
<tr>
<td></td>
<td>80 °C</td>
<td>10.27 ± 0.09$^a$</td>
<td>9.89 ± 5.64$^a$</td>
</tr>
<tr>
<td>0.57</td>
<td>95 °C</td>
<td>9.34 ± 0.10$^b$</td>
<td>5.11 ±3.78$^a$</td>
</tr>
<tr>
<td></td>
<td>110 °C</td>
<td>8.90 ± 0.16$^c$</td>
<td>2.54 ± 1.71$^a$</td>
</tr>
<tr>
<td></td>
<td>80 °C</td>
<td>21.63 ± 0.11$^a$</td>
<td>-25.22 ± 3.40$^a$</td>
</tr>
<tr>
<td>0.75</td>
<td>95 °C</td>
<td>20.42 ± 0.039$^b$</td>
<td>-29.66 ± 2.16$^a$</td>
</tr>
<tr>
<td></td>
<td>110 °C</td>
<td>19.11 ± 0.09$^c$</td>
<td>-30.40 ± 1.41$^a$</td>
</tr>
</tbody>
</table>

Values are means of triplicate determinations ± standard deviation.

$^{a,b,c}$ Different letters in the same property denote significant differences ($P < 0.05$).

$^1$ Onset glass transition temperatures
Table 3. 5– Gordon-Taylor equation constants and glass transition temperature of amorphous dry solid ($T_{gs}$) for apple pomace powders dried at 80°C, 95°C, and 110°C.

<table>
<thead>
<tr>
<th>Drying Temperature</th>
<th>K$^1$</th>
<th>$T_{gs}$ $^2$</th>
<th>R$^2$ $^3$</th>
</tr>
</thead>
<tbody>
<tr>
<td>80 °C</td>
<td>3.58</td>
<td>60.82</td>
<td>0.997</td>
</tr>
<tr>
<td>95 °C</td>
<td>3.68</td>
<td>50.66</td>
<td>0.979</td>
</tr>
<tr>
<td>110 °C</td>
<td>3.68</td>
<td>44.05</td>
<td>0.983</td>
</tr>
</tbody>
</table>

$^1$K: An empirical parameter

$^2$T$_{gs}$: The glass transition temperatures of dry solid

$^3$R$^2$: The regression coefficient
Table 3. 6– Parameters of GAB models for apple pomace powders

<table>
<thead>
<tr>
<th>Parameters</th>
<th>80 °C</th>
<th>95 °C</th>
<th>110 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>( m_0 ) (g H(_2)O/g solid) (^1)</td>
<td>0.060</td>
<td>0.058</td>
<td>0.067</td>
</tr>
<tr>
<td>( C ) (^2)</td>
<td>2.5945</td>
<td>2.3477</td>
<td>1.1522</td>
</tr>
<tr>
<td>( K ) (^3)</td>
<td>1.000</td>
<td>1.000</td>
<td>1.000</td>
</tr>
<tr>
<td>( R^2 ) (^4)</td>
<td>0.9980</td>
<td>0.9939</td>
<td>0.9997</td>
</tr>
</tbody>
</table>

\(^1\)\( m_0 \): Monolayer moisture content

\(^2\)\( C \): A factor associated with surface enthalpy

\(^3\)\( K \): Represents a multilayered moisture component

\(^4\)\( R^2 \): The regression coefficient
Table 3. Results from flowability testing expressed as the amount of powder emerged after 10, 20 and 30s

<table>
<thead>
<tr>
<th>Samples</th>
<th>Percentage at 10s(^1)</th>
<th>Percentage at 20s</th>
<th>Percentage at 50s</th>
<th>Total Percentage came out(^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>80°C</td>
<td>51.19%</td>
<td>93.48%</td>
<td>96.43%</td>
<td>96.57%</td>
</tr>
<tr>
<td>95°C</td>
<td>50.33%</td>
<td>71.48%</td>
<td>91.29%</td>
<td>94.67%</td>
</tr>
<tr>
<td>110°C</td>
<td>29.14%</td>
<td>38.29%</td>
<td>65.71%</td>
<td>74.38%</td>
</tr>
</tbody>
</table>

\(^1\)Percentage mass merging out of the cylinder at 10s

\(^2\)Total percentage merging out of the cylinder when no more samples came out
Figure 3.1– Particle size distribution of apple pomace powders
Figure 3.2– The percentage of the powder emerging from the Flowability standard tester as a function of time.
Figure 3.3 – Moisture isotherms of apple pomace powders
 CHAPTER 4
CHEMICAL PROPERTIES OF APPLE POMACE POWDERS PRODUCED BY VACUUM BELT DRYING

Abstract

Apple pomace was dried by vacuum belt drying at 80, 95 and 110 °C and ground into powders. Chemical properties of the three samples were evaluated including total phenolics content (TPC), monomeric anthocyanins (TMA), and dietary fiber content (TDF) and were compared to freeze-dried fresh apple and apple pomace. In all cases, TPC, TMA and TDF were higher in dried pomace than in freeze-dried whole apple, indicating that pomace is an excellent source of phytochemicals and fiber. Vacuum-dried pomace had 4.49 to 5.19 g GAE/100 mg TPC, with greatest retention for pomace dried at 80 and 95 °C. TPC for pomace dried at 80 or 95 °C were not significantly different than that for freeze-dried pomace. TMA levels (7.4 mg C3G/100 g) were highest in pomace vacuum-dried at 80°C. TDF ranged from 44.2-49.5% in vacuum-dried pomace and was not significantly different than TDF of freeze-dried pomace (48.0%). Increasing the vacuum-drying temperature from 80 to 95 °C reduced the drying time from 130 to 96 min without any deterioration of TPC, TMA or TDF.
Introduction

Apple pomace is the main byproduct from juice or cider making. It possesses a high phytochemical content that can be recovered for secondary food and non-food uses. Due to relatively high carbohydrate content, apple pomace is used as a substrate in a number of microbial processes for the production of organic acids, enzymes, single cell protein, ethanol, low alcoholic drinks and pigments. There has also been an increase in the utilization of apple pomace as a food processing residue for the extraction of value-added products such as dietary fiber, protein, natural antioxidants, biopolymers, pigments and other compounds with unique properties (Bhushan and others 2008).

Increased apple intake has been linked to reduced risk of lung cancer, cardiovascular disease, coronary and total mortality, symptoms of chronic obstructive pulmonary disease, and risk of thrombotic stroke (Wolfe and Liu 2003). The National Research Council recommends consuming five or more servings of fruits and vegetables a day which may protect against cardiovascular diseases and cancer through a variety of mechanisms. The health-promoting properties of apples have been associated with specific polyphenolic compounds and dietary fiber found in the fruit. Phenolic compounds are widely distributed in fruits and vegetables where they contribute to color and flavor. Their contribution during technological processes, particularly in browning phenomena (enzymic or not), has been demonstrated by several authors (Raynal and others 1989). In apples, the major phenolics include procyanidins, catechin, epicatechin, chlorogenic acid, phloridzin, and the quercetin conjugates (Bhushan and others 2008). The fiber in apples consists of cellulose, hemicellulose, lignin and pectin (Nawirska and Kwasniewska 2005). Studies have shown that most of the phytochemicals and dietary fiber
reside in the peel (Wolfe, Wu and Liu, 2003). Apple pomace, which still contains much of the peel, is therefore an excellent source for recovering phytochemicals and fiber.

Drying is a primary means of extending the shelflife of fruits and vegetables without chemical preservatives, and reduces both the size of package and the transport costs (Larrauri 1999). High temperatures and long drying times are often required to remove water from high-sugar products, and this may cause serious damage to the color, flavor and nutrients of the dehydrated product. Studies have shown that elevated drying temperatures adversely effect the phenolic content, color, and antioxidant activity of red grape pomace peels, especially when temperatures are higher than 100 °C (Larrauri and others 1997). Vacuum drying has been proposed as one means of limiting these problems, as drying can occur at lower temperature. Diminished heat transfer in vacuum can limit the drying rate, while it may be enhanced by infrared radiation (Drouzas and others 1999). In addition, the system can be operated in a continuous mode by depositing the product under an airlock onto a moving belt that passes over the heaters.

Most high fiber content products are milled to improve acceptability in the final food products and improve convenience of use. The fractions obtained can have different chemical composition, depending on the origin and history of the cell wall material (Larrauri 1999). Dried and ground apple pomace powder can be an easily handled product with extended shelf-life. Pomace powders can also be incorporated with other component in food products (Rupasinghe and others 2008; Sudha and others 2007; Masoodi and Chauhan 1998). Earlier studies showed that apple pomace can be vacuum-belt dried and ground into powders with good flowability and hygroscopicity properties.
The objective of this study was to evaluate the phytochemical and fiber properties of apple pomace powders dried at different temperatures. Total phenolic, anthocyanin, and dietary fiber of apple pomace powders dried by vacuum belt dryer at 80 °C, 95 °C and 110 °C were compared with freeze dried fresh apple and apple pomace.

Materials and methods

Chemicals and reagents

Citric acid, ascorbic acid, sodium hexametaphosphate (NaHMP), sodium phosphate (monobasic, anhydrous), sodium phosphate (dibasic, anhydrous), sodium hydroxide and hydrochloric acid were purchased from J.T. Baker (Phillipsburg, NJ). Folin-Ciocalteu’s phenol reagent, sodium carbonate, gallic acid, ethyl alcohol and a total dietary fiber kit were purchased from Sigma-Aldrich Chemical Co. (St. Louis, MO). Acetone was purchased from Fisher Scientific (Suwanee, GA).

Apple pomace powder processing

Apple pomace was obtained directly from the belt juice pressing line at Mercier Orchards (Blue Ridge, GA). The mash consisted of a mix of Rome Beauty, Fuji, Red Delicious and Golden Delicious apples. During pressing, 1% by weight of rice hulls (Riceland®®, Riceland Foods, Stuttgart, AR) was added to the slurry to improve juice yield. The apple pomace was immediately dipped into a browning inhibitor solution (2% ascorbic acid, 2% citric acid and 1% NaHMP) for 3 min and dewatered in cheese cloth. Preliminary studies showed that this solution resulted in no observable browning up to 28 days for pomace stored at 4 °C. The dewatered apple pomace was immediately frozen at -20 °C in polyethylene buckets until use.
The frozen apple pomace was thawed overnight at 4 °C and rinsed with distilled water prior to drying. A laboratory scale vacuum belt dryer (Zwag, CH-5312 Zchokke Wartman Ltd. Bucher, Döttingen, Switzerland) was used to dry the pomace at three different temperatures (either 80 °C, 95 °C or 110 °C) with 2.5-3.1kPa vacuum pressure. Apple pomace was maintained at uniform thickness (5mm) for drying runs at each temperature. Dried apple pomace was removed once the water activity has reached 0.2 ± 0.04 (as determined by preliminary trials). The pomace was first chopped in a food processor (Kitchen Aid KFP350WH Food Processor, St. Joseph, MI) for 40s. The seeds were removed by passing the chopped material through a sieve (American standard No. 6, 3.35mm opening). Samples were then ground for 1 min into powders with a Model 111338 grinder (General Electric, Fairfield, CT). Previous studies showed that this resulted in powders with geometric mean diameter on the order of 0.3-0.5 mm.

**Extraction of phytochemicals from apple pomace powder**

The extraction of phenolic compounds from pomace powder was performed as described by Wolfe and Liu (Wolfe and Liu 2003) with some modifications. Apple pomace powder (15 g) was combined with 150 ml of chilled 80% (v/v) acetone and blended using a PT-1200 Polytron homogenizer (Brinkmann Instruments, Westbury, NY) at 15,000 rpm for 10 min. The slurry was then filtered through Whatman No.1 filter paper in a Büchner funnel by suction filtration. The filter cake was washed twice with 15 ml of the acetone solution. The filtrate was recovered and evaporated using a rotary evaporator (Brinkmann Instruments Inc., subsidiary of Sybron Corp., Westbury, NY) at 45 °C until less than 10% of the initial volume remained. The extract was made up to 30 ml with deionized water and frozen at -30 °C until analysis. All extracts were performed in triplicate.
Extraction of phytochemicals from fresh apple and apple pomace

Fresh apples, from the same batch of apples that were used to make apple cider, was obtained from Mercier Orchards (Blue Ridge, GA). Apple pomace was made by a centrifugal juice extractor (Cuisinart CJE-1000, East Windsor, NJ) and fresh apples were cut into slices. Both the apple pomace and apple slices were dipped immediately into liquid nitrogen. The frozen apple slices and apple pomace were freeze-dried and ground with a food processor (Kitchen Aid KFP350WH Food Processor, St. Joseph, MI) and a Model 111338 grinder (General Electric, Fairfield, CT) into a powder. The extraction of phytochemicals from the fresh apple and apple pomace were similar to that described above. All extracts were prepared in triplicate.

Determination of total phenolics content

The total phenolics content of apple pomace powder, fresh apple and apple pomace were measured using a modified colorimetric Folin-Ciocalteu method (Wolfe and Liu, 2003). 0.5 ml of diluted extract (diluted the known extract 40 times) were added to a test tube. Folin-Ciocalteu’s phenol reagent (0.5 ml) was added to the solution. Next, 5 ml of 7% sodium carbonate solution was added into the test tubes, and the mixture was diluted to 12 ml with deionized water. The color was allowed to develop for 90 min, and the absorbance was read at 760 nm using an Agilent 8453 UV-Visible spectrophotometer. The absorbance was compared to a standard curve of gallic acid equivalents/100 mg of the vacuum belt dried pomace powder samples, freeze dried apple pomace, freeze dried fresh apples, and the concentration was determined after considering dilution factors.
Determination of anthocyanin content

Monomeric anthocyanin content of the fresh apples, apple pomace and apple pomace powder were measured using a spectrophotometric pH differential protocol (Boyles and Wrolstad 1993; Liu and others 2002). Briefly, the extracts were mixed respectively with 0.025 M potassium chloride pH 1 buffer and 0.4 M sodium acetate pH 4.5 buffer in a 1:8 ratio of extract to buffer. Deionized water was used to zero the spectrophotometer at 515 nm and 700 nm. The absorbance of the mixture was then measured at 515 and 700 nm against a blank (deionized water). The total monomeric anthocyanin content was calculated as follows:

\[
TMA \ (\text{mg} / \text{L}) = \frac{(A \times MW \times DF \times 1000)}{(\epsilon \times 1)}
\]  

(1)

where \(A\) is the absorbance = \((A_{515} - A_{700})_{\text{pH} \ 1.0} - (A_{515} - A_{700})_{\text{pH} \ 4.5}\); \(MW\) is molecular weight for cyanidin 3-\(\alpha\)-glucoside (449.2 g/mol); \(\epsilon\) is the molar absorptivity of cyanidin 3-\(\alpha\)-glucoside (26,900 L/mol·cm) and \(DF\) is the dilution factor. Anthocyanin content was expressed as milligrams of cyanidin 3-\(\alpha\)-glucoside equivalents (C3G) per 100 g of fresh apple, apple pomace or pomace powder.

Dietary fiber and protein content determination

Total dietary fiber content of powders dried at 80 °C, 95 °C, and 110 °C was compared with that of freeze dried fresh apple and apple pomace using an AOAC procedure (Method 985.29, 1997) with some modifications. Four 1 g ± 20 mg pre-dried samples of each material were mixed with 0.1 ml heat stable \(\alpha\)-amylase and incubated at pH 6.0, 95 °C for 15 min. Then protease was added to digest the protein at pH 7.5, 60 °C for 30 min. The pH of the solution was
adjusted to 4.5 and amyloglucosidase was added to remove the starch. The temperature was kept at 60 °C for 30 min. Ethanol was then added to precipitate the soluble dietary fiber. After the residues were filtered and washed with ethanol and acetone, they were dried at 70 °C in a vacuum oven (1430MS, VWR Scientific, Suwanee, GA). Half of the samples were analyzed for their protein content using a Perkin-Elmer 2400 Series II CHNS/O elemental analyzer (Waltham, MA). A factor of 6.25 was used to convert the nitrogen content to crude protein content. The other half of the samples were ashed for 5 h at 525 °C. The mass of each step was recorded to the nearest 0.1 mg. Blanks were run along with samples through the entire procedure to measure the effects of the reagents on the residues. The percentage of total dietary fiber (TDF) content was calculated as follows:

\[
\% \ TDF = \left(\frac{R_{\text{sample}} - P_{\text{sample}} - A_{\text{sample}} - R_{\text{blank}} - P_{\text{blank}} - A_{\text{blank}}}{SW}\right) \times 100
\]

where R stands for average residue weight (mg), P is the average protein weight (mg), A is the average ash weight (mg), and SW is the average sample weight (mg).

Statistical analysis

Results were analyzed by one-way analysis of variance (ANOVA) and statistical differences were examined by the Tukey’s HSD (Honestly Significant Difference) test. All statistical analysis and correlations were performed using JMP® 9.0.2 (SAS Institute Inc., San Antonio, TX). Differences at p < 0.05 were considered to be significant.

Results and discussion

The total phenolics contents (TPC) of the apple pomace vacuum-dried at 80 °C, 95 °C and 110 °C; freeze-dried apple pomace; and freeze-dried whole apple are shown in Table 4.1. For the dried powders, TPC varied from 4.49 to 5.19 g GAE/100 mg dry weight. Powders dried
at 80°C had higher levels of TPC than those dried at 110°C. Freeze-dried apple pomace had 5.11 g GAE/100 mg. There was no significant difference in TPC of freeze-dried pomace and pomace vacuum-dried at either 80 or 95°C. The TPC of freeze-dried fresh apple was 1.78 g GAE/100 mg and was lower than all other treatments. Others have measured between 110 to 357 mg GAE/100 g in fresh apple (Podsędek and others 2000; Wolfe and Liu 2003), or approximately 0.69 to 2.23 g GAE/100 mg when expressed on a dry weight basis. Fresh apple, which includes peel and pulp, has greater levels of soluble solids including fructose, glucose, sucrose and sorbitol (Fuleki and others 1994). As previously noted, the peel has greater concentrations of phytochemicals and fiber. Thus, as the pomace has much higher levels of peel, it too would be more concentrated with phenolic compounds than the whole apple.

High temperatures are known to degrade polyphenolic compounds, particularly when they are unbound and in high moisture systems (Sadilova and others 2007). The rate of breakdown is often commodity-specific, and some phenolic compounds are more susceptible to heat than others. For example, in blueberry juice heated between 40 and 80°C, the half-life for anthocyanins ranged from 180 to 5.1 h (Kechinski and others 2010). In dried fruit products, more mechanisms may come into play. Vega-Gálvez, Ah-Hen et al. (2012) noted that many phenolic glycosides are localized in the hydrophilic regions of the cell such as in vacuoles and apoplasts, or exist as soluble phenols in the cytoplasm and in the cell nuclei, and seem to receive protection from heat by the material of the cell walls. According to Maillard (1995), the decrease in phenolic compounds during drying of biological materials at higher temperatures can be explained in several ways: some of the phenolic acids (such as hydroxycinnamic acids) are bound to insoluble cell wall material, and these can be partially released with heat. Higher
temperatures also cause lignin and other complex polysachharides to be degraded and this further helps the release of phenolic acids. This allows greater thermal degradation of the phenolics to ensue.

The total monomeric anthocyanins (TMA) in the vacuum-dried apple pomace, freeze-dried apple pomace, and freeze-dried apple are shown in Table 4.1. TMA contents were 4.5-7.4 mg C3G/100 g for the vacuum-dried pomace powders, 2.67 mg C3G/100 g for freeze-dried pomace, and 1.44 mg C3G/100 g for freeze-dried whole apple. Highest levels were measured for vacuum-dried pomace prepared at 80°C. No significant differences for anthocyanin content was found, however, amongst powders dried at different temperatures, freeze-dried apple pomace and freeze-dried fresh apples.

Table 4.1 also lists the total dietary fiber (TDF) content of the powders, apple pomace and fresh apple. For vacuum-dried pomace, TDF ranged from 44.2% for powders prepared at 80°C to 49.5% for powders prepared at 110°C. There was no difference between TDF for vacuum-dried pomace and freeze-dried pomace (48.0%). Bhushan, Kalia et al. (2008) reported that apple pomace contains 35 and 60% dietary fiber depending on the type of apple and preparation method employed. Of this, approximately 36.5% exists as insoluble fiber, a relatively high amount for fruits, and 14.6% exists as soluble fiber. They also note that the main constituents of pomace fiber are pectins, cellulose, hemicelluloses, lignins and gums. TDF for the fresh apple was much lower (12.4%) than for dried apple pomace. Again, this reinforces that much of the fiber content resides with the peel. One study showed that particle size of powders affects the yield of dietary fiber, in that powders with larger particle size give a better recovery of TDF (Larrauri and others 1996).
To be an ideal fiber source, a well-balanced proportion of soluble fiber and insoluble fiber is required. As (Larrauri and others 1996) stated that apple pomace is a good source of fiber, although partial degradation of the soluble dietary fiber components can occur at high drying temperatures (Larrauri and others 1996). In this study, no additional loss of TDF was incurred by vacuum-drying at temperatures up to 110°C.

Conclusion

Previous studies have shown that apple pomace can be prepared by vacuum-belt drying with little of the heat-induced browning incurred with hot-air drying, and in a more rapid fashion than can be found with freeze-drying (Wolfe and Liu 2003). In addition, the pomace had good flow properties and low enough hygroscopicity that it could be handled without caking or clumping in short periods. In this study, we found that vacuum-dried pomace had similar total phenolics to freeze-dried pomace, and higher levels of monomeric anthocyanins, particularly when dried at either 80 or 95°C. As the drying time could be shortened from 130 to 96 min, the latter temperature may be preferred. In addition to phytochemicals, the pomace powder is a good source of dietary fiber. We found that the TDF recovered after vacuum-drying was no different than that for freeze-dried pomace. Future studies will focus on optimizing the drying process and studying the effects of drying on specific phytonutrients in the pomace.
Table 4.1 – Comparison of total phenolic contents (TPC), monomeric anthocyanins (TMA), and total dietary fiber (TDF) for vacuum-dried apple pomace powder, freeze-dried apple pomace, and freeze-dried fresh apple. TPC and TMA expressed per dry weight.

<table>
<thead>
<tr>
<th></th>
<th>Vacuum Dried</th>
<th>Freeze Dried Fresh Apple</th>
<th>Freeze Dried Apple Pomace</th>
</tr>
</thead>
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<tr>
<td></td>
<td>80 °C</td>
<td>95 °C</td>
<td>110 °C</td>
</tr>
<tr>
<td>TPC (g GAE/100g as delivered)</td>
<td>5.19 ± 0.09&lt;sup&gt;a&lt;/sup&gt;</td>
<td>4.79 ± 0.10&lt;sup&gt;a,b&lt;/sup&gt;</td>
<td>4.49 ± 0.24&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>TMA (mg C3G/100g as delivered)</td>
<td>7.4 ± 2.9&lt;sup&gt;a&lt;/sup&gt;</td>
<td>6.5 ± 2.3&lt;sup&gt;a&lt;/sup&gt;</td>
<td>4.5 ± 1.6&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>TDF (%)</td>
<td>44.2&lt;sup&gt;a&lt;/sup&gt;</td>
<td>48.4&lt;sup&gt;a&lt;/sup&gt;</td>
<td>49.5&lt;sup&gt;a&lt;/sup&gt;</td>
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<sup>a,b,c</sup> Different superscripts for the same property denote significant differences (P < 0.05).
CHAPTER 5

CONCLUSION

The drying temperatures (80°C, 95°C, 110°C) for vacuum belt dried apple pomace had a small effect on powder physical properties. The results of the comparison of total phenolic contents (TPC), monomeric anthocyanins (TMA), and total dietary fiber (TDF) for vacuum-dried apple pomace powder, freeze-dried apple pomace, and freeze-dried fresh apple showed that apple pomace powder is a good source of phytochemicals, as well as a good source of dietary fiber. A drying temperature of 95°C is preferred as it provides a relatively short drying time without unduly compromising the physical and chemical properties of the powder.
APPENDIX 1: Schematic of Vacuum Belt Dryer

1- Feed tank, 2- Radiating heating plate, 3- Conducting heating plate, 4- Conveyor belt, 5- Control panel, 6- Collecting tank, 7- Scraper, 8- Vacuum gauge, 9- Vacuum pump
REFERENCES


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