DEVELOPMENT OF BROCCOLI POWDER USING VACUUM BELT DRYING

by

PO-AN CHI

(Under the Direction of WILLIAM L. KERR)

ABSTRACT

The study determined if high quality broccoli powder could be produced by vacuum drying. The effect of temperature, maltodextrin and pretreatment on drying kinetics, physical characteristics of the powder and nutritional quality were studied. The Wang and Singh model best fit drying rate data. Powders exhibited a Type III isotherm and were best fit by the GAB model. With 10% maltodextrin, drying efficiency was 200% higher and hygroscopicity was lower than samples without maltodextrin. Blanching with 0.1% (w/v) $NaHCO_3$ and 0.1% (w/v) MgO resulted in greener samples with more vitamin C. Vitamin C content was two-fold greater in powders produced by vacuum drying and freeze drying than in hot-air drying. Steam blanching preserved 12.5% more of the endogenous antioxidants than that by boiling with $NaHCO_3$ and MgO. Hot-air drying reduced the total antioxidant content by 31.5% in comparison with vacuum drying. Freeze drying resulted in the lowest sulforaphane content.

Key words: Broccoli, Vacuum belt drying, Drying model, Vitamin C, Sulforaphane

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PO-AN CHI

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by

PO-AN CHI

Major Professor: William L. Kerr

Committee: Ronald B. Pegg

George A. Cavender

Electronic Version Approved:

Susan Barbour
Dean of the Graduate School
The University of Georgia
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TABLE OF CONTENTS

ACKNOWLEDGEMENTS IV

LIST OF FIGURES VIII

LIST OF TABLES IX

CHAPTER 1

INTRODUCTION 1

References 4

CHAPTER 2

REVIEW OF LITERATURE 7

Broccoli 7

Chlorophyll 9

Vitamin C 12

Glucosinolate 17

Maltodextrin 21

Drying 23

Types of Drying 26

Food Powders 32

Benefits of Vegetable Powder 33

References 42

CHAPTER 3

THE EFFECT OF PROCESS CONDITIONS ON DRYING AND PHYSICAL CHARACTERISTICS OF CONTINUOUS VACUUM BELT DRIED BROCCOLI 56

Abstract 57

Introduction 58

Materials & Methods 60

Results & Discussion 68

Conclusion 76

References 89

CHAPTER 4

THE EFFECT OF PROCESS CONDITIONS ON THE COMPOSITION OF CONTINUOUS VACUUM BELT DRIED BROCCOLI 94

Abstract 95

Introduction 96

Materials & Methods 98

Results & Discussion 105

Conclusion 110

References..113

CHAPTER 5

SUMMARY AND CONCLUSION 117

LIST OF FIGURES

Figure 2-1. Broccoli (Brassica oleracea var. italica). 35

Figure 2-2. Structure of chlorophyll. 35

Figure 2-3. Structure of ascorbic acid. 36

Figure 2-4. Structure of glucosinolate. 36

Figure 2-5. Structure of sulforaphane. 37

Figure 2-6 The mechanism of sulforaphane induced Phase 2 detoxification 37

Figure 2-7. The relation of drying rate and moisture content. 38

Figure 2-8. Water activity diagram. 39

Figure 2-9. Energy cost breakdown for freeze-drying process. 39

Figure 3-1 Drying kinetics of broccoli paste under vacuum belt dryer. 78

Figure 3-2 Moisture isotherm of broccoli powders at different conditions…………..79

Figure 3-3 Broccoli powder of different drying method and pretreatment…………...80

Figure 3-4 Broccoli powder of two level of maltodextrin and drying temperature..…80

Figure 4-1 High-performance liquid chromatography chromatograms of sulforaphane standard and broccoli sample using evaporative light scattering detector ……..111

LIST OF TABLES

Table 2-1 Glass transition temperature of sugars and maltodextrins 40

Table 2-2 Terms recommended by the British Pharmacopeia for use with powders... 41

Table 3-1 Moisture content, water activity and hygroscopicity constant of each treatment 81

Table 3-2. Five common drying models. 83

Table 3-3. Statistical analysis for Wang and Singh model and the predicted constants. 84

Table 3-4. Parameters of GAB model moisture isotherms. 85

Table 3-5. Color parameters of vacuum belt dried broccoli powders. 87

Table 4-1. The content of vitamin C, total antioxidant activity and sulforaphane in broccoli powders. 112

CHAPTER 1

INTRODUCTION

Broccoli is one of the most commonly consumed vegetables in the United States (PBH, 2015), with an average fresh broccoli consumption per person of 6.6 pounds in 2015. Due to the high amount of vitamin C, glucosinolates and sulforaphane, broccoli is often associated with strong antioxidant and anticarcinogenic properties (Fimognari & Hrelia, 2007; Vallejo, Tomás-Barberán, & Garcia-Viguera, 2002). The market value and the production of cauliflowers and broccoli are substantial. The market value was 1242 million US$ in 2013 and 1343 million US$ in 2014. In addition, the US production was 1.2 million tons in 2014 and 1.3 million tons in 2016 (FAO, 2018). The annual broccoli production is steadily growing by 4%.

In 2011 and 2015, 88% of the vegetable market was in the fresh segment (Mintel, 2011, 2015). The estimated value in 2015 of the remaining shelf-life stable and frozen segments which comprise 12% of the vegetable market was $6.1 billion. According to the surveys, many buyers' (44%) primary reason of eating vegetables is to reduce the risk of chronic disease. Most of the survey respondents (64%) only eat one to two servings of vegetables per day while FAO dietary guidelines suggest 2.5 servings per day. Because many consumers are pursuing a healthier lifestyle, four out of every five shoppers say that they are concerned about the nutritional content of their food, and this is consistent across all generations (FMI, 2016). In further 2016, value-add fruit, vegetable, and nutritional products (drinks, bars, and supplements) were in the top 10 grocery growth categories taking 27.6% (Nielsen, 2016). The sales of dietary supplements reached a new high of $41.2 billion (NBJ, 2017). The high market value indicates that there is still plenty of opportunity for innovating new vegetable products and increasing vegetable consumption.

Fresh broccoli is perishable. Without proper storage, broccoli will start wilting, turn yellow and develop mushy texture in 2-3 days at 20 °C. After harvest, broccoli is most likely refrigerated or frozen before it reaches consumers. The shelf life of broccoli is between 3-4 weeks at 0°C refrigeration. Broccoli can be stored in modified atmosphere (MA), film wrapped or frozen To extend the shelf life and appearance (Jacobsson, Nielsen, & Sjöholm, 2004). Drying is also an option to preserve broccoli. Dried vegetables are in favor due to consumer desire for healthier diet choices.

Most studies on broccoli focus on the vital nutrients and health benefits of consuming fresh broccoli, especially on anticarcinogenic properties (Barba et al., 2016;

Domínguez-Perles, Martínez-Ballesta, Carvajal, García-Viguera, & Moreno, 2010; Elbarbry & Elrody, 2011; Fimognari & Hrelia, 2007). In particular, glucosinolates and sulforaphane have been greatly investigated due to their strong anticarcinogenic property. Positive results have been shown in many in *vitro* and in *vivo* studies (Domínguez-Perles, Martínez-Ballesta, Carvajal, García-Viguera, & Moreno, 2010; Fimognari & Hrelia, 2007). Other studies have focused on certain types of drying processing conditions, such as microwave drying or tray drying (Mrkìc, Cocci, Rosa, & Sacchetti, 2006; Zhang & Hamauzu, 2004).

The objectives of this study were (a) to evaluate the physical and chemical properties and nutritional quality of dried broccoli with different pre-treatments using continuous vacuum belt dryer, (b) to predict the broccoli paste drying characteristics in vacuum belt drying, (c) to determine if the vacuum belt dryer is plausible to produce high-quality broccoli powder by comparing it to 3 different drying methods, and (d) to discuss the effect of processing conditions on the vitamin C content, total antioxidant activity and sulforaphane content of the final broccoli.

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CHAPTER 2

REVIEW OF LITERATURE

Broccoli

Broccoli (*Brassica oleracea* var. *Italica*), as shown in Figure 2-1, is an annual edible green plant in the *Brassicaceae* family. The broccoli plant, which grows in the first year and then flowers in the second year, has large flower heads which are arranged in a tree branching structure from a thick edible stalk. Leaves surround the mass inflorescence of broccoli. The prevalent hypothesis of the origin of broccoli traces back to Western Europe coasts and the Mediterranean region. The tender shoots and leaves of the wild *Brassica* species which were found by ancient people on the limestone cliffs, and they started to cultivate the plants, selecting for desirable traits (Maggioni, von Bothmer, Poulsen, & Branca, 2010; von Bothmer, Gustafsson, & Snogerup, 1995). Some wild species found on the aforementioned cliffs are taxonomically the same species, as are all the later domestic *Brassica* possessing 2n=18 chromosomes (Maggioni et al., 2010). From the eastern Mediterranean in the 16th century, broccoli was introduced to central and southern Italy where the diversification of var. *Italica* and var. *botrytis* took place (Giles, 1941; Gray, 1982). In the early 20th century, Italian immigrant market gardeners introduced a type of broccoli called calabrese, which is the only vegetable of the *Italica* group that has been extensively developed as a crop, to the eastern United States and cultivated it there. In the later 20th century, the United States calabrese spread back to Europe (Gray, 1982).

Broccoli gradually flowers yellow, and broccoli is harvested shortly before the buds flower. During the distribution chain, the respiration rate of the harvested broccoli is still high in comparison with other vegetables, such as asparagus, spinach, and sweet corn. Therefore, to lower the respiration and retain the consistent green appearance, the harvested broccoli heads must be bundled and cooled or packed with ice to 0°C and be stored under refrigeration immediately. Fresh broccoli is a very perishable vegetable. Even in good storage conditions, leaves may discolor, buds flower yellow and drop off, and the structure soften. Broccoli becomes undesirable after 14 days at 10°C (Rangavajhyala, V.M., & Kadam, 1998).

Calabrese broccoli is grown extensively in North America, China, and India. World-wide production of cauliflower and broccoli was 1.22 million tons in 2014, 1.29 million tons in 2015, and 1.32 million tons in 2016. North America, China, and India account for around 80% of the total cauliflower and broccoli production (FAO, 2018). In the United States, California (95%) produces the highest amount of broccoli for fresh market and processing production, followed by Arizona (5%). The production was 20 million tons for fresh market and 39,200 tons for processing in California, and 1 million ton for fresh market in Arizona in 2015. The value of fresh market vegetables in 2015 was 12 billion dollars, and broccoli accounted for around 8% (1 billion dollars) of that value (USDA, 2017). Broccoli is the 11th most consumed fresh vegetable in the United States with the per person consumption averaging 6.6 pounds in 2015 (AgMRC, 2017; PBH, 2015). Broccoli contains several vital nutrients and bioactive compounds, such as chlorophyll, vitamin C, and glucosinolates, that make broccoli attractive to consumers.

Chlorophyll

Chlorophyll, as shown in Figure 2-2, is a ubiquitous green pigment found in every green plant. This phytochemical is the keystone compound of photosynthesis, which absorbs light and generates energy for the plants. Chlorophyll is composed of tetrapyrroles with a magnesium atom bonded to the center. The porphyrin is esterified to a phytol, an acyclic diterpene alcohol, to form chlorophyll. There are 5 types of chlorophyll. Chlorophyll *a* and *b* are the most prevalent forms in higher plants, although chlorophyll *c*, *d,* and *e* can also be found in many photosynthetic algae, such as red, brown and yellow-green algae. In most green vegetables and fruits, the amount of chlorophyll *a* is higher than the amount of chlorophyll *b* in an approximate ratio of 3 to 1. The amount and distribution of chlorophyll are greatly influenced by species, climate, harvest seasons, post-harvest handling, and the type and the extent of food processing. The degradation of chlorophyll is called pheophytinization which results in the discoloration of vegetable and production of brown derivatives. Chlorophyll is sensitive to extreme pH and temperature. Thermal processing and acidification can produce several distinct brown derivatives such as pheophytins and pyropheophytins. Thermal processing increases the activity of endogenous chlorophyllase which is an enzyme intermediating between pheophytins and chlorophyll, accelerating the degradation of chlorophyll and extricating the magnesium ion (Mg^{2+}).

Regreening is a common rapid practice used to preserve the green color in food and chemical engineering. By reacting divalent metal salts of zinc (Zn^{2+}), copper (Cu^{2+}) or magnesium (Mg^{2+}) with pheophytin or pyropheophytin, chlorophyll is able to form a stable green metallochlorophyll complex. In comparison with natural chlorophyll, the Cu^{2+} and Zn^{2+} complexes are significantly more stable to extreme pH and thermal processing conditions and have been used to preserve green color in canning green beans and in generating green additives. Commercially water-soluble chlorophyll derivatives are often used as food grade color additives in Europe, Asia, and the United States.

Sodium copper chlorophyllin (SCC) is the most common chlorophyll derivative which is synthesized from chlorophyll extract treated with methanolic sodium hydroxide and replaced central Mg^{2+} with Cu^{2+} (Ferruzzi & Blakeslee, 2007). The network of alternating single and double bonds allow chlorophyll *a* and *b* to be very effective photoreceptors. The orbitals can delocalize stabilizing the structure, and the delocalized polyenes are highly absorptive in the visible spectrum, which allows the plants to absorb the sunlight energy (Streitwieser & Heathcock, 1981).

Many studies have examined the potential physiological effects of chlorophyll consumption and the health benefits. Several researchers have studied the effects of chlorophyll derivatives, although most have focused on the anticarcinogenic properties of SCC. Egner et al. (2001) found that chlorophyllin intervention may reduce the bioavailability of ingested aflatoxin B1 in human by chelating with carcinogens and hindering the absorption of aflatoxin B1. The randomized, double-blind, placebo-controlled chemoprevention trial of consuming chlorophyllin showed a 55% reduction in the aflatoxin B1 biomarker of urine samples (Egner et al., 2001). Lai et al. (1980) found a positive correlation between the antimutagenic activity of common vegetable extracts and the chlorophyll content. Chlorophyllin demonstrated higher inhibition of mutagenic activity than chlorophyll (Lai, Butler, & Matney, 1980). A diet high in chlorophyll has been associated with reducing the risk of colon cancer in humans (Balder et al., 2006). However, low doses SCC was found to promote colon carcinogenesis in rats treated with dimethylhydrazine (Nelson, 1992). Besides anticarcinogenic activity, chlorophyll derivatives also have other health benefits, such as antioxidant activity, mutagen trapping, regulation of detoxification pathways and induction of cell apoptosis (Ferruzzi & Blakeslee, 2007).

Vitamin C

Vitamin C, as shown in Figure 2-3, includes L-ascorbic acid and its oxidation product, dehydroascorbic acid. Besides L-ascorbic acid, there are three stereoisomers: D-ascorbic acid, D-isoascorbic acid, and L-isoascorbic acid. However, these stereoisomers do not have similar bioactivity in the human body. Within the human body, ascorbic acid involves in many physiological metabolisms and pathways such as the synthesis of collagen, synthesis of neurotransmitters and hormones, generation vitamin E and serving as a neuromodulator or electron transporter. Ascorbic acid is a strong reducing agent and readily oxidizes reversibly to dehydroascorbic acid. In the human body, it is often used to protect cellular components from free radicals and harmful oxygen-derived compounds. Primates, fruit bats, and guinea pigs are unable to synthesize ascorbic acid as they lack a gene that codes L-gulonolactone oxidase, the last enzyme in synthesis pathway of ascorbic acid. Thus, humans must acquire vitamin C from the diet. An estimated 90% of vitamin C comes from the ordinary diet (Drake & Frei, 2011). Many fruits and vegetables are good sources of vitamin C. Certain vegetables, such as broccoli, tomatoes, and peppers, are rich in vitamin C whereas carrots, lettuce and celery contain relatively low amounts of vitamin C (Drake & Frei, 2011). Due to the difference of genotype, climatic conditions and fertilizer, the content of ascorbic acid in *Brassica* vegetables can vary significantly (Podsędek, 2007)

In the food industry, ascorbic acid is widely used as a food additive to improve taste, prevent oxidation or serve as a stabilizer. It is easy to find ascorbic acid in common food products and beverages such as bread, wine, and frozen fruits. Ascorbic acid is prone to degradation during heating and storage. Preparation procedures and processing conditions should be taken into consideration when evaluating the suitability of processed fruits and vegetables as dietary sources of vitamin C. For example, oil-frying at high temperature might drastically reduce the vitamin C content of foods whereas steaming or boiling is considered to be less detrimental and preserves more vitamin C.

Several studies have shown that fruit and vegetable intake is associated with the reduced risk of some types of cancers. Vitamin C could be protective against cancer development via different mechanisms by acting as an antioxidant, stimulating the immune system, or inhibiting carcinogen formation. Most case-control studies have reported that consuming at least 85-110 mg/day of vitamin C shows protective effects on cancers of the digestive tract, including the mouth, pharynx, esophagus, and stomach (Carr & Frei, 1999). Dietary vitamin C might also be able to inhibit *Helicobacter pylori* which is strongly linked to gastric cancer (Suzuki, Iwasaki, & Hibi, 2009). However, vitamin C is not associated with reducing the risk of lung, breast, prostate and ovarian cancers, nor with cancer-related mortality. Plasma concentration of vitamin C might be a better way to reflect the association between cancer risk and vitamin C plasma concentration. Random control trials (RCT) of vitamin C supplements did not find positive effects on preventing cancers (Drake & Frei, 2011).

Coronary heart disease (CHD) is a major cause of mortality worldwide. An estimated 366,000 people died from CHD in 2015 (CDC, 2017). It is a disease caused by atherosclerosis in the coronary arteries. Many factors have been suggested for the pathogenesis of atherosclerosis, including increased levels of plasma low-density lipoproteins and oxidative stress, decreased levels of plasma high-density lipoproteins, endothelial dysfunction, and vascular inflammation. Vitamin C is a highly effective antioxidant against lipoprotein oxidation and is important in regulating endothelial and vasorelaxation functions. Thus, previous studies have investigated the relationship between vitamin C intake and CHD incidence. A meta-analysis study based on 14 prospective cohort studies concluded that higher dietary intake of vitamin C was associated with reducing the risk of CHD incidence (Ye & Song, 2008). An intake of 100mg/day vitamin C is the threshold for a protective effect on CHD; Levels above the threshold might not result in additional benefit.

Several studies examined the relationship vitamin C and the risk of gout (Choi, Gao, & Curhan, 2009; Gao, Curhan, Forman, Ascherio, & Choi, 2008; Huang et al., 2005). Vitamin C from diet and supplements have been reported to increase urinary excretion and found to have an inverse relation with serum levels of uric acid. Primarily, the intake of vitamin C supplements was found to have a dose-dependent protective effect against gout. An RCT of 184 adults reported that 500mg vitamin C supplements daily for 2 months decreased serum levels of uric acid in comparison with a placebo. From the available studies and data, they showed that increased vitamin C intake might be beneficial in lowering the uric acid in the blood, and therefore, preventing the deposit of urate crystals and gout development.

Oxidative stress in the brain has been strongly implicated in the pathogenesis of both ischemic and hemorrhagic stroke. Cohort studies showed an inverse correlation between vitamin C intake and the risk of ischemic stroke and intracerebral hemorrhage, but not cerebral infraction or subarachnoid hemorrhage. The inverse correlation was found primarily in cigarette smokers who are known for increased oxidative stress and have lower vitamin C status. Although many prospective cohort studies found an inverse correlation between vitamin C intake and stroke, not all found a protective effect. In comparison with dietary intake assessments, plasma or serum levels of vitamin C are a more reliable measure of body vitamin C status. Studies using blood levels of vitamin C also demonstrated the same inverse correlation results. However, the population-based studies showed no protective effect against stroke when taking oral vitamin C supplements (Drake & Frei, 2011). The researchers suggested that plasma vitamin C is a good indicator of fruit and vegetable consumption, but vitamin C itself is not protective against stroke or at least not the only or main component in fruits and vegetables accounting for the beneficial effect on stroke risk. Overall, current studies suggest that a vitamin C rich diet might reduce the risk of stroke.

The idea that vitamin C could be used in preventing and decreasing the severity of the common cold has been known for a few decades. Douglas and colleagues have published three reviews on prevention and treatment of the common cold (Douglas & Hemilä, 2005). Their most recent meta-analysis indicated that there is no significant effect of vitamin C supplements on incidence of the common cold for the typical consumer. However, there is a significant effect on the individuals under cold or physical stress. Individuals who are under physical stress, such as marathon runners, skiers, and soldiers training in the subarctic area, showed a 50% decreased in common cold incidence.

Observational studies have shown that a vitamin C rich diet could prevent several chronic diseases, including heart disease, stroke, and certain cancers. However, RCT trials of vitamin C supplements did not show the same results (Lykkesfeldt & Poulsen, 2010). The inconsistent results suggest that the vitamin C might not by itself provide beneficial effects on preventing chronic diseases. Instead, the components in fruits and vegetables may be effective, or vitamin C and the components have a synergetic effect on health. Overall, there are only a few conclusions on the health benefit of vitamin C due to the experiment design conditions which have several limitations and make the results difficult to interpret.

Glucosinolate

Glucosinolates (GLSs), as shown in Figure 2-4, are secondary metabolites in many plants of the order *Brassicales*, including *Brassicaceae* family (also called *Cruciferae* family), *Caricaceae* family and *Capparidaceae* family, and in some *Euphorbiaceae* family. Broccoli, radish, mustard seed, and caper are examples of *Brassicales* which can produce glucosinolates (Matthäus & Özcan, 2002). GLSs are constituted of glucose linked with thiohydroximate-O-sulfonate and a side chain. The GLSs, based on the side chains, can be categorized into aromatic, heterocyclic, aliphatic and w-methylthioalkyl GLSs. More than 200 different GLSs have been identified. Cruciferous plants use GLSs as a defense system against insects and predators, because of the natural strong pungent smell and taste of GLSs. Myrosinase (EC 3.2.1.147) is an enzyme that hydrolyzes GLSs. GLSs and myrosinase are separated in different intracellular or extracellular compartments. When cruciferous plants are damaged, the myrosinase can interact with GLSs and begins to catalyze and hydrolyze the GLSs into a β-D-glucose and a thiohydroximate-O-sulfonate. However, thiohydroximate-O-sulfonate is unstable. Thiohydroximate-O-sulfonate will spontaneously rearrange itself (Lossen rearrangement), resulting in a sulfate ion and metabolites which depend on the GLS side chain and the hydrolyzing conditions. At acidic pH, the presence of ferrous ions (Fe^{2+}) and a plant protein called epithiospecifier protein (ESP), the thiohydroximate-O-sulfonate tends to form nitriles. In addition, if the GLSs have a terminal unsaturated side chain, a unique form of nitrile called epithionitriles will form. While at neutral pH, the formation of isothiocyanates is favored. Furthermore, if the side chain of the thiohydroximate-O-sulfonate has a β-hydroxy group, the isothiocyanates spontaneously cyclize into oxazolidine-2-thione. Similarly, if the side chain of the thiohydroximate-O-sulfonate has an indolyl-methyl group, the intermediate compound will be unstable and break into thiocyanate ion and indole-3-carbinol (Barba et al., 2016).

Domestic cooking of *Brassica* vegetables can cause different levels of GLSs in the vegetables (Mithen, Dekker, Verkerk, Rabot, & Johnson, 2000). First-order kinetics can model thermal degradation of GLSs. Dekker, Hennig, and Verkerk (2009) reported the stability of GLSs in five Brassica vegetables as follows: gluconapin > glucobrassicin > 4-methoxyglucobrassicin. Similar results in red cabbage were also reported by Oerlemans, Barrett, Suades, Verkerk, & Dekker (2006). Different types of thermal processing can also affect the GLSs content. Several studies have shown that boiling *Brassica* vegetables deactivates myrosinase, but also results in leaching substantial amount of GLSs into the cooking water (Ciska & Kozłowska, 2001; Jiao, Yu, Hankin, Low, & Chung, 1998; Rosa & Heaney, 1993; Vallejo, Tomás-Barberán, & Garcia-Viguera, 2002). Verkerk and Dekker (2004) also pointed out that microwave heating may increase the chemical extractability of GLSs. GLSs are the precursors of isothiocyanates which are also sulfur compounds. Previous studies have suggested that the degradation of products of GLSs possess bactericidal and fungicidal features (Guerrero-Díaz, Lacasa-Martínez, Hernández-Piñera, Martinez-Alarcon, & Lacasa-Plasencia, 2013; Sarwar, Kirkegaard, Wong, & Desmarchelier, 1998; Sotelo, Lema, Soengas, Cartea, & Velasco, 2015).

Glucoraphanin is the most abundant GLS in broccoli. After hydrolysis by myrosinase, the major isothiocyanate is sulforaphane (4-methylsulfinybutyl isothiocyanate) (SF), as shown in Figure 2-5. Much epidemiological evidence indicates SF, found in broccoli, influences several health benefits and biological activities. SF has attracted much attention as it is considered a promising agent for cancer chemoprevention. Cancer chemoprevention is defined as the use of certain drugs or substances to keep cancer from forming, growing, or coming back, via inhibiting carcinogenesis stages (Fimognari & Hrelia, 2007). Many studies reported the correlation between SF consumption and reduction of cancer risk in lung, bladder, breast, prostate, colon and non-Hodgkin lymphoma (Elbarbry & Elrody, 2011). For example, consuming 3 or more servings of cruciferous vegetables per week has an inverse relationship with the risk of prostate cancer, and consuming 5 servings per week has an inverse relationship with the risk of bladder cancer in males (Cohen, Kristal, & Stanford, 2000; Michaud et al., 1999). The mechanism of cancer chemoprevention is complicated and involving both interfering cancer inhibition and inducing detoxification. SF not only inhibits several Phase 1 cytochrome P450 (CYP) enzymes which are responsible for activating procarcinogens to carcinogens but also induces Phase 2 detoxification enzymes, such as glutathione S-transferases. The simplified mechanism is shown in Figure 2-6. Besides cancer chemoprevention, SF is also suggested to have other health benefits, including reducing the risk of diabetes, atherosclerosis, cardiovascular, gastric and neurodegenerative diseases (Elbarbry & Elrody, 2011).

Maltodextrin

Maltodextrin is a term for a mixture of saccharides with a spectrum of different molecular weight distribution between polysaccharides and oligosaccharides. Maltodextrin is a hydrolyzed starch with dextrose equivalent (DE) value smaller than 20. DE value is a measurement of the total reducing power of all sugars present relative to glucose as 100 based on dry weight. Thus, a product with a higher DE has stronger reducing power, meaning the product was hydrolyzed to a greater degree, and also has shorter chain length. For DE higher than 20, corn syrup is used (Chronakis, 1998; Gabas, Telis, Sobral, & Telis-Romero, 2007).

In the preparation of dried fruit and vegetable powders, it is sometimes necessary to add adjutant such as maltodextrin to improve processing procedures and product characteristics. The glass transition temperature (T_g) can be defined as the temperature where a hard-amorphous food system changes from a glassy state to a rubbery state with changes in molecular mobility (Jakubczyk, Ostrowska-Ligeza, & Gondek, 2010). The glass transition temperatures and molecular weights of several monosaccharides, disaccharides, and maltodextrins are shown in Table 2-1(Umesh Hebbar, Rastogi, & Subramanian, 2008).

Maltodextrin can increase the glass transition temperature and prevent stickiness. High molecular weight and low DE maltodextrins are often used to avoid powder stickiness and to promote flowability as they have higher glass transition temperatures (Gabas et al., 2007). Jaya, Das, and Mani (2006) showed that adding maltodextrin and tricalcium phosphate improved the hygroscopicity, degree of caking and flowability of mango powders. Jakubczyk et al. (2010) showed that the hygroscopicity of apple puree powder was reduced by adding maltodextrin.

Due to the properties of maltodextrin, it has been used in the food industry. Some of the maltodextrin functional properties include bulking, gelling, crystallization prevention, binding, adding texture, anticaking, serving as oxygen barriers, freezing control, substituting of fat/oil and promotion of dispensability and solubility (Setser & Racette, 1992). Because of the organoleptic characteristics, maltodextrin is also used to substitute for fat and to develop calorie-reduced products. Most of all, maltodextrin is cheap in comparison with other hydrocolloids. Maltodextrins have also been proven useful in reducing Maillard reactions and in microencapsulation of food components such as fat, vitamins, minerals, and colorants (Chronakis, 1998). DE value is important to the functionality of encapsulation. Microencapsulated materials (core) are covered by coating materials (wall) that protect the core and release it when the condition is right. Higher DE value protected the encapsulated core from oxidation. Several studies have looked up how maltodextrins play in the wall system (Kenyon & Anderson, 1988; Reineccius, 1991; Sheu & Rosenberg, 1995).

Drying

Drying is the oldest method to preserve food. It is a technique to reduce the moisture in the food. Drying methods range from simple sun drying to sophisticated spray drying or freeze drying, but the principle of drying is that the water presented in food is removed by evaporation or sublimation as a result of applying heat. Drying initiates with free water near the surface of the product being removed by gas diffusion to surrounding media. As the surface empties, the capillary forces may draw water up from deeper regions, and the moisture content will equilibrate with the drying media. If the rate of moisture content changed is plotted by time, various drying periods can be seen. The relation of drying rate and moisture content is shown in Figure 2-7. In Phase A-B, the surface of the wet solid comes into equilibrium with the media. This phase is usually short. Phase B-C is the constant drying rate period. In this period, the surface of the solid is saturated with the water as the water evaporates from the surface and the water moves from inside the solid to the solid surface. After point C, the water migration to the surface is not enough to maintain surface saturated, the equilibrium no longer holds and the drying rate decline. The point C is called the critical point. Phase C-E is the falling rate period. From C point, the surface of the solid increase and approach to the drying media. Several falling rate periods may be observed due to the latent heat of vaporization (Brennan, 2012; Kerr, 2007).

The principle reason for drying is to extend the shelf life of food. Drying reduces both the moisture and the water activity of the food. When the water activity is reduced to a certain level, the growth and development of spoilage and pathogenic microorganisms are inhibited. As shown in Figure 2-8, the enzyme activities and chemical reactions such as oxidation rate are also inhibited, which delays the undesirable changes of food (Brennan, 2012). Besides extending the shelf life of food, drying can also minimize the volume of food and the packaging requirement and achieve lower transportation weight and lower cost of storage and transportation (Brennan, 2012; Sacilik, 2007; Zomorodian & Dadashzadeh, 2009). However, drying is a high energy consuming process (Brennan, 2012). Several studies have also addressed some problems on the physical properties change on the conventional convective drying, such as loss of color, flavor and nutrition (Brennan, 2012; Chua, Mujumdar, Chou, Hawlader, & Ho, 2000; Guiné & Barroca, 2012; Krokida, Tsami, & Maroulis, 1998; Sablani, 2006). Especially, temperature sensitive nutrients, such as vitamins C and A, degrade drastically. The texture and the microstructure of the dried foods are often changed due to the heating and moisture loss (Koc, Eren, & Ertekin, 2008). Sinesio, Moneta, Spataro, and Quaglia (1995) found that freeze-dried products received a higher score in juiciness and fruity aroma while air dried products were characterized as tough, cracked and burned flavor. In different drying methods, different drying conditions and techniques would be applied to reduce the operational cost, time and energy, or to increase the yield and the quality of the products. The common methods of drying are, freeze drying, vacuum drying, osmotic drying, fluidized bed drying, microwave drying, and combination drying (George, Cenkowski, & Muir, 2004).

Types of Drying

Osmotic dehydration (OD)

Osmotic dehydration is commonly used to preserve fruits, vegetables and meats. It is usually done by preserving foods in high-sugar or high-salt solution. Water diffuses from the food material tissues into the solution because of the higher osmotic pressure in the hypertonic solution. When it balances, the water in the hypertonic solution and the food should be the same. However, the final product is not completely dried, and the rate of mass transfer during OD is low. The osmotic agent in the food brings the resistance for the mass exchange of water in further dehydration processes such as convective drying and freeze-drying (Khin, Zhou, & Perera, 2005). Several studies applied partial vacuum, high-intensity electrical field pulses, supercritical CO_2, centrifugal force, or ultra-high hydrostatic pressure to improve the mass transfer (Azuara, Garcia, & Beristain, 1996; Knorr, 2003; Rastogi, Eshtiaghi, & Knorr, 1999; Rastogi & Niranjan, 1998; Rastogi & Raghavarao, 2004; Sagar & Kumar, 2010).

Heat pump drying

Conventional drying wastes much energy on heating air, venting a stream of moist and exhausting hot air, which is a significant quantity of energy lost from the process. In order to reduce the energy lost in the conventional drying, heat pumps were introduced to conventional drying system to recover the latent heat of water evaporation in the exhausted hot air. By installing an evaporator of a heat pump in the exhaust, the heat is recycled from cooling down (recovering the sensible heat) and dehumidifying (recovering the latent heat) the exhausting hot air and is added to the refrigerant. The refrigerant is then circulated to the condenser and releases the heat to the entering air, thus raising the temperature. Heat pump drying is suggested to have higher energy efficiency and uses 22 to 40% less energy than the electrical heating in producing dried fruits (Kohayakawa et al. 2004; Gabas et al. 2004). Other characteristics, such as color and rehydration, were more desirable when the heat pump is used under atmospheric conditions or replacing nitrogen and carbon dioxide with air (Hawlader et al. 2006; Alves Filho et al. 2004; Uddin et al. 2004).

Microwave Drying

Microwave energy is nonionizing electromagnetic radiation in the frequency range of 300 MHz to 300 GHz. A magnetron is equipped to ramp up the domestic outlet current frequency of 60 Hz to the most commonly used frequency in microwaves, 2,450 MHz. The microwave energy couples with the material, and the heat is generated within the product through molecular excitation. A temperature gradient occurs where the product center temperature is greater as the material absorbs the microwave energy and forces the moisture out (Gaukel, 2017; Sagar & Kumar, 2010). Because microwaves can only heat the product, it is recommended to combine other techniques, such as vacuum or forced air, to remove the moisture vapor. Several studies have shown that combining another drying method with microwave drying would increase the quality of final products, such as carrots (Jun Wang & Xi, 2005) and banana (Maskan, 2000). The advantages of microwave drying are shorter drying time, quick starting and stopping, improved product quality, heating from the interior of the material and lower energy consumption (Tulasidas et al. 1995a; Sagar and Suresh 2009).

Spray drying

A spray dryer includes a feed pump, atomizer or spray nozzles, air heater, air disperser, drying chamber, powder recovery systems, and process control systems. When the atomized fluid feed comes in contact with the hot air, the moisture evaporates. The properties and the quality of the spray dried product are influenced by the apparatus feed flow rate, particle size, viscosity, spray dryer inlet and outlet temperatures, pressure and type of atomizer (Chegini et al. 2008, Tonon et al. 2008). Spray dried products have low water activity and small volume that are suitable for transportation and storage. The rapid evaporation keeps the liquid feed atomized droplets temperature relatively low, so the product is not significantly affected by the thermal processing. Another advantage of spraying drying is that it can process fluid materials rapidly while providing a relatively good control on the particle size distribution (Obón et al. 2009). However, contacting with high-temperature hot air, thermo-sensitive components, such as vitamins and enzymes, can be damaged or inactivated during the spray drying process (Murugesan & Orsat 2011). Overall, spray drying is a great method to fast dry fluid materials into solid particles whiling maintaining the quality.

Freeze drying

In freeze drying, a food material is rapidly frozen and then subjected to a high vacuum that removes ice in the food material by sublimation. Freeze drying can dry and preserve the food with maximum quality and nutrients due to the absence of water and lower temperatures required for processing. It can be applied to varied shapes of products. Also, the deteriorative and microbiological reactions are stalled during the drying process. Above are the reasons that freeze drying is in favored in the production and is considered one of the best drying methods. For example, freeze drying is often used on high-value foods, such as perishable commodities, nutraceutical foods, baby foods, herbs or coffee, and special end-use foods, as those used in the military. The high rehydration ratio of freeze-dried products makes it excellent for ready-to-eat meals or soups and dietary supplements (Ratti, 2001). However, freeze drying is very energy-consuming and costly. In comparison with air-drying, it costs 4-8 times more. Most of the energy is spent on sublimation which takes about 45% of the entire freeze-drying. The energy cost breakdown for freeze drying is shown in Figure 2-9.

Collapse is another frequent problem when using freeze-drying (Ratti, 2001). In comparison with air-dried food, freeze-dried food has higher porosity and larger surface area which causes higher oxygen transfer and promotes rapid oxidation of lipid and fat-soluble vitamins (Sablani, 2006). Many studies improved freeze-drying by combining with other techniques, such as microwave and desiccants (e.g., silica gel). Microwave energy helps sublimation in the frozen regions to decrease freeze-drying time up to 60-75% (Rosenberg & Bogl, 1987). The desiccant can replace the condenser and achieve a 50% reduction in total costs in comparison with traditional freeze-drying (Bell & Mellor, 1990).

Vacuum Belt Drying

The continuous vacuum belt dryer is an alternative method to dehydrate vegetables and fruits. The material is introduced by a pump attached to a feed hopper that goes through the airlock, which is then transferred to the belt and conveyed over a series of heating plates while remaining under vacuum (Kim & Kerr, 2013). The products of continuous vacuum drying have good quality, in addition to shorter the processing time (Wang, Li, Chen, Bao, & Yang, 2007). In conventional drying, darker color is expected due to the increased product temperature and increased chemical reaction rates. Oxygen is usually the major factor in these deteriorations. Continuous vacuum belt drying has no such problem. Oxidative deterioration is minimized by reducing oxygen exposure throughout the entire drying processing. Vacuum belt drying has been employed to dehydrate fruits, fruit juices and herbs. It is considered a good dehydration method for hygroscopic products, and sticky or pasty foods. Kim and Kerr (2013) successfully produced good quality blueberry powder using vacuum belt dryer. They overcame the stickiness problem, which often happens when drying high sugar fruit juice, by using maltodextrin to improve flowability and hygroscopicity. Yan and Kerr (2013) produced vacuum belt dried apple pomace which had similar color and total phenolics content to freeze-dried pomace and a higher level of monomeric anthocyanins in a shorter period of time. Wang et al. (2007) showed that vacuum belt drying was useful in retaining the volatiles in banana powders. They identified the fractions of volatile compounds in the banana powder and showed that vacuum belt drying could retain more full-bodied and mellow aroma compounds such as eugenol, elemicin and other alkyls, alkenes, and alkynes.

Food Powders

From raw materials like flours and spices to processed products, such as instant coffee and milk powder, food powders are a large part of products found in the food industry. They can be classified either by composition and microstructure or by their particle size, size distribution, functionality, and chemical and physical properties. Food powders vary largely in shape, from extremely irregular (ground spices) to approximate spherical or well-defined crystalline (starch or granulated sugar). Size reduction processes are often used to prepare food powders from the food materials. These processes make food powders rarely in a spherical shape. The pulverized particles are often compressed in length, breadth, and thickness, and resemble polyhedrons with nearly plane faces, in a number of 4 to 7, with sharp edges and corners. Sometimes, they may be plate-like or needle-like. As particles get smaller, the edge may become smoother, and the particles can be considered spherical. Most industrial powders such as clay, chalk, and cement are normally symmetrical with a definite shape such as cubic, hexahedral, etc., because of the intimate structure of their forming elements. In contrast, food powders are mostly organic in origin. The structures of the individual grain shapes can vary largely since their chemical compositions are more complicated than those of inorganic industrial powders (Barbosa-C´anovas, Ortega-Rivas, Juliano, & Yan, 2006). The compressibility, flowability and bulk density of powders are highly dependent on the particle size and its distribution (Barbosa-Canovas et al. 1987). Common convention considers that the median size of a particle material to be considered powder should be less than 1 mm. Median size means that 50% of the particles is larger than the median size of all particles (Barbosa-C´anovas et al., 2006). The terms of the size of powders have been recommended by *British Pharmacopoeia* referring to standard sieves apertures (Table 2-2).

Benefits of Vegetable Powder

Fruits and vegetables are important sources of various essential nutrients, including vitamins, minerals, and dietary fibers. Fresh fruits and vegetables are often high in moisture, which makes them susceptible to bacteria, yeasts, and molds, and easily spoil within a few weeks. Several studies have proposed different methods of postharvest treatments, drying, packaging, genetic engineering to extend the shelf life of vegetables and fruits (Ahvenainen, 1996; Jiang, 2013; Kader, Zagory, Kerbel, & Wang, 1989; Matas, Gapper, Chung, Giovannoni, & Rose, 2009). The most common method of extending shelf life is to reduce moisture and pulverize the material into powders. Minerals, dietary fibers, and vitamins are relatively stable in low moisture foods. The cost of the vegetable powder is lower than fresh produce when considering the volume and storage during transportation. These features make fruit and vegetable powders a useful supplement for people who are unable to obtain certain nutrients through their diet (Jiang, 2013). Vegetable powders have plenty of nutritional and functional properties. Air-dried foods are less prone to lipid oxidation than freeze-dried products due to lower porosity (Sablani, 2006). Vegetable powders with higher content of bioactive compounds may translate to a higher tissue content of bioactive compounds and higher bioactivity *in vivo* and have benefits on health (Liu, Volker, Jeffery, & Erdman Jr, 2009). Vegetable powders are widely used for flavoring and coloring. They are used as baking ingredients, formula, functional granules and tables (Jiang, 2013). More than 20% of the world’s perishable produce is dehydrated to increase the stability and prolong shelf-life (Grabowski S, 2003).

Figure 2-1. Broccoli (*Brassica oleracea* var. *italica*).

(https://en.wikipedia.org/wiki/File:Broccoli_and_cross_section_edit.jpg)

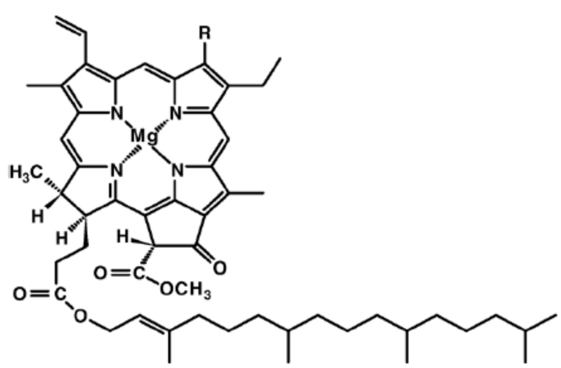

Figure 2-2. Structure of chlorophyll.

(Ferruzzi, M. G., & Blakeslee, J. (2007). Digestion, absorption, and cancer preventative activity of dietary chlorophyll derivatives. *Nutrition Research, 27*(1), 1-12.)

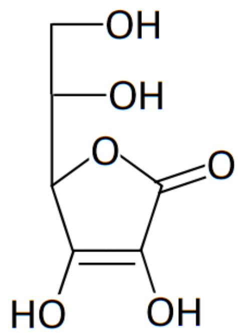

Figure 2-3. Structure of ascorbic acid.

(Drake, V. J., & Frei, B. (2011). Vitamin C in human disease prevention. In W. Herrmann & R. Obeid (Eds.), *Vitamins in the Prevention of Human Diseases* (pp. 347-362). Berlin: Walter de Gruyter.)

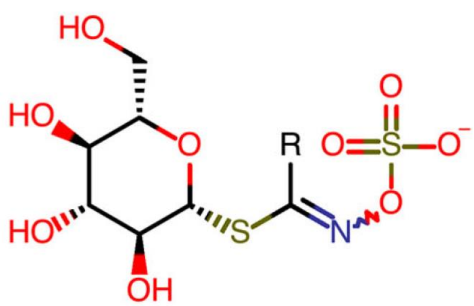

Figure 2-4. Structure of glucosinolate.

(Barba, F. J., Nikmaram, N., Roohinejad, S., Khelfa, A., Zhu, Z., & Koubaa, M. (2016). Bioavailability of glucosinolates and their breakdown products: Impact of processing. *Frontiers in Nutrition, 3:24*.)

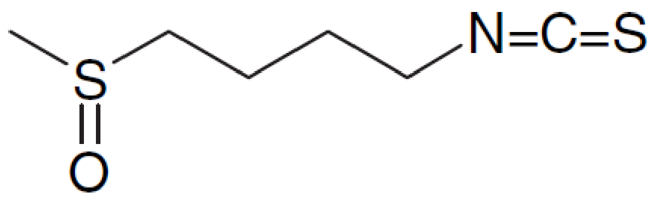

Figure 2-5. Structure of sulforaphane.

(Lim, S., Lee, J., & Kim, J.-K. (2009). Analysis of isothiocyanates in newly generated vegetables, Baemuchae (× *Brassicoraphanus*) as affected by growth. *International Journal of Food Science & Technology, 44*(7), 1401-1407.)

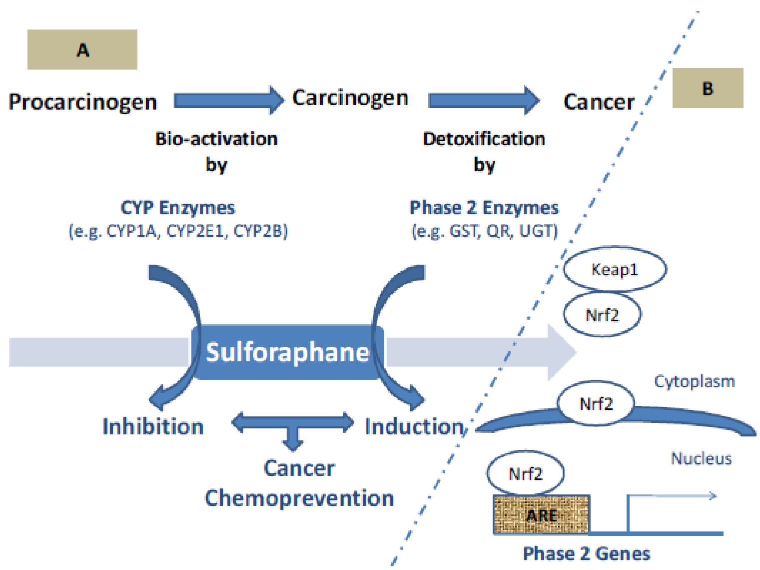

Figure 2-6 The mechanism of sulforaphane induced Phase 2 detoxification.

(Elbarbry, F., & Elrody, N. (2011). Potential health benefits of sulforaphane: a review of the experimental, clinical and epidemiological evidences and underlying mechanisms. *Journal of Medicinal Plants Research, 5*(4), 473-484.)

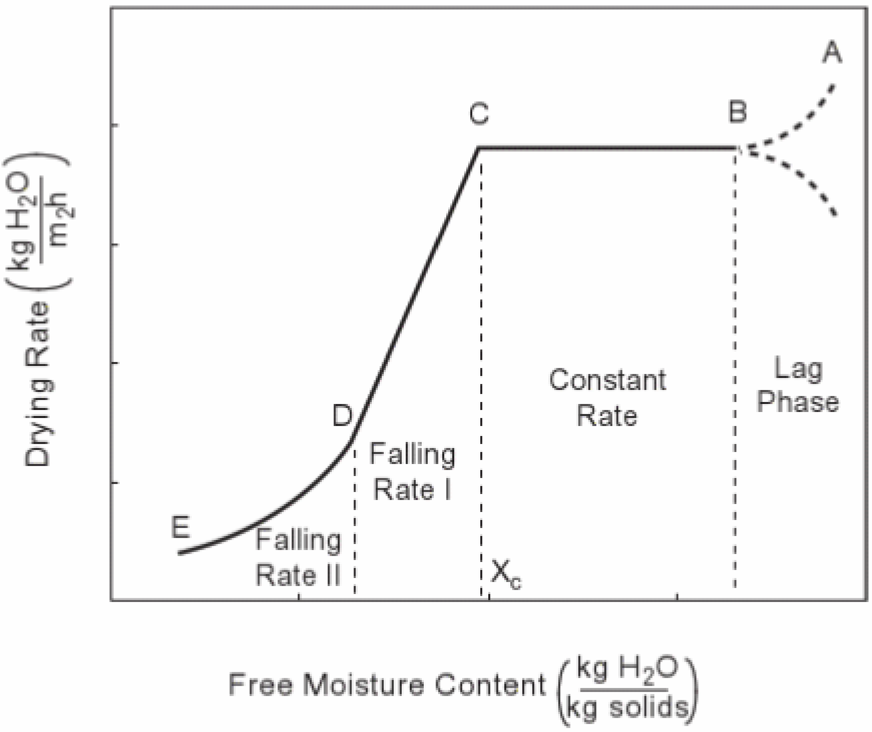

Figure 2-7. The relation of drying rate and moisture content.

(Brennan, J. G. (2012). Evaporation and Dehydration. In J. G. Brennan & A. S. Grandison (Eds.), *Food Processing Handbook* (pp. 77-130). Weinheim: John Wiley & Sons, Inc.)

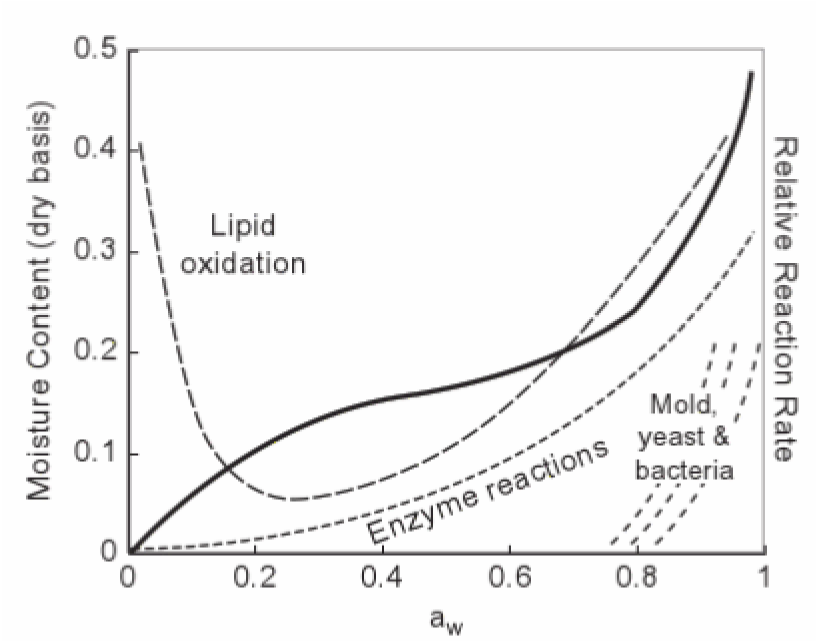

Figure 2-8. Water activity diagram.

(Brennan, J. G. (2012). Evaporation and Dehydration. In J. G. Brennan & A. S. Grandison (Eds.), *Food Processing Handbook* (pp. 77-130). Weinheim: John Wiley & Sons, Inc.)

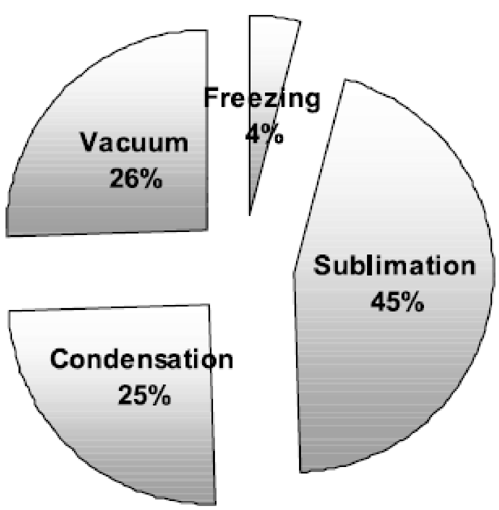

Figure 2-9. Energy cost breakdown for freeze-drying process.

(Ratti, C. (2001). Hot air and freeze-drying of high-value foods: a review. *Journal of Food Engineering, 49*(4), 311-319.)

Table 2-1 Glass transition temperature of sugars and maltodextrins

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

(Umesh Hebbar, H., Rastogi, N.K., & Subramanian, R. (2008). Properties of dried and intermediate moisture honey products: A review. *International Journal of Food Properties, 11*(4), 804-819.)

Table 2-2 Terms recommended by the British Pharmacopeia for use with powders.

	B.S. meshes	
Powder type	All passes	Not more than 40% passes
Coarse	10	44
Moderately coarse	22	60
Moderately fine	44	85
Fine	85	-
Very fine	120	-

(Barbosa-C´anovas, G. V., Ortega-Rivas, E., Juliano, P., & Yan, H. (2006). Particle Properties *Food Powders: Physical Properties, Processing, and Functionality*. New York: Kluwer Academic/Plenum Publishers, (Chapter 2).)

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CHAPTER 3

THE EFFECT OF PROCESS CONDITIONS ON DRYING AND PHYSICAL CHARACTERISTICS OF CONTINUOUS VACUUM BELT DRIED BROCCOLI[1]

Chi, P. and Kerr, L. W. To be submitted to LWT – Food Science and Technology

Abstract

The experiment examined the effects of two levels of maltodextrins (0% and 10% maltodextrin), three levels of temperature (80 °C, 95 °C and 110 °C) and two types of pre-treatments (chemical blanching and steam blanching) on the drying characteristics of vacuum belt dried broccoli. The Wang and Singh model was the best fit among the five drying models with highest adjusted R^2 (above 0.97) and the lowest SSE (below 0.16). The effect of maltodextrin, temperature drying method and pretreatment on final broccoli powder's physical traits were studied. Broccoli powders were found a Type III isotherm behavior using Guggenheim-Anderson-de Boer model. With 10% maltodextrin, the drying efficiency of certain groups were 200% higher and the hygroscopicity was lower. Vacuum belt dried broccoli had lower K value and m_o value than freeze dried broccoli. Blanching with 0.1% $NaHCO_3$ and 0.1% MgO gave the hue angle on average 14.18° greener than steam blanched.

Introduction

Fruits and vegetables are highly seasonal. In the peak season, high production surpasses the market demand and leads to economic losses for the growers. It also results in large quantities of waste. Therefore, many of the preservation methods have been adopted to reduce the waste in order to provide a steady supply in the off-season. Amongst all the traditional preservation methods, drying has been commonly applied to a variety of fruit and vegetables. The extended shelf-life and reduced volume of dried products are favored by the producers (Prakash, Jha, & Datta, 2004).

Hot-air drying is one of the simplest ways to dehydrate food. The main disadvantages are low energy efficiency, low-quality product and long drying time during the falling rate period. Due to the low thermal conductivity of food materials, heat transfer to the inner sections of foods is limited during conventional heating (Jun Wang & Xi, 2005). Freeze drying is a good way to maintain the quality, but it is a slow and energy consuming process. The final product is easily rehydrated because the removal of the ice crystals leaves a beehive-shaped porous structure (Cohen & Yang, 1995). Vacuum belt drying is an alternative dehydration method. It is suitable for solid, liquid, sticky, pasty or hygroscopic products and is a promising method to produce high-quality products with higher flavor retention and color preservation. It also helps to prevent oxygen from participating in lipid oxidation and browning reaction, which results in higher quality products. In addition, the vacuum belt drying process is short and low cost (Kim & Kerr, 2013; Wang, Li, Chen, Bao, & Yang, 2007; Yan & Kerr, 2013)

Blanching is often used to inactive enzymes in fresh vegetables in order to prevent deteriorating appearance, texture and nutritional values. Maltodextrin can provide a few benefits such as increasing drying efficiency and preventing stickiness (Fang & Bhandari, 2012; Jaya, Das, & Mani, 2006). In addition, temperature plays an important role in drying. Higher drying temperature increases the drying process, but it may also lead to undesirable changes in appearance (Lin, Durance, & Scaman, 1998).

To better understand the drying behavior of various foods and to optimize the processing parameters, many theoretical and empirical models have been developed. Studies have been conducted on thin layer drying of several vegetables such as dill and parsley leaves (Doymaz, Tugrul, & Pala, 2006), *Panax Notoginseng* (Liu et al., 2009), and thin broccoli stem (Simal, Rossello, Berna, & Mulet, 1998). However, there is a lack of literature specifically on vacuum dried broccoli paste drying kinetics.

The objective of this study is to determine the better processing conditions to produce higher quality vegetable powder by investigating the physical properties of the broccoli powders. Moisture sorption, isotherm behavior, hygroscopicity, drying rate and color were used to determine the quality of the final product.

Materials & Methods

Sample Preparation

Fresh broccolis (*Brassica oleracea* var. *Italica*) were bought from US Foods (Norcross, GA) and were refrigerated at 4 °C before treatments. Fresh broccolis were cut into 2-3 cm pieces and separated into two groups. The first group was steam-blanched at 100 °C for 5 min and then immediately refrigerated to cool down (to 4 °C). The second group was boiled in a solution of 0.1% (w/v) $NaHCO_3$ and 0.1% (w/v) MgO for 5 min and refrigerated immediately. The two groups were ground by a food processor (Univex, M12B, Univex Corporation, Salem, NH) first, and further ground to a smooth paste by a stone miller (Super Masscolloider, MKCA6-3, MASUKO Sangyo Co., LTD, Saitama-Pref, Japan). Two levels of maltodextrin (GPC B730, Grain Processing Corporation, Muscatine, IA), 0% and 10% maltodextrin, were applied to all groups after the grinding. Every 1 kg of broccoli paste was mixed with the assigned level of maltodextrin by a food processor (Cuisinart, FP-12DCN, Conair Corporation, East Windsor, NJ) for 3 min. All the pretreated samples were stored at 4 °C prior to subsequent drying.

Vacuum Belt Drying

The broccoli paste was dried by a laboratory-scale vacuum belt dryer (Zwag, LKM-101, Zchokke Wartman Ltd. Bucher, Dottingen, Switzerland). The dryer was comprised of a vacuum chamber with a 20.33 cm-wide Teflon-coated conveyor belt that passes over 3 individually controlled heating plates and one cooling plate. Each heating plate and cooling plate was 113 cm-length and 22.9 cm-wide. A radiation plate of 22.9 cm-wide and 85 cm-length 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 the vacuum on the chamber. The pressure was held below 1.3 kPa during the drying processing. An attached touch screen panel and programmable controller were used to monitor and designate the heater plates' temperatures. Two different temperatures (80 °C and 95 °C) were used to dry the broccoli paste. The three heating plates and the radiation plate were set and preheated to the designated temperature. Approximately 300 g broccoli paste were processed in each batch. The drying times for the different temperatures were 150 and 120 min for 80 °C and 95 °C, respectively.

Hot-air drying

An Alto-Shaam CombiTherm (CTP7-20E, Menomonee Falls, WI) was used to hot-air dry the broccoli paste. Approximately 300 g of broccoli paste was dried at 60 °C, 0% humidity with an air flow of 2.25 m/s for 12 hours for each trial based on previous research (Mrkic, Redovnikovic, Jolic, Delonga, & Dragovic-Uzelac, 2010).

Freeze drying

A laboratory scale freeze dryer (RD53S5, Millrock Technology, Kingston, NY) was used to freeze dry the broccoli paste. The freeze dryer temperature, pressure and drying time were controlled by a laptop (Vostro 2420, Dell, Round Rock, TX) and the software (OptiDry. Ink, Millrock Technology, Kingston, NY). The freeze dryer was programmed as followed:

Shelf Temperature (°C)	-40	-10	0	10	20	30
Time (min)	120	120	120	120	600	120
Vacuum (mTorr)	500	100	100	100	100	100

Powder grinding

Dried broccoli samples were immediately placed in a vacuum chamber for 15 min for cooling. Approximately 30 g dried broccoli samples were ground by a Nutribullet (NB-101B, Nutribullet LLC., Los Angeles, CA) for 15 s. Ground samples were separated by No.60, No.70, and No.80 sieves and then sealed in labeled PET/Al pouches (StandUpPouches, ABC Packaging Direct LLC., Westlake, OH). The pouches were stored at -80 °C in an Ultra-Low Temperature Freezer (MDF-U76VC-PA, Panasonic Corporation, Osaka Japan) to prevent moisture migration, oxidation and enzyme reactions.

Moisture content and water activity

The moisture content of the final dried broccoli powder was determined using the Association of Official Analytical Chemists (AOAC) Method 934.06 with some modification. Approximately 1-2 g of broccoli powder samples were weighed in 5 cm aluminum weighing pans. Weighed samples and pans were recorded then dried in a vacuum oven (Model VWR 1430 MS, Optics Planet Inc., Northbrook, Il, USA) at 105 °C and 33 kPa for 8 hours or until a constant weight was reached. This measurement was conducted in duplicate. The moisture content was calculated on both wet basis (M_{wb}) and dry basis (M_{db}).

$$M_{wb} = \frac{w_i - w_f}{w_i} \times 100 \tag{1}$$

$$M_{db} = \frac{w_i - w_f}{w_f} \times 100 \tag{2}$$

where, w_i is the initial weight of the sample, w_f is the final weight of the sample.

Water activity of broccoli powders was determined by an Aqualab water activity meter model Series 3 (Decagon Devices Inc., Pullman, WA) at 22 ± 2 °C. The meter was calibrated with saturated $MgCl_2$ solution (0.33). Water activities were measured in duplicate.

Dynamic hygroscopicity

The dynamic hygroscopicity was conducted following the method reported by Jaya et al. (2006) and Kim and Kerr (2013). A 100 x 15 mm petri dish 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 a constant temperature (23 °C) and relative humidity (75%). The weight of the petri dish was tared. Approximately 1 g of broccoli powder was evenly spread on the petri dish in order to maximize the moisture pick up speed. Due to the moisture adsorption, the increase of the weight was monitored and recorded every 2 min for 24 h by a connected laptop computer (Model CF-74, Panasonic, Japan). The measurement was repeated in duplicate.

The data were fit to the moisture sorption equation:

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

The constant K describes how quickly the sample absorbed moisture, m_e described the steady-state moisture content when exposed to humid environment, m_i described the initial moisture content, and m described the total moisture absorbed.

In addition, the hygroscopicity was calculated according to Jaya and Das (2004):

$$\text{Hygroscopicity (\%)} = \frac{\frac{b}{a} + W_i}{1 + \frac{b}{a}} \tag{4}$$

where, *a* was the initial sample mass (g), *b* was the weight (g) at the steady state, and Wi was the initial wet basis moisture content (%).

Color

The color of broccoli powders was measured by a Chromameter (CR-300, Minolta Co., LTD., Japan). A white tile was used to calibrate the meter. The calibration was set as Y = 93.2, x = 0.3131, and y = 0.3191. The powder samples were placed in a 100x15mm petri dish. The Chroma meter was placed on the powders which covered all the area of the lens. Lightness (L*), chroma (c*) and hue (h°) values were measured and recorded. L* represents the lightness, ranging from 0 (black) to 100 (white). c* represents the color saturation, ranging from 0 to 100 (high saturation), and h° represents the primary color value with associated angles of 0° (red), 90° (yellow), 180° (green), 270° (blue) and

360°(red).

Moisture isotherms

Five saturated salt solutions were prepared to create stable environments with relative humidity (RH) between 11% and 75%. The salt solutions used were LiCl (0.11), CH_3CO_2K (0.22), $MgCl_2$ (0.33), $Mg(NO_3)_2$ (0.52) and NaCl (0.75). Approximately 1.2 g broccoli powder samples were weighed into the small plastic caps designed to fit in Aqualab water activity meter and placed on the rack in desiccators containing saturated salt solution. The sample mass and cap weights were recorded. A slight vacuum was applied to the desiccators to seal the desiccators. After 4 weeks, weights and water activities of broccoli powders were measured. Dry basis moisture (M_{db}) content was calculated by the following equation:

$$M_{db} = \frac{w_f - w_s}{w_s} \tag{5}$$

where, w_f was the total mass of wet samples and w_s was the mass of solids in the original samples. The adsorption isotherms were fit to the Guggenheim-Anderson-de Boer (GAB) equation:

$$M_{db} = \frac{m_o k c a_w}{(1 - k a_w)(1 - k a_w + c k a_w)} \tag{6}$$

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

Drying model

The drying model experiments were conducted in the laboratory-scale vacuum belt dryer (Zwag, LKM-101, Zchokke Wartman Ltd. Bucher, Dottingen, Switzerland). Two different pretreated broccolis were used. Three different temperatures (80 °C, 95 °C, and 110 °C) and two levels of maltodextrin (0% and 10%) were decided based on preliminary experiments. The belt speed was set at 0.5 cm/min. Broccoli paste samples were introduced by a pump (ERFY0 18/2-7, Antriebstechnik Bauknecht, Vienna, Austria) in an interval of 10 min. After 150 min, the vacuum dryer was stopped. The dried broccoli samples were removed from the dryer, and the moisture content was measured according to the modified AOAC method 934.06. The same procedures were repeated twice.

Statistical Analysis

The experimental design consisted of two levels of maltodextrins (0% and 10% maltodextrin), three levels of temperature (80 °C, 95 °C and 110 °C) and two types of pre-treatments (chemical blanching and steam blanching). JMP® Pro 13 (SAS Institute Inc.) and SAS® University Edition (SAS Institute Inc.) were used to analyze the data.

Several drying models were used to fit the data. Two-Way ANOVA was performed at a confidence level of 95% ($p < 0.05$). Tukey's honest significant difference (HSD) was performed to investigate differences between treatments.

Results & Discussion

Moisture content and water activity

The moisture content and the water activity of the samples are shown in Table 3-1Table 3-1. Raw broccoli paste was 99% and 0.99. The pretreatments and 10% MD did not produce differences in the moisture content. Even though the moisture content was similar, freeze-dried samples had a lower water activity than the vacuum dried samples. No interactions were found between the variables.

Modeling of drying characteristics

Drying curves (moisture ratio vs. time) are plotted in Figure 3-1 (a) to Figure 3-1 (d). The data obtained from the experiment were fitted with 5 drying models listed in Table 3-2. The Page model resulted in poor fit among all groups. The Lewis, Henderson and Pabis, Logarithmic, and Wang and Singh model gave similar and better predications than the Page model. The best model to describe continuous vacuum belt drying broccoli paste behavior was the Wang and Singh model which gave the highest adjusted R^2 (above 0.97) and the lowest SSE (below 0.16). The Wang and Singh model value of adjusted R^2, SSE, a and b value of different groups are reported in Table 3-3. The a value is a good indicator of drying efficiency. The lower the value, the faster the sample was dried. As the temperature increased, the efficiency increased, and the a value decreased. Maltodextrin also increased the drying efficiency. With 10% maltodextrin, the drying efficiency of some groups is 200% higher than without maltodextrin. The pretreatments did not affect the drying efficiency.

Dynamic hygroscopicity

The hygroscopicity and related constants are shown in Table 3-1. Broccoli powders from all treatments had less than 5% hygroscopicity. Powders usually absorbed moisture quickly due to larger surface area. A powder with hygroscopicity value less than 10% is considered as good 'non-hygroscopic' powder (Jaya et al., 2006). By Multi-Way ANOVA, the significance of drying temperature, pretreatments and maltodextrin were all relevant. Pretreatments, drying method, and maltodextrin resulted in significant differences of hygroscopicity. With 10% maltodextrin, the hygroscopicity of the broccoli powders was lowered by 0.23%. According to Gabas, Telis, Sobral, & Telis-Romero (2007) and Roos (1993), high molecular weight of maltodextrin can increase the glass transition temperature, decrease hygroscopicity and prevent stickiness in low moisture dried powders. The hygroscopicity constant K was decreased with 10% maltodextrin, except when the samples were freeze-dried. A possible explanation for the freeze-dried samples was that the sublimation left small holes on the samples and led to larger surface area. Thus the hygroscopicity was higher. Kim and Kerr (2013) also reported adding maltodextrin lowered the K values in blueberry powders, suggesting a lower hygroscopicity. Jaya et al. (2006) showed increasing maltodextrin levels could decrease the hygroscopicity of mango powder. Goula and Adamopoulos (2008) also found that the hygroscopicity of spray dried tomato pulp was lower when the inlet air temperature was higher and when the maltodextrin level was greater. This study showed similar results in that adding maltodextrin could reduce hygroscopicity. Steam blanching resulted in 0.46% higher hygroscopicity than blanching in boiling water. Regarding the drying methods, hot-air drying resulted in higher hygroscopicity by 1.62% which was double the hygroscopicity of the vacuum belt dried samples. Interactions were found between pretreatments, drying methods and maltodextrin since One-Way ANOVA did not show significance in pretreatments and maltodextrin.

Moisture isotherms

The moisture isotherm of broccoli powder after different pretreatments is shown in Figure 3-2. The equilibrated moisture content of the broccoli powders increased with water activity. Different physical characteristics were observed at different levels of water activity. After 4 weeks, broccoli powders still showed free-flowing behavior at a_w 0.11. At a_w 0.75, The color of the powders turned brown, the surface turned pale, and showed signs of caking. The oxygen and the light might cause the color change in the environmental chambers. When 10% maltodextrin was added, M_{db} was lower and as was the hygroscopicity results. Samples without maltodextrin absorbed more moisture, and the effect of absorbing moisture was greater at higher water activity. According to the International Union of Pure and Applied Chemistry (IUPAC) classification of adsorption isotherms, the isotherms of broccoli powders were most similar to Type III (Brunauer, 1945). Type III isotherm is described by adsorption on macroporous adsorbents with weak adsorbate (Donohue & Aranovich, 1998). A second or multi-layer adsorbate is present before a complete monolayer is formed. As adsorption proceeds, adsorption is facilitated because the adsorbate interaction with adsorbed layers is greater than the interaction with the adsorbent surface. Other vegetable powders also showed Type III isotherm behavior. Balaswamy, Jyothirmayi, and Rao (2004) reported that dried curry leaf chutney powder had a Type III isotherm. W. Wang and Zhou (2013) also showed that spray-dried soy sauce powder had a Type III. However, spray dried tomato powder had a Type II isotherm (Goula, Karapantsios, Achilias, & Adamopoulos, 2008). At higher water activity (0.75), samples with lower levels of MD absorbed more water. It has been suggested that adding MD could decrease hygroscopicity by reducing the number of active sites that can bind with water. Instead of binding with water, the hydroxyl groups bind with the maltodextrins (Catelam, Trindade, & Romero, 2011).

The results were fitted with GAB model (Table 3-4). In the GAB model, monolayer moisture content (m_o) is often associated with the amount of water that was strongly absorbed to the specific sites on the food surface. It has been used as a critical value to estimate food stability (Da Silva et al., 2013). That is, the higher the m_o, the higher the degradation reaction rates and the lower the food stability is. The k value describes the interactions between the molecules and the adsorbent. The c value is often related to the enthalpy of moisture adsorption. Most of the data fitted the GAB model with $R^2 > 0.99$, except 10% MD SF, 10% MD BF and 10% SH which were above 0.9. Freeze-dried broccoli powders had the higher m_o, perhaps due to the larger surface area of the small pores. Hot-air dried broccoli powder had smaller m_o than the other methods. Smaller m_o

might be due to the long hours of drying and the collapsed structure. Adding 10% maltodextrin decreased m_o in the steam vacuum dried broccoli, and it was similar to the results of others (Gabas et al., 2007; Kim & Kerr, 2013). However, in BV, SF, BF and BH groups, adding maltodextrin did not result in lower m_o. The higher m_o in these groups might indicate that other variables such as drying method and pretreatments had a stronger effect on the m_o. The c value of steam-blanched vacuum dried broccoli increased as the level of MD increased. Gabas et al. (2007); Kim and Kerr (2013) show a similar result on vacuum dried pineapple and blueberry. However, the other groups have found that as the level of MD increased, the c value decreased. With 10% MD, k value decreased in steam blanched vacuum dried groups. Telis and Martínez-Navarrete (2009) also reported that adding MD will slightly decrease the k value in freeze-dried grapefruit juice. The other groups in this study, however, did not align with the decreasing trend.

Color

The results for lightness (L*), chroma (c*), hue (h°) and ΔE of each group are shown in Table 3-5. Broccoli powder of different treatments was shown in Figure 3-3 and Figure 3-4. The color was between yellow-green to green. In general, the L* for the broccoli powders were between 37.94 and 69.1, the c* was between 29.47 and 41.01, and the h° was between 97.15° and 121.15°. The raw broccoli paste which had color values of L* = 54.71, c* = 35.56, and h° = 116.8°. The greenest dried broccoli powder was produced by 0.1% $NaHCO_3$ and 0.1% MgO boiled hot-air dried method had L* = 42.735, c* = 30.975, h° = 121.15. ΔE indicated the difference between broccoli raw and broccoli powders. The lower the ΔE, the closer the color of broccoli powder to raw broccoli. Blanching with 0.1% $NaHCO_3$ and 0.1% MgO and drying at 80 °C vacuum belt drying with 10% maltodextrin resulted in the most similar color to raw broccoli.

Using three-way ANOVA, L* was found to be influenced by the drying method. Freeze drying produced the lightest color among all. The lightness of freeze-dried samples was on average 54% lighter than that of the 80 °C vacuum dried samples. Lin et al. (1998) showed similar findings that freeze-dried carrot slices had higher lightness than vacuum microwave dried and air-dried samples. Due to the white color of maltodextrin, adding 10% maltodextrin resulted in 14% lower L* than those without added maltodextrin. Steam blanched samples were 8% lighter than boiled samples. Brewer, Begum, and Bozeman (1995) also observed that the stem-blanched broccoli was slightly darker than boiled-blanched. Treatment with 0.1% $NaHCO_3$ and 0.1% MgO stabilized the chlorophyll and resulted in a darker color. Interactions were found between pretreatments and drying methods, pretreatments and maltodextrin, and between pretreatments, drying methods and maltodextrin.

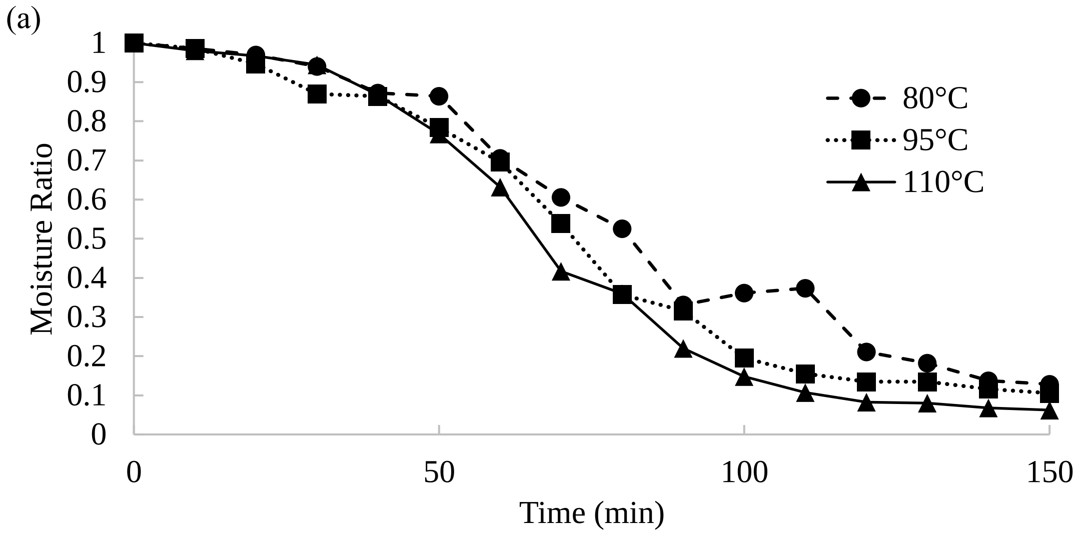
When 10% maltodextrin was added, the chroma was 13.3% more saturated. Steam blanching gave broccoli a more saturated chroma than blanching by boiling. Drying methods led to significant differences, where freeze drying gave the broccoli powder 18% more saturation than 80 °C vacuum drying. Similar results were found by Wojdyło, Figiel, and Oszmiański (2009), where the freeze-dried strawberries had higher chroma than vacuum dried, conventional dried and microwave dried samples. Interactions were found between pretreatments and drying methods, and drying methods and maltodextrin.

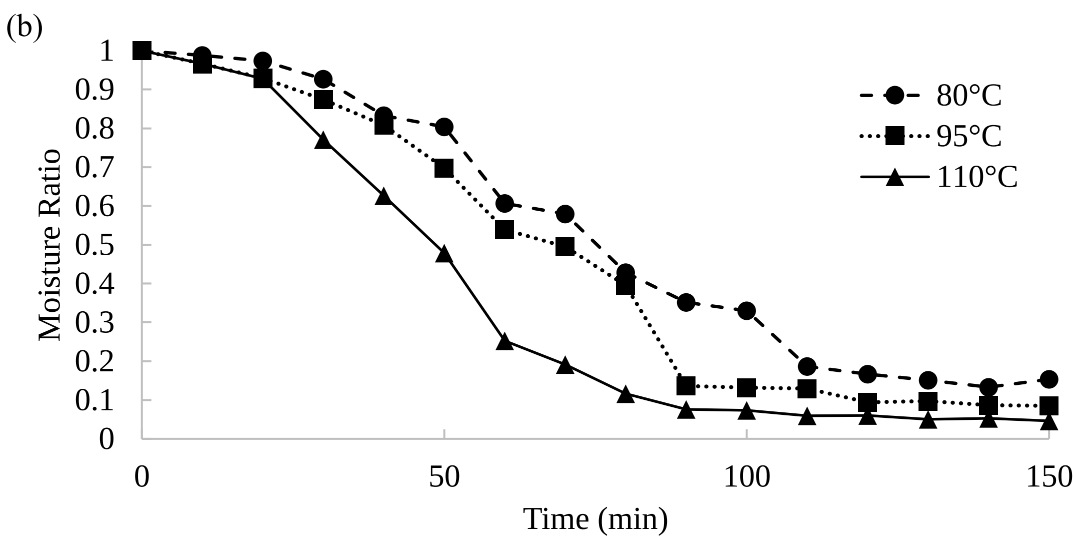
Color has a major influence on consumer selection of produce, as quality is often judged by color (Francis & Clydesdale, 1975). The amount of chlorophyll contributed to how green the broccoli powder was. Blanching broccoli with 0.1% $NaHCO_3$ and 0.1% MgO resulted in the hue angle of the dried powder 14.18° greener than that which was steam-blanched. It showed that the Mg_2^+ in alkaline condition could stabilize the chlorophyll in the blanching process. Koca, Karadeniz, and Burdurlu (2007) showed that the kinetic parameters for chlorophyll *a* and *b* degradation in green peas were lower in higher pH, indicating it was in more stable alkaline environments. Adding 10% maltodextrin resulted in 4.48° decreasing in h° (less green) in samples than without maltodextrin. The hue angle change might be due to the dilution of chlorophyll when

10% maltodextrin was added to the broccoli paste. Broccoli vacuum dried of 80 °C had a similar hue angle to freeze-dried broccoli. The h° of 95 °C vacuum drying was lower than the h° of 80 °C vacuum drying and was similar to hot-air drying. The color change agrees with the study of Van Loey et al. (1998), that showed that the degradation rate of chlorophyll was higher when the temperature was higher. Higher oxygen during drying processing might also degrade chlorophyll in the broccoli as well. Interactions were found between pretreatments and drying methods, and drying methods and maltodextrin.

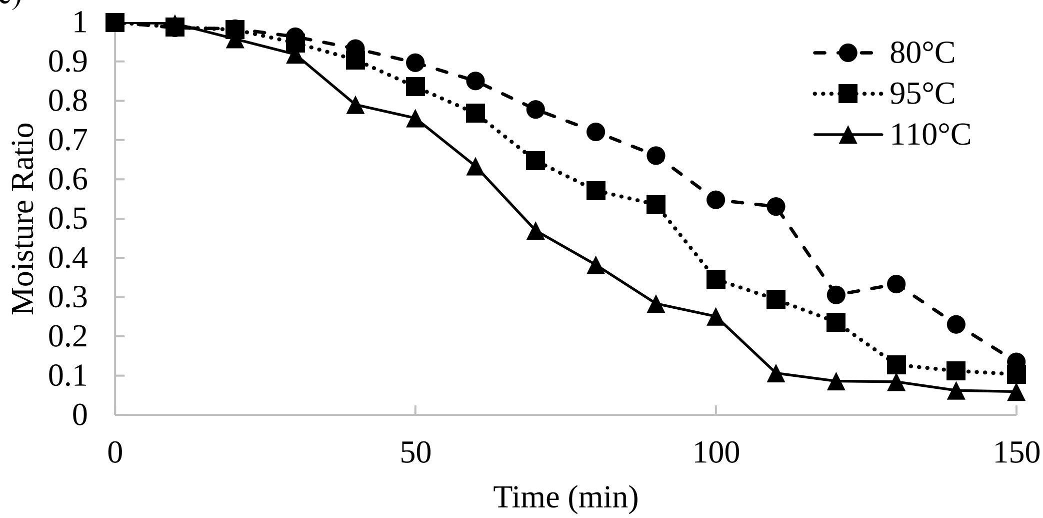
Conclusion

High quality of vacuum belt dried broccoli powders can be produced within 2 hours. Pre-treatment with 0.1% $NaHCO_3$ and 0.1% MgO can preserve the greenness of the broccoli. Freeze drying could result in lighter color products. The drying kinetics of broccoli paste best fitted the Wang and Singh model. Adding 10% maltodextrin doubled the drying rate. Hot-air drying increased the hygroscopicity of the broccoli powder. The broccoli powders isotherms were most similar to Type III isotherms. Freeze-dried product had higher monolayer water content which means lower stability and needs more preservation methods than vacuum dried broccoli powder.


(a)
Moisture Ratio
Time (min)
80°C
95°C
110°C
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1
0
50
100
150


(b)
Moisture Ratio
Time (min)
80°C
95°C
110°C
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1
0
50
100
150

(c)



(d)

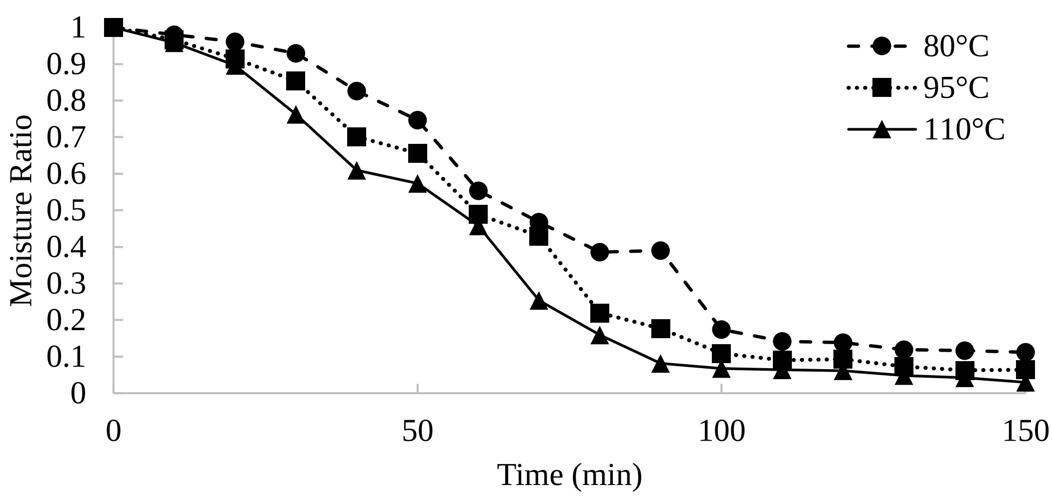


Figure 3-1 Drying kinetics of broccoli paste under vacuum belt dryer.

(a) steam blanched, 0% MD (b) steam blanched, 10% MD (c) Boiling blanched, 0% MD

(d) Boiling blanched, 10% MD.

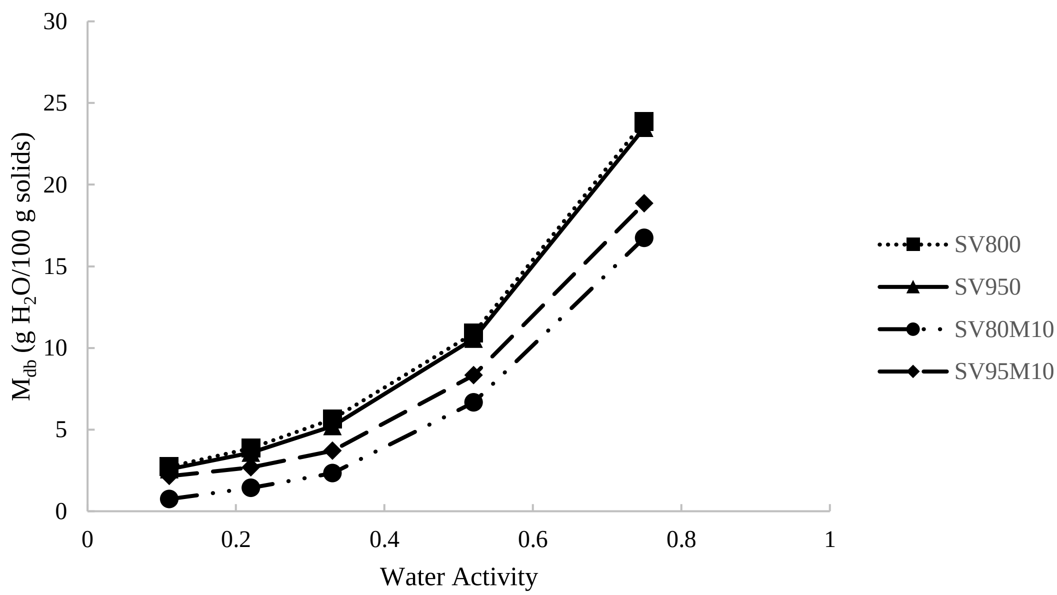


Figure 3-2 Moisture isotherm of broccoli powders at different conditions.

(S – steam blanched, 80 and 95 – drying temperatures (°C), V – vacuum belt dried, and M10 – 10% maltodextrin)

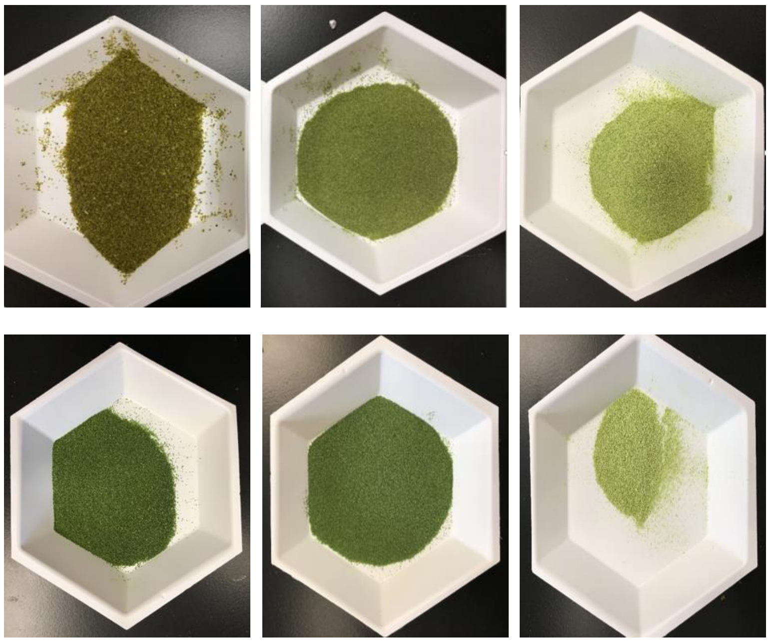


Figure 3-3 Broccoli powder of different drying method and pretreatment.

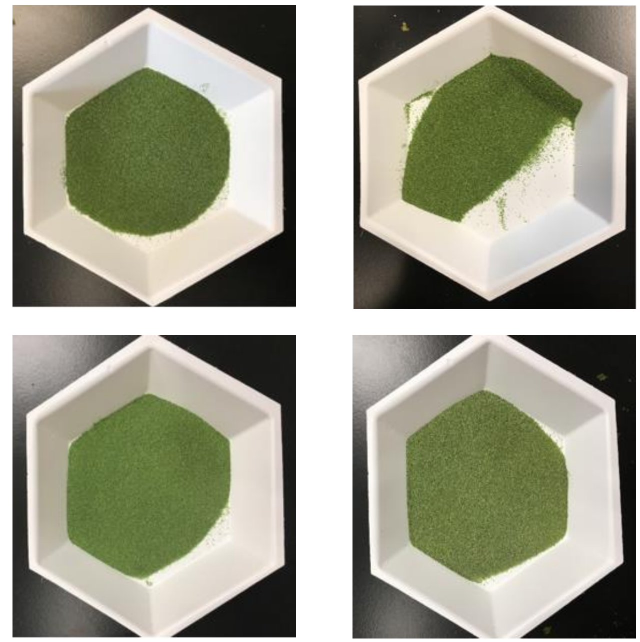


Figure 3-4 Broccoli powder of two level of maltodextrin and drying temperature.

Table 3-1 Moisture content, water activity and hygroscopicity constant of each treatment

Sample	**Moisture content (%)**	**Water activity**	**Hygroscopicity (%)**	**Hygroscopicity constant**	
				K (min^{-1})	R^2
Vacuum dried					
SV80 [1]	3.95 ± 1.83^{a}	0.42 ± 0.04^{abcd}	2.37^{d}	0.0157	0.9768
SV80M10	1.94 ± 0.73^{a}	0.41 ± 0.04^{abcd}	1.45^{g}	0.0098	0.9675
BV80	2.90 ± 0.15^{a}	0.40 ± 0.02^{abcd}	1.90^{ef}	0.0192	0.9936
BV80M10	2.22 ± 0.19^{a}	0.37 ± 0.01^{abcd}	1.59^{fg}	0.0070	0.9598
SV95	3.80 ± 1.44^{a}	0.44 ± 0.07^{abcd}	2.30^{d}	0.0098	0.9993
SV95M10	2.22 ± 0.16^{a}	0.39 ± 0.01^{abcd}	1.58^{fg}	0.0084	0.9677
BV95	3.49 ± 1.22^{a}	0.42 ± 0.01^{abcd}	2.20^{de}	0.0135	0.9954
BV95M10	2.11 ± 0.14^{a}	$0.40+0.02^{abcd}$	1.53^{g}	0.0058	0.9969
Freeze dried					
SF	3.45 ± 0.48^{a}	$0.17+0.07^{d}$	2.15^{de}	0.0370	0.9971
SFM10	3.78 ± 0.16^{a}	0.19 ± 0.11^{cd}	2.32^{d}	0.0392	0.9463
BF	3.43 ± 0.11^{a}	0.26 ± 0.21^{bcd}	2.15^{de}	0.0528	0.9963
BFM10	3.91 ± 0.40^{a}	0.25 ± 0.14^{bcd}	2.39^{d}	0.0645	0.9961
Hot-air dried					

SH	7.96 ± 3.83^{a}	0.48 ± 0.02^{abc}	4.28^{a}	0.0656	0.9898
SHM10	7.65 ± 2.62^{a}	0.61 ± 0.01^{a}	4.28^{a}	0.0043	0.9913
BH	5.45 ± 2.69^{a}	0.44 ± 0.01^{abcd}	3.03^{c}	0.0284	0.9975
BHM10	5.99 ± 2.48^{a}	0.52 ± 0.003^{ab}	3.46^{b}	0.0102	0.9892

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

[1] (S – steam blanched, B – boiling blanched, 80 and 95 – drying temperatures (°C), V – vacuum belt dried, F – freeze dried, H – hot-air dried, and M10 – 10% maltodextrin)

Table 3-2. Five common drying models.

Model	Equations
Lewis	$\mathrm{MR}^{*} = \exp(-\mathrm{kt})$
Page	$\mathrm{MR} = \exp(-kt^{n})$
Henderson and Pabis	$\mathrm{MR} = \mathrm{a}\exp(-\mathrm{kt})$
Logarithmic	$\mathrm{MR} = \mathrm{a}\exp(-\mathrm{kt}) + \mathrm{c}$
Wang and Singh	$\mathrm{MR} = 1 + \mathrm{at} + \mathrm{b}t^{2}$

* (MR – moisture ratio, t – minute)

Table 3-3. Statistical analysis for Wang and Singh model and the predicted constants.

Drying method	**Maltodextrin level**	**a (min^{-1})**	**b (min^{-2})**	**Adjusted R^2**	**SSE**
S80 [1]	0%	-0.0045	-1.00E-05	0.9886	0.0776
S80	10%	-0.0062	-1.94E-06	0.9855	0.0908
S95	0%	-0.0067	-2.52E-07	0.9797	0.1218
S95	10%	-0.0085	1.20E-05	0.9763	0.1279
S110	0%	-0.0074	2.00E-06	0.9710	0.1693
S110	10%	-0.0139	4.90E-05	0.9733	0.109
B80	0%	-0.0123	3.70E-05	0.9807	0.0823
B80	10%	-0.0074	4.83E-06	0.9809	0.1114
B95	0%	-0.0032	-2.00E-05	0.9928	0.0513
B95	10%	-0.0101	2.20E-05	0.9801	0.097
B110	0%	-0.0070	-1.33E-07	0.9830	0.0985
B110	10%	-0.0123	3.70E-05	0.9807	0.0823

[1] (S – steam blanched, B – boiling blanched, 80, 95 and 110 – drying temperatures (°C))

Table 3-4. Parameters of GAB model moisture isotherms.

Sample	MD level	k [2]	c	m_o	R^2	SSE
SV80 [1]	0%	1.125	0.166	89.9	0.9988	1.834
SV80	10%	0.760	4.608	0.9	0.9992	0.521
SV95	0%	1.078	0.253	55.6	0.9985	2.133
SV95	10%	1.028	0.394	26.6	0.9970	2.756
BV80	0%	0.964	0.558	21.7	0.9994	0.763
BV80	10%	1.108	0.212	45.0	0.9976	1.553
BV95	0%	0.814	1.057	13.0	0.9954	5.873
BV95	10%	1.042	0.416	20.5	0.9977	1.617
SF	0%	1.221	0.063	267.3	0.9942	10.279
SF	10%	1.294	0.018	654.2	0.9331	100.300
BF	0%	1.035	0.137	157.4	0.9937	11.232
BF	10%	1.293	0.020	554.5	0.9010	147.000
SH	0%	0.621	9.498	0.5	0.9965	3.506
SH	10%	0.823	8.239	0.2	0.9656	12.288
BH	0%	0.872	1.673	4.0	0.9890	8.964

BH	10%	0.988	1.202	3.2	0.9951	1.811

[1] (S – steam blanched, B – boiling blanched, 80 and 95 – drying temperatures (°C), V – vacuum belt dried, F – freeze dried, H – hot-air dried)

[2] (k, c and m_o were parameter in GAB model.)

Table 3-5. Color parameters of vacuum belt dried broccoli powders.

Sample	**MD level**	**L**	**c**	**h**	**ΔE**
SV80 [1]	0%	47.13 ± 0.83^{cd}	35.12 ± 0.24^{bcd}	106.8 ± 0.25^{e}	12.59 ± 0.69
SV80	10%	47.61 ± 0.18^{c}	38.97 ± 0.1^{ab}	103.8 ± 0.04^{f}	15.22 ± 0.03
SV95	0%	42.75 ± 1.10^{def}	31.26 ± 0.22^{de}	99.9 ± 0.21^{gh}	21.15 ± 0.83
SV95	10%	46.59 ± 0.56^{cd}	37.24 ± 0.85^{abc}	97.2 ± 0.07^{i}	21.29 ± 0.35
BV80	0%	38.88 ± 0.22^{fg}	31.23 ± 0.44^{de}	120.8 ± 0^{a}	16.88 ± 0.09
BV80	10%	45.78 ± 0.91^{cde}	38.23 ± 0.66^{ab}	118.7 ± 0.21^{ab}	9.52 ± 0.71
BV95	0%	37.94 ± 0.15^{g}	29.47 ± 0.05^{e}	119.1 ± 0.04^{ab}	17.99 ± 0.12
BV95	10%	42.69 ± 0.79^{def}	36.72 ± 0.12^{abc}	116.3 ± 0.07^{c}	12.09 ± 0.77
SF	0%	67.71 ± 0.03^{b}	38.31 ± 0.01^{ab}	111.9 ± 0^{d}	14.16 ± 0.03
SF	10%	66.12 ± 0.88^{b}	37.79 ± 1.66^{abc}	110.9 ± 0.67^{d}	13.12 ± 0.18
BF	0%	69.1 ± 3.66^{ab}	40.09 ± 3.88^{a}	115.4 ± 1.84^{c}	15.57 ± 2.42
BF	10%	73.6 ± 0.34^{a}	36.83 ± 1.52^{abc}	115.8 ± 1.24^{c}	19.01 ± 0.17
SH	0%	45.96 ± 0.03^{cde}	33.03 ± 0.01^{cde}	101.5 ± 0^{fg}	17.85 ± 0.01
SH	10%	49.89 ± 0^{c}	39.44 ± 0^{ab}	98.6 ± 0.04^{hi}	19.20 ± 0.03
BH	0%	42.03 ± 1.00^{efg}	31.75 ± 1.09^{de}	121.0 ± 0.28^{a}	13.91 ± 0.53

BH	10%	46.71±1.09^{cd}	41.01±0.27^{a}	117.1±0.04^{bc}	9.70±1.05

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

[1] (S – steam blanched, B – boiling blanched, 80 and 95 – drying temperatures (°C), V – vacuum belt dried, F – freeze dried, and H – hot-air dried)

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CHAPTER 4

THE EFFECT OF PROCESS CONDITIONS ON THE NUTRITIONAL COMPOSITION OF BROCCOLI POWDERS

Chi, P. and Kerr, L. W. To be submitted to LWT – Food Science and Technology

Abstract

Fruit and vegetable powders are important food ingredients that can contribute to the flavor, color, and nutrient of a product. The quality of dried powders depends on the drying method and processing condition. Continuous vacuum belt drying is a viable alternative for producing powders while preserving the nutrients and bioactive compounds. Vitamin C content was two-fold higher in vacuum dried and freeze-dried broccoli powders than hot-air dried powders. Blanching with 0.1% $NaHCO_3$ and 0.1% MgO preserved the vitamin C content in broccoli powder, and it had a positive effect when the drying method was vacuum belt drying. Steam blanching preserved 12.5% more antioxidant activity than blanching in boiling water. Hot-air drying reduced total antioxidant by 31.5% in the sample in comparison with the vacuum drying. Due to extensive drying time and vacuum, sulforaphane was volatilized in the freeze dryer resulting in the lowest amount in freeze-dried broccoli powders.

Introduction

Broccoli is one of the commonly consumed fresh vegetables and is full of many different health-promoting compounds such as vitamins, flavonoids, other phenolic compounds, carotenoids, glucosinolates, and minerals. Studies have shown that frequent consumption of cruciferous vegetables such as broccoli, Brussels sprouts, cauliflower, and cabbage can reduce the risk of certain types of cancer (Prochaska, Santamaria, & Talalay, 1992).

Vitamin C is an important water-soluble nutrient and antioxidant found in fruits and vegetables. Humans are not able to synthesize vitamin C due to lack of gulonolactone oxidase which is needed for vitamin C synthesis. In the human body, vitamin C is often used to protect cellular components from free radicals and harmful oxygen-derived compounds. It not only prevents diseases like scurvy but also provides other health benefits such as lowering uric acid in the blood or reducing the risk of coronary heart disease. Due to the importance of vitamin C for human nutrition, it is often used as an indicator of changes during food processing (Drake & Frei, 2011; Frikke-Schmidt, Tveden-Nyborg, & Lykkesfeldt, 2011; Santos & Silva, 2008).

Glucosinolates are a group of organic compounds that contain sulfur and nitrogen. They are responsible for pungent odor and bitter taste and are used as a defense mechanism to predators in cruciferous vegetables (Cartea & Velasco, 2008; Traka & Mithen, 2009). Due to the potential for chemoprevention, glucosinolates and the breakdown products have gained many scientific interest. Sulforaphane is produced from glucoraphanin when broccoli cells are disrupted. Several studies reported that sulforaphane could induce apoptosis and inhibit cancer development by inhibiting several Phase 1 cytochrome P450 enzymes and inducing Phase 2 detoxification enzymes (Elbarbry & Elrody, 2011; Gamet-Payrastre et al., 2000; Gerhäuser et al., 1997).

Many vegetables are cooked by boiling, microwave or steam before consuming. Cooking often affects the physical characteristics and nutrients of the food. Some nutrients are sensitive to heat, light, and oxygen and are degraded during processing (Santos & Silva, 2008). It has been reported that baking and boiling cause less reduction of ascorbic acid, total phenolics, lycopene and antioxidant activity of tomatoes while frying significantly reduces nutrients (Sahlin, Savage, & Lister, 2004). Zhang and Hamauzu (2004) showed that ascorbic acid, carotenoids, and antioxidant activity were affected by both the cooking method and the cooking time.

The objective of this study was to evaluate the processing factors and to compare nutritional quality of broccoli powders prepared by different drying methods. Vitamin C, total antioxidant activity, and sulforaphane were used as indicators due to their protective benefits on the human health.

Materials & Methods

Sample Preparation

Fresh broccolis (*Brassica oleracea* var. *Italica*) were bought from US Foods (Norcross, GA) and were refrigerated at 4 °C before treatments. Fresh broccolis were cut into 2-3 cm pieces and separated into two groups. The first group was steam-blanched at 100 °C for 5 min and then immediately refrigerated to cool down (to 4 °C). The second group was boiled in a solution of 0.1% (w/v) $NaHCO_3$ and 0.1% (w/v) MgO for 5 min and refrigerated immediately. The two groups were ground by a food processor (Univex, M12B, Univex Corporation, Salem, NH) first, and further ground to a smooth paste by a stone miller (Super Masscolloider, MKCA6-3, MASUKO Sangyo Co., LTD, Saitama-Pref, Japan). Two levels of maltodextrin (GPC B730, Grain Processing Corporation, Muscatine, IA), 0% and 10% maltodextrin, were applied to all groups after the grinding. Every 1 kg of broccoli paste was mixed with the assigned level of maltodextrin by a food processor (Cuisinart, FP-12DCN, Conair Corporation, East Windsor, NJ) for 3 min. All the pretreated samples were stored at 4 °C prior to subsequent drying.

Vacuum Belt Drying

The broccoli paste was dried by a laboratory-scale vacuum belt dryer (Zwag, LKM-101, Zchokke Wartman Ltd. Bucher, Dottingen, Switzerland). The dryer was comprised of a vacuum chamber with a 20.33 cm-wide Teflon-coated conveyor belt that passes over 3 individually controlled heating plates and one cooling plate. Each heating plate and cooling plate was 113 cm-length and 22.9 cm-wide. A radiation plate of 22.9 cm-wide and 85 cm-length 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 the vacuum on the chamber. The pressure was held below 1.3 kPa during the drying processing. An attached touch screen panel and programmable controller were used to monitor and designate the heater plates' temperatures. Two different temperatures (80 °C and 95 °C) were used to dry the broccoli paste. The three heating plates and the radiation plate were set and preheated to the designated temperature. Approximately 300 g broccoli paste were processed in each batch. The drying times for the different temperatures were 150 and 120 min for 80 °C and 95 °C, respectively.

Hot-air drying

An Alto-Shaam CombiTherm (CTP7-20E, Menomonee Falls, WI) was used to hot-air dry the broccoli paste. Approximately 300 g of broccoli paste was dried at 60 °C, 0% humidity with an air flow of 2.25 m/s for 12 hours for each trial based on previous research (Mrkic, Redovnikovic, Jolic, Delonga, & Dragovic-Uzelac, 2010).

Freeze drying

A laboratory scale freeze dryer (RD53S5, Millrock Technology, Kingston, NY) was used to freeze dry the broccoli paste. The freeze dryer temperature, pressure and drying time were controlled by a laptop (Vostro 2420, Dell, Round Rock, TX) and the software (OptiDry. Ink, Millrock Technology, Kingston, NY). The freeze dryer was programmed as followed:

Shelf Temperature (°C)	-40	-10	0	10	20	30
Time (min)	120	120	120	120	600	120
Vacuum (mTorr)	500	100	100	100	100	100

Powder grinding

Dried broccoli samples were immediately placed in a vacuum chamber for 15 min for cooling. Approximately 30 g dried broccoli samples were ground by a Nutribullet (NB-101B, Nutribullet LLC., Los Angeles, CA) for 15 s. Ground samples were separated by No.60, No.70, and No.80 sieves and then sealed in labeled PET/Al pouches (StandUpPouches, ABC Packaging Direct LLC., Westlake, OH). The pouches were stored at -80 °C in an Ultra-Low Temperature Freezer (MDF-U76VC-PA, Panasonic Corporation, Osaka Japan) to prevent moisture migration, oxidation and enzyme reactions.

Vitamin C Analysis

Vitamin C analysis was conducted using the Association of Official Analytical Chemists (AOAC) Method 967.21 with slight modifications. Extraction solvents, ascorbic acid standard solution, and indophenol standard solution were prepared according to the method. Three 2 ml ascorbic acid standard solutions were transferred to each of three 50 ml Erlenmeyer flasks containing 5 ml HPO_3-CH_3COOH solution. Indophenol standard solution was used to titrate the ascorbic acid standard using a 50 ml burette until a light rose pink color lasted over 5 s. Similarly, three blanks containing 7 ml HPO_3-CH_3COOH solution and an equal volume of H_2O of the average volume of indophenol standard solution used in the direct titrate were titrated. After subtracting the average blanks from the standards, the concentration of indophenol standard solution was calculated and expressed as mg ascorbic acid equivalent/ml. Broccoli powder samples were sieved through No.80 were used. Ten ml HPO_3-CH_3COOH-H_2SO_4 solution were added to the 1g of broccoli powder samples to extract vitamin C. The mixture was vortexed for 10s and was centrifuged at 4000 rpm for 5 min. Five ml HPO_3-CH_3COOH solution was added to 2 ml extracted sample and were titrated with indophenol standard solution. The ascorbic acid content was determined by the equation:

$$\text{mg ascorbic acid/g sample} = (X-B)^{*} (F/E)^{*}(V/Y) \qquad (1)$$

where, X is the average ml of sample titration, B is the average ml of sample blank titration, F is the mg ascorbic acid equivalent to 1.0 ml indophenol standard solution, E is the sample mass., V is the volume of initial assay solution, and Y is the volume sample aliquot titrated.

Total Antioxidant Activity

Total antioxidant activity was determined according to the method of Zhang and Hamauzu (2004) with some modifications. One g of broccoli powder was homogenized with 10 ml of 80% methanol by 15s vortexing. The homogenate was added with 10 ml of 80% methanol followed by vortexing for another two successive extractions. A total of

30 ml homogenate was centrifuged at 4000 rpm for 10 min. The supernatant of the methanol extract was collected and diluted to various concentrations (60%, 50%, 40%, 30%, and 20%) for measurement of total antioxidant activity. After preliminary studies, 50% broccoli extract was chosen as an appropriate concentration for accessing antioxidant activity in the samples. A 0.1 mM solution of DPPH in 80% methanol was prepared, and 4 ml of this solution was mixed with 0.2 ml of the diluted extract. A control was mixed with 0.2 ml of distilled water instead of the diluted extract. The mixture was left to stand at room temperature for 60 min incubation. The absorbance at 517 nm was measured. Standard curves were constructed based on 4 different concentration of Trolox (0.01, 0.02, 0.03 and 0.04 mg Trolox /ml). The DPPH scavenging activities of broccoli extracts were reported as mg Trolox equivalent (TE)/100 g dry weight basis. Each sample was measured in triplicate (Brand-Williams, Cuvelier, & Berset, 1995; Zhang & Hamauzu, 2004).

Sulforaphane Analysis

The sample preparation and analysis of sulforaphane were conducted following the method reported by Bertelli, Plessi, Braghiroli, & Monzani (1998) and Lim, Lee, & Kim (2009) with some modification. Three g of broccoli powder samples were extracted 3 times with 10 ml methylene chloride each time at room temperature (~20 °C) to extract and purify the sulforaphane. The methylene chloride extract was filtered with Grade 1 Whatman filter paper and evaporated with a rotary vapor, and then re-dissolved with 3 ml methylene chloride. The crude broccoli extracts were purified by solid phase extraction (SPE) using Bakerbond SPE silica gel (SiOH) 6 ml disposable columns that containing 500 mg sorbent weight. Before using the SPE silica gel, the silica gel cartridge was conditioned with 3 ml methylene chloride. Three ml re-dissolved crude extracts were loaded onto the silica cartridge. The silica cartridge was washed with 3 ml ethyl acetate which were then discarded. The sulforaphane was eluted with 3 ml methanol for sulforaphane analysis. The analysis of sulforaphane was conducted on an Agilent 1260 high-performance liquid chromatography (HPLC) system using a Sedex 85 evaporative light scattering detector (ELSD) and a 4mm x 250 mm, 5 μm particle size, Ultrasphere C18 reverse-phase analytical column. An isocratic mobile phase system was used to separate sulforaphane. The mobile phase consisted of water, acetonitrile, and tert-butanol (8:1:1) containing 5 mM ammonium acetate. The column temperature was set at 40 °C, and the flow rate was 0.8 mL/min. The ELSD drift tube was set at 50 °C using N_2 as a nebulizer gas at 3.0 bar. The gain was set at 8.

Statistical Analysis

The experimental design consisted of three drying methods, including vacuum belt drying, freeze drying and hot-air drying, and two types of pre-treatments (chemical blanching and steam blanching). In vacuum belt drying, two levels of temperature (80 °C, 95 °C) were applied. JMP® Pro 13 (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 between treatments.

Results & Discussion

Vitamin C

The vitamin C content of each sample is reported in Table 4-1. The data in this study indicated that broccoli boiled with 0.1% $NaHCO_3$ and 0.1% MgO had a 48.6% higher content of vitamin C than steamed broccoli. It has been shown that blanching procedures can result in significant losses of vitamin C (Fennema, 1977). Domestic boiling considerably affects the content of vitamin C in the broccoli (Zhang & Hamauzu, 2004). Vitamin C is a hydrophilic compound. When broccoli is boiled in water, the water-soluble vitamin will leak out from the tissue and result in lower vitamin C content in the end. Several studies showed boiling blanching results in losses of vitamin C. Yadav, and

Sehgal (1995) reported losses of ascorbic acid in spinach and amaranth during cooking procedures such as boiling, stewing, frying, blanching and pressure cooking. Gliszczyńska-Świgło et al. (2006) showed that the loss of vitamin C in broccoli upon water-cooking for 5 min was approximately two-fold higher. However, it has to be considered that the stability of vitamin C is related to the storage conditions and the composition of the aqueous solutions or the foods (Santos & Silva, 2008). Most of the studies used water as a blanching medium. Although broccoli was boiled in water, the added $NaHCO_3$ resulted in alkali conditions in the blanching step and resulted in higher retention of vitamin C in the sample. A similar result was found in green leafy vegetables blanched in 0.1% $NaHCO_3$ and 0.1% MgO in that they had higher retention in vitamin C than blanching in water (Gupta, Lakshmi, & Prakash, 2008). As a pretreatment, immersing in alkali solution was better at preserving vitamin C in prunes (Santos & Silva, 2008).

Drying at 80 °C and 95 °C in the vacuum belt dryer did not produce significant differences in vitamin C content related to temperature. The vitamin C content in vacuum-dried and freeze-dried samples was two-fold greater than the vitamin C content in hot-air dried samples. Depending on variables such as pH, temperature, light, the presence of enzymes, oxygen, and metallic catalyzers, vitamin C can be easily degraded

(Johnston, Steinberg, & Rucker, 2001). Although vacuum dried broccoli was processed at a higher temperature than freeze-dried broccoli, the two methods resulted in similar vitamin C content. Thus, the drying temperature might not be the factor causing the difference among the drying methods. The main difference between the 3 drying methods is the presence of oxygen in the hot-air drying. However, with oxygen present, vitamin C oxidation rate increased as the drying temperature increased. The high temperature, high oxygen condition significantly deteriorated vitamin C content in samples during the process. Due to low oxygen, vacuum dried samples did not have high vitamin C oxidation rate even though the drying temperature was higher than used for hot-air drying. Using Two-Way ANOVA, an interaction between pretreatments and drying method was found. When broccoli was steamed, freeze drying and hot-air drying tended to retain more vitamin C than vacuum belt drying. When broccoli was blanched in 0.1% $NaHCO_3$ and 0.1% MgO, vacuum belt drying retained more vitamin C than the other two drying methods.

Total antioxidant activity

Water-soluble compounds such as vitamin C and phenolics play an important role in determining total antioxidant activity. The total antioxidant activity of the sample was expressed as Trolox equivalent per 100 g dry weight (Table 4-1). Steamed broccoli preserved 11.1% total antioxidant than broccoli boiled with 0.1% $NaHCO_3$ and 0.1% MgO. A similar outcome was reported by Gliszczyńska-Świgło et al. (2006). When broccoli was cooked in boiling water, the antioxidants slowly leached from the cutting surface to the cooking water. Only 28.1% of total phenolics were retained. Other antioxidants, such as carotenoids, remained unchanged after 5 min of steaming (Khachik et al., 1992).

The data also indicated that the total antioxidant activity of dried broccoli powder was affected by the drying method. Although freeze-drying, or vacuum belt drying did not produce significant differences, nor did the 15 °C difference in vacuum belt drying, hot-air drying reduced total antioxidant by 38.2% in the sample in comparison with the 95 °C vacuum drying. The fact that vacuum belt drying and freeze-drying produced similar results illustrated that when processing was under vacuum, short-time high-temperature drying could achieve the same quality broccoli powder as long-time low-temperature drying. The amount of oxygen during drying process might be a major factor determining how much antioxidant remained in the sample. In hot-air drying, the antioxidants in broccoli paste were constantly exposed to oxygen until the sample was dried. Freeze drying and vacuum belt drying, on the other hand, dried broccoli under vacuum and preserved the antioxidants even if the processing temperature was higher. Using Two-Way ANOVA, the interaction between drying method and pre-treatments was not found. Based on the study, steamed broccoli and freeze drying or vacuum drying were a better way to preserve antioxidants.

Sulforaphane

Sulforaphane content of each sample was expressed in mg per g dry powder (Table 4-1). Blanching method did not affect the amount of sulforaphane in the samples. In the vacuum belt dryer, drying at 80 °C resulted in 20.7% higher Sulforaphane than drying at 95 °C, indicating that drying at higher temperature degraded the sulforaphane. Sulforaphane was not sensitive to oxygen during the drying process, because the hot-air drying did not result in lower sulforaphane content. Freeze drying caused the greatest decrease in sulforaphane among the three drying method. According to experimental data, the boiling point of sulforaphane is 368.2 °C at 760 mmHg, and the vapor pressure is 0.8 mmHg at 25 °C (ChemSpider, 2018). Although freeze-drying occurs at a lower temperature, the extensive time of drying, low pressure, and sublimation may volatilize the sulforaphane. Unfortunately, no current studies addressed the effect of freeze drying on sulforaphane have been published. By Two-Way ANOVA, no interaction was found between pretreatments and drying method.

Conclusion

Broccoli powder can be produced by vacuum belt drying, freeze drying, and hot-air drying. The hot-air drying significantly reduced the vitamin C content and total antioxidant activity compared to vacuum drying or freeze drying. The freeze-drying significantly reduced the sulforaphane content. Pretreatment affected vitamin C content and total antioxidant activity, but not the sulforaphane content. In vacuum belt drying, the temperature was not a significant factor because the drying time was short. This study showed that vacuum belt drying could produce good quality broccoli powder within 2 hours. Future study will focus on the investigation of the cost-effectiveness of producing the vacuum dried broccoli powder.

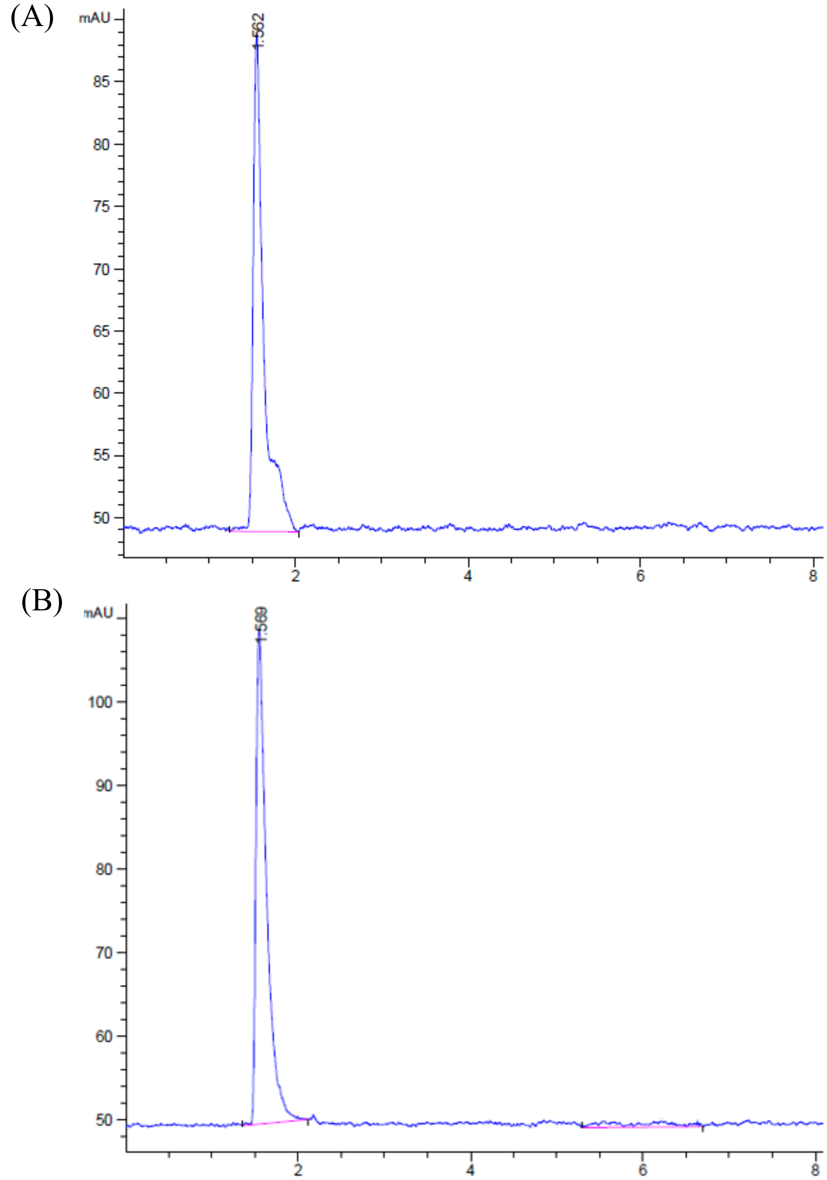

Figure 4-1 High-performance liquid chromatography chromatograms of sulforaphane standard and broccoli sample using evaporative light scattering detector (A: sulforaphane standard; B: sulforaphane in broccoli powder).

Table 4-1. The content of vitamin C, total antioxidant activity and sulforaphane in broccoli powders.

	Vitamin C (mg/g dry powder)	**Total antioxidant activity (TE/100g dry powder)**	**Sulforaphane (mg/g dry powder)**
SV80	1.730±0.017^{c}	140.991±33.157^{ab}	13.661±3.184^{a}
BV80	3.032±0.157^{a}	145.659±20.739^{ab}	12.976±0.679^{ab}
SV95	1.303±0.067^{d}	161.898±40.110^{a}	10.968±1.106^{bc}
BV95	2.517±0.102^{b}	155.501±15.093^{ab}	10.994±0.429^{bc}
SF	2.039±0.157^{c}	172.810±8.692^{a}	9.980±1.223^{c}
BF	2.105±0.122^{c}	146.896±5.421^{ab}	11.184±0.897^{bc}
SH	0.927±0.023^{d}	117.753±18.206^{bc}	11.636±0.679^{abc}
BH	1.264±0.012^{d}	79.089±16.920^{c}	12.531±0.732^{ab}

(SV80: steam-blanched vacuum dried at 80 °C, BV80: boil-blanched vacuum dried at 80 °C, SV95: steam-blanched vacuum dried at 95 °C, BV95: boil-blanched vacuum dried at 95 °C, SF: steam-blanched freeze dried, BF: boil-blanched freeze dried, SH: steam-blanched hot-air dried, BH: boil-blanched hot-air dried.)

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CHAPTER 5

SUMMARY AND CONCLUSION

Vacuum belt drying is an alternative method to dehydrate foods and produce high-quality products in a shorter period. Broccoli powder was successfully produced using vacuum belt drying. In modeling the drying characteristics of broccoli paste, the Wang and Singh model was the best fit among the five drying models with highest adjusted R^2 (above 0.97) and the lowest SSE (below 0.16). Broccoli powders were found a Type III isotherm behavior using Guggenheim-Anderson-de Boer (GAB) model. Groups with 10% maltodextrin had 200% higher drying efficiency than groups without maltodextrin, and the hygroscopicity was lower than groups without maltodextrin. Vacuum belt dried broccoli had lower K value and m_o value than freeze-dried broccoli. Blanching with 0.1% $NaHCO_3$ and 0.1% MgO gave the hue angle on average 14.18° greener than steam-blanched.

On the nutritional quality aspect, vitamin C content was two-fold higher in vacuum drying and freeze-drying than hot-air drying. Blanching with 0.1% $NaHCO_3$ and 0.1% MgO can preserve the vitamin C content in broccoli powder, and it had a positive effect when drying method was vacuum belt drying. Steam blanched preserved 12.5% more antioxidants than boiling blanched. Hot-air drying reduced total antioxidant by 31.5% in the sample in comparison with the vacuum drying. Due to extensive drying time and vacuum, sulforaphane was volatilized in the freeze dryer resulting in the lowest amount.

It can be concluded that the best conditions to produce high quality broccoli powder are blanched with 0.1% $NaHCO_3$ and 0.1% MgO using vacuum-belt drying at 80 °C. Broccoli paste processed at this condition has higher nutritional quality while maintaining color and the other physical characteristics. Future studies can investigate the glucosinolates content and sensory appearance of the broccoli powder.