

EVALUATION OF DRYING TECHNOLOGIES FOR MUSCADINE POMACE TO
PRODUCE AN ANTIOXIDANT RICH FUNCTIONAL FOOD INGREDIENT

by

TRIPTI VASHISTH

(Under the Direction of Ronald B. Pegg and Rakesh K. Singh)

ABSTRACT

Vacuum belt drying (VBD), freeze drying (FD) and hot air drying (HA) for dehydration of muscadine pomace were evaluated. The criteria of comparison were total phenolic content (TPC), antioxidant activity (AA), water activity and moisture content. The effect of sample thickness on the drying time was also investigated. The pomace discs of two thicknesses (4 ± 0.5 and 2 ± 0.3 mm) were dried for 16 time-temperature combinations for VBD (60 and 90 min; 60-120°C), 12 time-temperature and air velocity combinations for air drying (180 and 240 min; 70 and 80°C; 0.2, 0.4 and 0.6 m/s) and one for freeze drying. In lyophilized samples, TPC and AA were found to be 582.52 and 607.53 $\mu\text{molGAE/g}$ extract and 2.21 and 2.29 $\text{mmol Fe}^{2+}\text{eq/g}$ extract for 2 ± 0.3 and 4 ± 0.5 mm thickness, respectively. TPC and AA of TV1, TV2, and TH2 for 2 mm thickness and TV9, TV1, TV8, TV7, TV3, and TV15 for 4 mm thickness were found to be not significantly different ($p > 0.05$) from lyophilized samples. These results indicate that VBD is a promising drying technique.

INDEX WORDS: Muscadine pomace, vacuum belt drying, freeze drying, hot air drying, total phenolic content, antioxidant activity.

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DEDICATION

I would like to dedicate all my work to my parents Mr. OP Sharma and Mrs. Urmila Sharma, my husband Mr. Vijendra Sharma and my whole family. Without their love and support, I would never have been able to accomplish this.

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CHAPTER 1

INTRODUCTION

In 2004, 150 million Americans spent over \$20.5 billion on dietary supplements, which is twice the amount spent in 1994 (Burdock and others 2006). Consumers are becoming more vigilant about the food they eat, as they have become more aware of the relationship between diet and disease; therefore, opportunities are emerging for nutraceutical products derived from fruits and vegetables. This leads to tremendous development and extension of the nutraceutical market.

Grape (genus *Vitis*) and grape products had an economic value of approximately \$3 billion in US (NASS, 2004). During wine or juice making from grapes, high quantities of by-products are generated which are primarily used as feed for animals due to their high fiber content (Palma and Taylor, 1999). By-products left after juice pressing or wine production comprise mainly seeds and skin. Though not technically a grape Muscadine berries belong to *Vitis* family, and their biological name is *Vitis rotundifolia*. Muscadines are an integral part of southeastern agriculture in the United States. They are well suited to the warm and humid climate. Muscadines are used for wine making, juice production, making of jams and jellies and are also consumed fresh. The berries are known to have a very thick skin and a large seed as compared to other grape types and this, therefore, creates a big problem for by-product disposal and utilization. Recently, muscadines have gained increased research attention due to findings which demonstrate that the berries are a rich source of phytochemicals, which might assist in prevention of many diseases such as coronary heart disease, cardiovascular disease, and cancer

(Yi and others 2005; Banini and others 2006). Pastrana-Bonilla (2003) reported that most of the phenolics in muscadines are present in the seeds and skin, and on the contrary, the pulp possesses very low contents of phenolics. The seeds of muscadines are high in antioxidant activity, this mainly comes from the high concentration of (+)-catechin, (-)-epicatechin and the gallic acid. Major phenolics present in muscadine skin are ellagic acid (a dimer of gallic acid), and the aglycones of myricetin, kaempferol, quercetin. Resveratrol has also been found, but in low concentrations (Pastrana-Bonilla 2003). Thus, muscadine skin and seeds have become highly desirable for the production of phytochemical-rich and hence antioxidant-rich nutraceuticals or dietary supplements, which may help in curing a number of chronic disease states (Yilmaz and Toledo, 2004). Utilization of these by-products will not only be good in terms of ecology but also add to the value of the fruit, and can be used as a cheap source of high-quality polyphenolic compounds (Yilmaz and Toledo, 2004; Ector and others 1996). Pomace, which is skin, seeds, and pulp left after juice/wine production, can also be used as a food ingredient providing antioxidant activity to the food system and thereby can be used to replace existing synthetic antioxidants such as butylated hydroxyanisole (BHA) and butylated hydroxytoluene (BHT), which may cause undesirable effects on human health (Jayaprakasha and others 2003). Shrikhande (2000) reported on the significant number of dietary supplements present in the market derived/produced from grape pomace. A number of patents have been issued for grape – related preparations or processes to meet the need of the dietary supplements industry to produce antioxidant-rich nutraceuticals or food ingredients.

Fresh muscadine pomace is highly perishable due to high water activity and therefore, dehydration is a necessary processing technique for increasing its shelf-life for further application and as a food ingredient. A commonly used drying method for high nutrient value-

added products is vacuum freeze drying. During freeze drying, moisture is removed by sublimation from the frozen product and therefore minimum deterioration occurs to the structure and texture of the product. There is minimal loss of nutrients during freeze drying due to the fact that the exposure temperature for the product is very low, usually from 30-40°C. The main disadvantage of freeze drying is the requirement of longer drying times to achieve a desired moisture content, which contributes to the high operating cost. Hot air drying, on the other hand, is a conventional and inexpensive drying method, which involves exposure of the food product to high velocity hot air; the moisture evaporates with the convection current. With air drying, dehydrated product often achieves an extended shelf-life, but the quality and nutrient value of the product deteriorates due to exposure to high temperatures. Furthermore, the oxidation of the food product occurs due to continuous exposure to air for a long time. Therefore, a combination of two processes like vacuum and exposure to moderate temperatures can serve the purpose of reducing the time of operation to achieve desired moisture content and also due to the vacuum, mass transfer takes place at a faster rate. Hence, there will be minimal loss of nutrients due to shorter exposure to high temperatures and prevention of oxidation as the drying takes place under vacuum. As well as, due to short drying time, the operating cost will be reduced as compared to that of freeze drying. Vacuum belt drying is a continuous process unlike freeze drying and the product can be dried at a much faster rate. Due to presence of four different heating zone temperatures, the vacuum belt drier can be set according to desired moisture content; therefore, exposure to high or low temperatures can be set according to the rate of moisture evaporation from the product.

There exists a wide amount of diversity in the types of the grape seed extracts or skin extracts on the basis of total phenolics content. The variation is mainly due to the cultivar selection, the

processing method and handling. Usually products with highly purified phenolic compounds are processed by methods that are expensive, less flexible and low yielding (Shrikhande, 2000). There is a good amount of literature present on antioxidant values, and of extraction methods for polyphenolic compounds of winery by-products, but few articles discuss the drying of muscadine pomace and the effect of different types of drying on the quality of nutraceuticals produced. Existing literature primarily discusses the separation of seeds and skin, given that, making use of seeds and skin separately, requires their separation from the pomace and then extraction of polyphenolics resulting in increased cost of the product. Therefore, the objectives of this research were the following:

1. To compare the efficacy of different time-temperature combinations for vacuum belt drying, and time-temperature as well as air velocity combinations for air drying of muscadine pomace at retaining of polyphenolic compounds with antioxidant activity.
2. To evaluate the impact of vacuum belt dried, hot air dried and lyophilized muscadine pomace samples on the water activity (a_w), moisture content (MC), total phenolics content (TPC), and antioxidant activity (AA).
3. To investigate the impact of pomace disc thickness during FD, VBD, and HAD on a_w , MC, TPC, and AA.

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CHAPTER 2

REVIEW OF LITERATURE

Muscadine

Grapes, with an annual production of 65 million metric tons, are considered as the world's largest temperate fruit crop. The grape (genus *Vitis*) and grape products have an economic value of approximately \$3 billion in the US (NASS, 2004). Grapes are taxonomically classified under the order of Ramnales, the family of *Vitaceae* and the genus of *Vitis* (Vine and others, 2002). Furthermore, the genus *Vitis* is divided into two subgenera *Euvitis*, the true grapes with origins in Asia and Europe, and the *Muscadinia*, muscadine grapes that originated in the southern US and Mexico. Muscadines are biologically quite different from other grapes, as *Muscadinia* have 20 chromosome pairs whereas the *Euvitis* have only 19 (Yi and others 2005).

Muscadines have been extensively cultivated in the southeast US since the 16th century. The scientific name of muscadine is *Vitis rotundifolia*. Other common names include Bullace, Scuppernong, and Southern Fox. Georgia is one of the largest producers of muscadines covering 1200 acres annually, and growing. In fact, there are over 300 cultivars of muscadines grown in the southeastern US. Muscadines are widely used in the production of wines, juices, beverages, dietary supplements, jams, jellies and raisins. Approximately 80-85% of muscadines are processed for juices, jams, jellies, and wine while the remainder of the crop is marketed as fresh (Ector and others 1996).

Muscadines are well suited to grow in the warm, humid conditions of the southeast, and have vigorous, deciduous vines that grow 18-30 m in the wild. They have a tight, non-shedding bark, warty shoots and unbranched tendrils. The color of fruits varies from greenish bronze to

purple, to almost black. Muscadine grapes need a growing season of about 100 days on the vine for the fruit to mature (Olien, 2001). Muscadines grow best on fertile, sandy loams and alluvial soils. The fruits have thick and tough skins and are round shape. The fruit is formed in loose clusters of 3 to 40 grapes. They are around 1 inch in diameter and contain 5 hard, oblong seeds. Unlike traditional grape varieties, muscadines are harvested as individual berries that ripen over an extended harvest period. Some of the widely-grown varieties of muscadine include Black Beauty, Black Fry, Darlene, Fry, Higgins, Jumbo, Scuppernong, Sugargate, Summit, Magnolia, Fry Seedless, Noble, Redgate, Regale, and Sterling.

Muscadines are an excellent source of fiber. Ector (2001) reported that the fiber contents of both bronze-skinned and purple-skinned muscadines are greater than most other fruits, and almost 3 times higher than other members of *Vinifera* family. It has been proven by many researches over the years that fiber-rich foods help in the prevention of constipation, hemorrhoids, and diverticular disease. Fibers are also reported for reducing the incidence of certain types of cancer and may also help in improving ones diabetes states (AACC, 2001).

Muscadine juice

The United States of America is the largest user of grape juice and grape juice concentrates. Muscadine juice has a unique flavor and bouquet. Its composition is almost similar to that of the whole muscadine except that the fiber (predominantly in the skins) and oil (in seeds) have been removed (Bates and others 2001). Sugar level (mainly glucose and fructose), acid contents, flavor constituents such as methyl anthralite and other volatiles, tannins and color substances contribute to the quality of the juice. The color of muscadine juice is largely due to the presence anthocyanin pigments located near the skin. Muscadine grapes yield about 492 l of juice per ton whereas other grape varieties tend to yield 681 l/ton (Ahmedullah, 1989).

By-product utilization

The processing of plant based foods results in the production of by-products, many of which are rich in bioactive compounds like phenolic compounds (Schieber and others 2001). The skin, seed, and pulp remaining after the processing of fruit for juice or wine is called the pomace (Meyer and others 1997). In 2005, between 56-80 thousand metric tons of pomace was generated as a waste in the US alone. Thus, by-product generated creates a lot of problems in terms of storage and transformation, or elimination with respect to ecological and economical aspects (Alonso and others 2002). The disposal of by-products, cost of drying, storage, and shipment of pomace are economically-limiting factors. Therefore, efficient, inexpensive, and ecologically-suitable utilization of these by-products is becoming more important (Lowe and Buckmaster, 1995). By-products of plant food processing can be drastically reduced through new or modified processing methods or reused with an aim at converting these wastes into bio-fuels, food ingredients, and other value-added bioproducts (Makris and others 2007). Several studies have been conducted to evaluate industrial by-products as a potential source of antioxidants. The antioxidant potentials of by-products obtained after the processing of citrus fruits, apples artichokes, cauliflower, carrot, celery, and onion have been studied. The skin and outer layer of many fruits have been found to contain a higher amount of phenolics than the edible fleshy part. For example, the peels from apples, peaches, and pears were reported to contain twice as many of total phenolics as that found in the peeled fruit (Gorinstein and others 2002). Soong and Barlow (2004) reported that the total phenolics content of seeds from several fruits; *i.e.*, mango, avocado, and jackfruit, were higher than that of the edible flesh; these seeds can serve as a good source of antioxidants.

Consumers are becoming aware of the health benefits associated with diet and especially of the high nutritious value of fruits and vegetables. Still, a large number of population lacks an appropriate intake of fruits and vegetables; therefore, dietary supplements, food fortification, and nutraceuticals can solve the problem by consumption of plant components that may have health benefits. Studies have shown that compounds of natural origin are more preferred as functional ingredients by consumers over artificial ones. Moreover, compounds like polyphenols cannot be chemically synthesized and therefore must be extracted from plants (Schieber and others 2001).

It is estimated that approximately 5-7 million tonnes of grape pomace is produced by the wine industry (Jackson, 1994). Presently, grape pomace is widely used for cattle feed or soil conditioning (Mazza, 1995). Numerous applications have been suggested for the utilization of winery by-products, including the production of anthocyanin, cosmetics, citric acid, ethanol, grape seed oil, and dietary supplements (Mazza, 1995).

Industrial wastes (by-products) after wine making and juice extraction are attractive sources of natural antioxidants (Moure and others 2001). By-products of wine and juice production are also characterized by considerably high contents of phenolics compounds. Pomace recovered after juice extraction or wine production serves an inexpensive source of polyphenolics which can be extracted and used as a food supplement or in phytochemical production (Alonso and others 2002). Makris and others (2007) reported that wine industry by-products are very rich sources of antioxidant polyphenols as compared with other agri-food solid wastes. Similarly, Lapornik and others (2005) reported that grape pomace contains high levels of antioxidant compared to other fruit crops.

Canadaigua Wine Co. released two color agents derived from grapes; they are stable at pH 3 to 4.5, and possess a wide range of application (Morris and others 2004). Grape seed

extract, on account of its nutritional value, color and flavor, is extensively used in fruit-flavored beverages and beverage mixes. Soon its use as an ingredient in snack bars, cereals, dairy desserts is expected. Rojas and Brewer (2007) reported that grape seed extracts are more effective antioxidants compared to oregano extracts and a rosemary oleoresin extract at retarding lipid oxidation in cooked meats during refrigerated storage.

Grape seed oil is also a by-product of the grape industry, produced from seeds by either pressing or soluble extraction (Axtell, 1992; Peterson, 2001). Chemical profiling has revealed that grape seed oil is low in saturated fats and high in unsaturated ones. For example, one tablespoon of grape seed oil has about 10 mg of vitamin E, which is higher than that of sunflower or safflower oil (Morris and others 2004). Lu and Yeap Foo (1999) identified 17 polyphenolic constituents by NMR spectroscopy in Chardonnay grape pomace. Torres and Bobet (2001) showed a new class of compounds, aminoethylthio-flavan-3-ol conjugates, from grape pomace by thiolysis of proanthocyanidins in the presence of cysteamine. Shi and others (2003) reported that grape seeds contain anywhere from 5 to 8% polyphenols.

Drying of grape pomace at high temperatures may cause a significant reduction of extractable polyphenols and also affect antioxidant activity and free-radical scavenging activity (Larrauri and others 1997; Larrauri and others 1998).

French paradox

The French paradox refers to the observation by Samuel Black in 1819, that French people suffer relatively low incidences of coronary heart diseases (CHD) as compared to US and UK populations, despite a high intake of saturated fats and relatively high plasma cholesterol levels in the population; that is, similar to US and UK populations (Renaud and De Lorgeril, 1992; Klatsky, 2008). The main difference in the diets of these countries is that the French

prefer to consume wine with their meals, whereas Americans and British prefer beer, spirits, or non alcoholic beverage. There is one more noticeable difference amongst the diet and that is the French consume a greater amount of fruits and vegetables. There has been a substantial amount of evidence indicating a negative correlation between CHD mortality and wine consumption, which indicates the positive impact of wine intake (Dávalos and others 2005). The possible mechanism suggested behind the “French Paradox” is the capability of wine phenolics (e.g. procyanidin, resveratrol, ellagic acid, catechin.) to protect LDL cholesterol from oxidation due to an antioxidative effect (Frankel and others 1993). Antiulcer properties have also been demonstrated by the consumption of wine (Saito and others 1998). Resveratrol, a wine phenolic constituent, has also been reported to possess high antioxidant activities and may suppress or prevent cancer (Pace-Asciak and others 1995). The authors also concluded that antiplatelet activity was observed by the action of wine phenolics in dealcoholized red wine. Folts (2002) found that the consumption of red wine and purple grape juice caused high antiplatelet activity in the blood of dogs, monkeys, and humans. Frankel and others (1993) found that 1000 times diluted, wine was able to equally inhibit the *in vitro* lipid oxidation as 10 µmol/L quercetin. The authors also reported that the inhibition came from non-alcoholic constituents in the wine.

Lindsay and others (2002) concluded that the wine consumption is one of the factors associated with a reduced risk of Alzheimer’s disease. Balu and others (2005) reported that aged rats showed improvement in their superoxide dismutase levels up to 50% as compared to control rats; there was no significant ($p>0.05$) improvement in young rats, when given daily dose of grape seed extract. This study also suggests a beneficial effect of the grape seed extract on age-related changes in lipoxygenase and the antioxidant system in various regions of the brain & spinal cord, such as cerebral cortex, striatum and hippocampus.

Recently, the “French Paradox” has stimulated new interest to investigate the health benefits afforded by grape seed extracts.

Phenolic compounds

Phenolic compounds are secondary metabolites of plants that are derivatives of pentose phosphate, shikimate, and phenylpropanoid pathways (Randhir and others 2004). Phenolic compounds are classified according to their structural complexity and biosynthetic origin. Dietary phenolics are classified into six groups comprising flavonoids, isoflavonoids, lignans, stilbenes, phenolic acids, and phenolic polymers (Figure 2.1). Flavonoids can be defined as “a class of secondary plant metabolites derived from the condensation of a cinnamic acid with three malonyl-CoA groups” (Bloor, 2001). The basic skeleton of a flavonoid is that of diphenylpropane (C₆-C₃-C₆) consisting of 15 carbon atoms (Figure 2.2). A flavonoid consists of two benzene rings (A, B) connected by the six carbon atom ring (C). Rings A and C are condensed together and ring B is attached with ring (C) at C₂. Flavonoids can be monmeric, dimeric, or oligomeric in nature. According to the level of oxidation in the C ring flavonoids can be divided into 14 classes (Seigler, 1998), of which only 5 classes are widely found in diet (shown in Figure 2.3) and those are the following:

(1) Anthocyanidins: They have a flavylum cation structurally related to the flavonols. They are responsible for the color in fruits like red, blue, and purple as seen in cherry, grapes, and eggplant etc. Examples include petunidin, peonidin, delphinidin, pelargonidin, malvidin, and cyanidin. They exist as aglycones and the glycosylated form of an anthocyanidin is called an anthocyanin. Like anthocyanidins, anthocyanins are water soluble and produce color. The basic chemical structure of major anthocyanins are shown in Figure 2.4

(2) Flavanols: Flavanols are also referred to as flavan-3-ols and possess 2-phenyl-3,4-dihydro-2*H*-chromen-3-ol skeleton. Flavanols do not possess a carbonyl group at the 4 position compared to flavonols, and do not have a double bond between C₂ & C₃. Black grapes, red wine and green tea are good source of flavanols and are rich sources of catechin, epicatechin, epicatechin gallate and epigallocatechin-3-gallate.

(3) Flavonols: Flavonols are known as “3-hydroxyflavones” and have the basic skeleton of 3-hydroxy-2-phenylchromen-4-one. They differ from flavones due to the presence of a 3-hydroxy substituent. Examples include quercitin, kaempferol, gossypetin, and quercetagenin. Onions, broccoli, apples, grapefruits, red wine and tea are rich sources of flavonols.

(4) Flavones: They possess a 2-phenylchromen-4-one skeleton. Flavones lack an OH group at C₃. Typical examples of flavones include rutin, apigenin, luteolein, diosmetin, apigenin, and chrysin. Onions, apple skins, berries, and tea are good source of flavones.

(5) Flavanones: The basic skeleton is 2,3-dihydro-2-phenyl-chromen-4-one. They differ from flavones in having double bond at position 2,3 in C ring. Citrus fruits are rich in flavanones like myricetin, hesperidin, naringin, and naringenin.

Some researchers have classified isoflavonoids as a member of flavonoid family. Isoflavonoid differs from other members of the flavonoid family in having ring B attached to ring C at the 3 position instead of 2. Some examples of isoflavonoids are genistein, diadzein, and glycitein.

Flavonoids are naturally present as glycosides. Due to the attached sugar moiety, their adsorption in the intestinal tract facilitates. Mainly, glucose is the sugar moiety of glycosylated flavonoids, but rhamnose, arabinose, and xylose can also be found. Flavonoids play a diverse role: they are responsible for the color of many fruits and vegetables and they protect plants from

UV radiation, insects, and herbivores (Haslam, 1989). Moreover, flavonoids are also known for antimicrobial and antioxidant activities and are also known for their preventive action for many diseases (Harborne and Williams, 2000). Plant polyphenols have physiological functions in plants and pharmacological functions in living organisms. Earlier plant polyphenols were considered undesirable constituents of food due to their bitter taste, astringency, and also because of the interaction between polyphenolics, mainly procyanidins, and the glycoprotein in saliva (Rider and others 1992). The astringent flavor of polyphenols makes the plant unattractive to potential predators, and consequently survives. Flavonoids are known to possess antioxidant activity, which helps to reduce the risk of cardiovascular diseases, and have also demonstrated an ability to protect tissues against free-radical attack and lipid peroxidation. Many researchers have also shown the effect of flavonoids in the prevention of atherosclerosis, cancer, inflammation, and the lowering of plasma cholesterol. Flavonoids that contain multiple OH substitutions have very strong antioxidant activities against peroxy radicals (Cao and others 1997). The medicinal and therapeutic properties of flavonoids makes desirable to consume fruits and vegetables. In the US diet, the dietary intake of flavonoids may be only a few hundred milligrams (Hollman and katan 1999), although dietary intake of flavonoids is not adequately estimated due to a lack of information of the flavonoid content in the food consumed and the bioavailability of specific flavonoids. Polyphenolic compounds have been shown to play roles in the reduction of platelet aggregation, modulation of cholesterol synthesis and adsorption, and reduction of blood pressure (Liu, 2003).

Tannins are high-molecular-weight compounds and can be divided in two subcategories: those being hydrolyzable tannins, which are esters of gallic acid or ellagic acid and

proanthocyanidins, which are polymers of flavan-3-ol monomers. Grape seed is very rich in proanthocyanidins and this may be responsible for its many health benefits.

The term “phenolic acid” refers to a large variety of compounds having at least one phenolic hydroxyl group and one carboxyl function; however, some authors prefer classification into phenolic acids and phenol aldehydes (Harborne and Dey 1989). Phenolics can be classified on the basis of hydrogenation, hydroxylation, methylation, malonylation, sulphation and glucosylation. Phenolic acids are precursors of flavonoids. Example of common phenolic acids include salicylic acid, *p*-hydroxybenzoic acid, 3,4-dihydroxybenzoic acid, vanillic acid, gallic acid, syringic acid, gentistic acid, caffeine acid and ferulic acid.

Synthesis of flavonoids and phenolic acid

Polyphenols are secondary plant metabolites. They are synthesized through the shikimate pathway, which follows from pentose phosphate, phenylpropanoid, and the glycolytic pathway (Randhir and others 2004). In the shikimate pathway, simple carbohydrates derived from pentose phosphate and the glycolytic pathway are converted to aromatic amino acids like phenylalanine, which is the substrate for the phenylpropanoid pathway in which synthesis of various secondary metabolites occurs. In the phenylpropanoid pathway, phenylalanine is converted to *trans*-cinnamic acid by the enzymatic activity of phenylalanine ammonia lyase catalyase (Figure 2.5). *Trans*-cinnamic acid is the precursor for other phenolic acid derivatives. By the activity of hydroxylase, *p*-coumaric acid is converted into caffeic acid and then to ferulic acid. Chalcone and resveratrol are produced in another reaction where one molecule of *p*-coumaric acid condenses with three molecules of malonyl-CoA. Different flavonoids are produced from the chalcone by action of chalcone isomerase (Figure 2.6).

Bioavailability of polyphenols

The chemical structure of the phenolic compound dictates their level of bioavailability, antioxidant activity, and specific interaction with cell receptors and enzymes (Scalbert and Williamson 2000). There are a limited number of studies concerning the adsorption and bioavailability of phenolic compounds. A widely accepted concept is that polyphenols are absorbed in the intestine by passive diffusion and for this to occur, glycosylated polyphenols are converted to the aglycones by the activity of glycosidase in the food or gastrointestinal mucosa.

Phenolic compounds as antioxidants

The antioxidant activity of phenolic compounds is due to their ability to scavenge free radicals, donate hydrogen atoms or electrons, or chelate metal cations (Afanas'Ev and others 1989). Radical-scavenging and metal chelating activities of polyphenolic compounds can be estimated by their structure.

Free radicals are basically defined as molecules that have an unpaired electron in their outer shell. They are highly unstable and very reactive. Free radicals are produced in the body as a by-product of normal metabolism. They are very unstable due to the presence of unpaired electrons and therefore, it become very important to scavenge these free radicals otherwise a chain reaction can be initiated by them causing damage to biomolecules and body cells. Free radicals can be useful for the body by inducing different processes like signal transduction and gene transcription. The harmful effects of free radicals are oxidation of proteins, amino acids, lipids, and DNA which can lead to many diseases like CHD, cancer, dementia, etc.

Antioxidants are compounds which prevent the oxidation of substrates caused by free radicals. “Any compound that has a reduction potential lower than the reduction potential of a free radical (or oxidized species) is capable of donating a hydrogen atom to that free radical

unless the reaction is kinetically unfeasible” (Decker, 2002). Antioxidants can be natural or synthetic. In the human system, antioxidants prevent or delay the oxidation of biomolecules and thereby reduce and delay the risk of chronic diseases. In food systems, antioxidant prevents lipids from becoming oxidized and therefore helps to maintain the original state of food. As well as dietary intake of antioxidants facilitate the normal physiological functions in human body. The antioxidant activity of polyphenolic compounds are greatly influenced by a number of factors like “position and degree of hydroxylation, polarity, solubility, reducing potential, stability of phenolic compound during food processing operations, stability of the phenolic radical, and the acid or ring group in the phenolic structure” (Decker and others 1998). Soleas and others (2001) mentioned that the antioxidant activity can also be influenced by glycosylation. The radical scavenging activity of polyphenols is attributed to their chemical structure ($C_6-C_3-C_6$) which helps in radical stability and delocalization of lone pair of electron. Many researchers have shown that the number of hydroxyl groups in a molecule highly influences the antioxidant activity of the compound. Even the position of the hydroxyl group can change the antioxidant ability to some degree. Knudsen and others (1996) reported that antioxidants taken in combination are expected to be more efficacious than taken individually.

Sánchez-Moreno and others (2000) reported that lipid peroxidation was equally inhibited by gallic acid, resveratrol, and tannic acid and that the inhibition was higher than BHA or α -tocopherol. Similarly, Cuendet and others (2000) concluded that *trans*-piceid, a resveratrol glucoside, was equally effective as BHT in scavenging free radicals. Ohshima and others (1998) showed that flavonoids like catechin, epicatechin, and gallic acid can inhibit the breaks in DNA strands induced by the presence of nitroxyl radicals. These compounds are also successful in scavenging nitric oxide, peroxynitrite and nitroxyl radicals. Torel and others (1986) found that

flavonoids like catechin, kaempferol and quercetin were remarkably efficient in inhibiting lipid oxidation.

Hou and others (2004) investigated the effect of flavonoids like quercetin, kaempferol, and myricetin on free radical or copper-initiated LDL oxidation and found them to be effective antioxidants. Similarly, in a study by Safari and Sheikh (2003) to examine the effect of flavonoids on copper-mediated oxidation of LDL, quercetin was found to be the most effective at preventing oxidation. Morel and others (1993) concluded that catechin, quercetin, and disometin in decreasing order are capable of chelating iron and therefore play a significant role at reducing the extent of lipid peroxidation.

There are several methods available to measure antioxidant capacity of polyphenolic compounds. One example is the Trolox equivalent antioxidant capacity (TEAC) (Miller and others 1993). In this method 2,2-azobis(3-ethylbenzothiazoline-6-sulfonate) (ABTS^{•+}) is used to generate free radical, ferric-reducing ability of plasma (FRAP) (Benzie and Strain, 1996), oxygen radical absorbance capacity (ORAC) (Cao and others 1995), 2,2-azobis (2-amidinopropane) dichloride (AAPH) is used as a peroxy radical generator in ORAC, color reduction of 2,2-diphenyl-1-picrylhydrazyl radical (DPPH[•]) (Sanchez-Moreno and others 1998), a mixture of FeCl₃-EDTA, hydrogen peroxide and ascorbic acid is used to generate free hydroxyl radicals for an antioxidant assay using DPPH[•], 2-thiobarbituric acid reactive substances (TBARS) this method quantifies the reaction product from the decomposition of lipid peroxides during inhibition of lipid peroxidation by antioxidants (Plumb and others 1996), and electron paramagnetic resonance (EPR) spectroscopy (Bors and others 2001), by the EPR spectroscopy not only the antioxidant activity is estimated but also the information on the intermediate structures is elucidated.

In the classical FRAP assay, the reduction of a ferric-tripyridyltriazine complex to its ferrous colored form in the presence of plasma antioxidants is determined. Therefore FRAP measures the total reducing ability of antioxidants. The assay is based on a single electron transfer (SET) mechanism. In the SET-based method, antioxidants are oxidized by oxidants (Fe^{3+}) and therefore a single electron transfers from the antioxidant molecule to Fe^{3+} . The change in absorbance of the solution is measured by visible spectrophotometry at a $\lambda=595\text{nm}$, and for quantification of the reducing power of antioxidants, absorbance value are used. Below is the equation showing the SET from the antioxidant (AH) to Fe^{3+} .



Health benefits of polyphenolic compounds

Over the past two or three decades, research has been conducted to foster the hypothesis that the consumption of fresh fruits is associated with a reduced risk of cardiovascular diseases, cancer, Alzheimer's, ischemic, strokes and many other chronic diseases. Through a number of studies it has been shown that the health benefits of fruits are mainly due to endogenous polyphenolic compounds which act like antioxidants in the body. In a study by Verlangieri and others (1985) a negative correlation was observed between the consumption of fresh fruit and CVD, diabetes mellitus, and cystic fibrosis in the American population. Dauchet and others (2006) observed a 7% decrease in the risk of CHD by consumption of fruits in a cohort study including both men and women. In another epidemiological study to investigate the relationship of flavonoids and CHD, Knekt and others (2002) reported lower incidences of ischemic heart disease and a negative correlation between heart diseases and the intake of kaempferol and quercetin. Similarly, Keli and others (1996) conducted a Zutphen study in which they concluded that there was clear correlation between the intake of flavonoids and a lower risk of CHD. They

also indicated that there was no association between intake of vitamin C and vitamin E and stroke risk. Moreover, these authors reported that quercetin was most effective against the risk of stroke from all flavonoids.

Cancer is defined by the American Cancer Society (ACS) as “a group of diseases characterized by uncontrolled growth and spread of cells”. According to the ACS, factors like lifestyle (diet and exercise), environmental changes and genetic factors play an important role in induction of cancer. The National Cancer Institute suggests that people consume a diet rich in phytochemicals like fruits and vegetables throughout their lifetime to reduce the risk of cancer, as prevention is the major line of defense against all cancers (Hartle and others 2005).

Kuroda and Hara (1999) showed that an inverse relation was observed in Japan based on an epidemiological study of the consumption of green tea and standardized mortality rates due to cancer. Han (1997) observed anticarcinogenic activity of EGCG, ECG, EGC, EC and green tea during chemical carcinogenesis. Friedman and others (2007) in a study on tea flavonoids concluded that catechins and other flavonoids were successful in reducing cancers in the human cancer cell lines of breast, colon, hepatoma, and prostate.

In a case control study by Rossi and others (2007) on patients with squamous-cell carcinoma of the esophagus, the effects of flavanones and flavonols were found to negatively correlate with the risk of cancer. Fink and others (2006) concluded that flavonoids reduce the risk of breast cancer in postmenopausal women.

Singletary and others (2003) concluded that there was a significant decrease in the development of tumors in mice which were given anthocyanin-rich concord grape juice. Kang and others (2003) reported that there was a reduction in cell growth of human colon cancer cell lines in mice which ingested anthocyanins as compared to those which did not. Jung and others

(2006) reported that it is likely that purple grape juice inhibits the initiation stage of carcinogen-induced breast cancer.

Engelhart and others (2002) in a study on the association between the intake of flavonoids and risk of Alzheimer's disease showed that the risk of Alzheimer's disease decreased with intake of flavonoids.

For antimicrobial properties of polyphenolics most of the interest has centered upon resveratrol from the *vitis* family because of its function as a phytoalexin. Phytoalexins are a group of plant chemicals of low-molecular-weight induced by fungal attack (Soleas and others 1997). Jayaprakasha and others (2003) found that grape seed extracts exhibited moderate antibacterial activity against many bacteria like *B. cereus*, *S. aureus*, and *B. subtilis*. In a study on the mode of antibacterial action of epicatechin and other flavonoids in green tea, Ikigai and others (1993) concluded that bactericidal flavonoids, mainly catechins, primarily attack the bacterial membranes and damage them. Quercetin and kaempferol were found to have moderate antimicrobial activity against *Staphylococcus aureus*, *Staphylococcus epidermitis*, and *Bacillus subtilis* (Rauha and others 2000).

Phytochemicals in muscadine berries

The phytochemical profile of muscadines has been documented in a few studies. Muscadine berries are rich in phenolic compounds and antioxidants which has prompted more research to evaluate of the antioxidant composition of muscadines (Figure 2.7). Muscadine grapes are distinguishable from most other grapes varieties due to predominance of anthocyanin 3,5-diglucosides of delphindin, cyaniding, and petunidin and by the presence of ellagic acid and ellagic acid precursors which are unique among the *Vitis* family (Lee and Talcott 2002). In muscadines and other members of the *Vitaceaes* family, color and taste are highly influenced by

the concentration of anthocyanin and proanthocyanidins. Approximately 90% of the total anthocyanins are 3,5-diglucosides of delphinidin, cyanidin and petunidin and the remaining 10% comprise 3,5-diglucosides of peonidin and malvidin (Huang and others 2008). The anthocyanin 3, 5-diglucosides are more resistant to thermal processing to monoglucosides. Presence of ellagic acid also makes a distinguishable attribute to muscadines, which is commonly found in berries (Lee and Talcott 2004). Muscadine grape contains appreciable concentration of neutral and acid polyphenolics that act as inhibitors of oxidation as compared to *Vitis vinifera* and *Vitis labrusca* (Meyer and others 1997; Frankel and others 1995). Pastrana-Bonilla and others (2003) reported the average total phenolic content of ten different cultivars of muscadine whole fruit, seed, skin, pulp and leaves were 0.25, 2.18, 0.37, 0.02, 0.35 g/100 g gallic acid equivalent fresh weight respectively and the antioxidant activity was 2.4, 12.8, 281.3 and 236.1 μ M TEAC/g of fresh weight for pulp, skins, seeds and leaves, respectively. The total Anthocyanin content in skin of purple muscadines was 132.1mg/100g of fresh weight. The total phenolics in muscadine grape are 5 times more concentrated in seed than in skin and 80 times more than in pulp (Hartle, 2005). In muscadine grape seed, two most prominently found phenolic compounds are catechin and epicatechin and skin is highly enriched with ellagic acid, myricetin, quercetin, and kaempferol (Pastrana-Bonilla and others 2003). Yilmaz and Toledo (2004) reported that the concentration of gallic acid, monomeric catechin, and epicatechin in muscadine seed were 99, 12, and 96 mg/100g of dry matter. Ector and others (1996) reported the resveratrol concentration in muscadine berries, juice, pomace, purees, seeds and wine, varied from 2.7 - 62.2 μ g/g and also concluded that the seed had highest concentration of resveratrol than any other part of the plant. Magee and others (2002) in a study on effect of fungicides on resveratrol content of muscadine cultivar found that, the average amount of resveratrol in skins and seeds of muscadine were 2.84

and 0.83 µg/g respectively. Pastrana-Bonilla and others (2003) found the *trans*-resveratrol concentration to be lowest i.e., 0.2 mg/100 g fresh weight. They also suggested that the discrepancy between their results and earlier documented results about resveratrol concentration in muscadine can be due to inability of separating ellagic acid from resveratrol with UV detection alone or due to varietal differences in muscadine berries. In study on the effect of processing on the total phenol content in muscadine grape, Auw and others (1996) showed that the total phenolics ranged from 162 – 1096 mg/L gallic acid equivalent.

Banini and others (2006) reported that daily intake of muscadine wine or dealcoholized muscadine grape wine has been shown effective in lowering blood glucose level, insulin and glycated hemoglobin, indicating better glycemic control, improved insulin sensitivity and decreased tendency towards impaired liver function was also observed in subjects with Type 2 diabetes. Yi and others (2005) concluded from their study that polyphenols present in muscadine poses cancer preventing properties.

As the consumers are becoming aware of the health benefits due to intake of muscadine, the demand for fresh and processed muscadine is increasing (Huang and others 2009). Striegler and others (2005) reported that the muscadine industry is currently experiencing its greatest growth in decades and the future looks excellent.

Health benefits of grape pomace

In the studies by Nigdikar and others (1998); and Shrikhande (2000), it was reported that plasma polyphenols of individuals increased 38% with red wine, 27% with white wine with added 1g red wine polyphenols, and 28% with red wine polyphenol capsules, but not with white wine capsules. Plasma lipid peroxides also decreased by 32% with red wine, 29% with white wine plus 1g red wine polyphenols and 28% with red wine polyphenol capsules and there was

increase of 23% with white wine. Shrikhande (2000) commented in his paper that the intake of winery by-products in long term may result in reduced incidences of atherosclerosis that is mainly concluded from the study carried by (Yamakoshi and others 1999), where the researchers suggested that the major polyphenol in red wine is procyanidin which traps the reactive species in plasma and intestinal fluid of the arterial wall, thereby inhibiting oxidation of LDL. Procyanidin extract from grape seed has been shown to have in vivo activity and exhibits same level of antioxidant activity against lipid oxidation and in preventing oxidative damage in tissue, as vitamin E and also by decreasing the production of free radicals (Sato and others 2001; Tebib and others 1997; Bouhamidi and others 1998; Bagchi and others 1998). Grape seed extract has been demonstrated to reduce the rate of myocardial infarction and also atherosclerosis (Sato and others 1999; Yamakoshi and others 1999). In a study on cardioprotective effects of grape seed proanthocyanidin in ischemic/reperfused rats, Sato and others (1999) observed that the rate of myocardial infarction was lower in mice fed with GSPE in comparison to control. Grape seed extract is also known for inducing apoptosis in cancer cells (Mertens-Talcott and others 2006; Yi and others 2005), preventing periodontal diseases (Houde and others 2006), and is also found to be photoprotective against ultraviolet-induced oxidative stress (Mantena and Katiyar, 2006).

Laurent and others (2007) reported that grape seeds at 400 mg/L modulated cell proliferation and differentiation and led to morphological and functional changes in Caco-2 cells. Bomser and others (2000) found that the grape seed extract containing mainly oligomeric and polymeric proanthocyanidin showed antitumor activity in mouse skin epidermis.

In a study on hamsters fed on high fat diet Decorde and others (2008) reported that the consumption of grape seed extract has beneficial effects on preventing diet induced obesity by improving oxidative stress markers

Muscadine pomace (By-product)

Muscadines are approximately 40% skin, 50% pulp and 10% seed. Therefore it can be assumed that almost 50% of the fruit is lost as a press fraction (pomace). Thus use of muscadine pomace will have a great impact on the muscadine industry by decreasing the waste disposal problem and increasing the market value per ton of fruit (Ector, 2001).

Muscadine skin and seeds are a rich source of health benefitting polyphenolic compounds (Torres and Bobet, 2001), and seeds and skins are a better source of phenolics as compared to the pulp (Pastrana-Bonilla and others 2003). Seeds of muscadine have the highest antioxidant activity as compared to other fruit parts possibly due to the presence of procyanidins, catechin and epicatechin (Pastrana-Bonilla and others 2003). High concentrations of anthocyanin, gallic acid, catechin, epicatechin, ellagic acid and resveratrol found in the seeds and skins give muscadine a high antioxidant capacity (Ector and others 1996; Striegler and others 2005). Muscadine seed skin contains oligomeric, and polymeric proanthocyanidin, and the degree of polymerization is higher in skin (Souquet and others 1996; Prieur and others 1994). Huang and others (2008) reported that anthocyanins of muscadine are mainly concentrated in the skin. Lee and Talcott (2004) concluded that the ellagic acid concentration in muscadine juice was considerably lower than in skin or pulp. They also compared ellagic acid content in different cultivars of muscadine and highest concentration was found in Albermarle and Noble cultivars. Apart from providing health benefits because of the presence of anthocyanins in muscadine pomace it can be potentially used as colorant due to its bright, attractive color and high solubility can easily substitute synthetic colors in foods (Mazza and Miniati, 1993).

Mertens-Talcott and others (2006) reported that grape seed extract induces apoptosis in cancer cells. Greenspan and others (2005) reported the anti-inflammatory activity *in vivo* and *in*

vitro of the powder prepared from muscadine skin. They reported that extracts of muscadine were effective in inhibiting the release of superoxides, interleukin-1 and tumor necrosis factor. Yi and others (2005) from their study concluded that the phenolics from muscadine skin are potentially capable of prevention of colon cancer by inhibiting the cancer cell growth and inducing apoptosis.

Muscadine skin and seeds have high concentrations of polyphenolic compounds. Pomace of muscadine berries due to occurrence of major flavonoids can be used as a potential source of nutraceutical because of their high antioxidant (both *in vitro* and *in vivo*), antimutagenic (Weyant and others 2000), antiplatelet (Pace-Asciak and others 1995), cardioprotective (Sato and others 2001), antiatherosclerotic (Yamakoshi and others 1999), antimicrobial, antiulcer activities (Saito and others 1998) and also due to the benefits to gastrointestinal health (Tebib and others 1997).

Drying

Drying refers to the removal of moisture from a substance (Ratti, 2001). Drying is one of the oldest and important aspect of food preservation and processing (Lin and others 1998). The basic concept behind removal of free water is to reduce microbiological spoilage and increase shelf life (Kwok and others 2004). Usually in drying, the target is to reach the water activity (a_w) at which the product quality and shelf life of the product are acceptable and desired. Dehydration reduces weight which is an important consideration while shipping, and also need of refrigeration, making it easier to pre mix retail products (Adams, 2004). Temperature and time to which food is exposed for drying is directly proportional to the amount of damage incurred to the food (Lin and others 1998). Various changes may take place in food during processing. These changes alter the physical as well as biochemical aspects of the food like color, texture, deterioration of aroma compounds or degradation of nutritional substances,

porosity and sorption characteristics of the materials (Achanta and Okos 1995). Therefore, the drying method and physiochemical changes that occur during the drying process affect the quality of the dehydrated product.

Air drying is one of the oldest method of food preservation where the food to be dried is exposed to continuously flowing hot stream of air and moisture evaporates (Ratti, 2001). It is one of the most widely used operations for food dehydration. In conventional hot air drying nutrient loss can occur because of the breakage of covalent bonds and also because of being an open system antioxidants can get oxidized. High temperatures for long time are often responsible for the loss of texture, color, flavor and nutritional value of food (Schadle and others 1983; Yang and Atallah 1985). Usually the temperature for air drying is between 50-90°C, the air humidity is between 10-40% and air velocity between (1-4 m/s).

In conventional heat drying, the drying rate is usually high in the beginning of drying but as the moisture gradient drops, the drying rate falls. Hence, the center of the product do not attains the desired temperature and continuous heat treatment causes in case-hardening i.e., at the surface of the product over heating occurs.

Spray drying is used to remove water from a liquid by transforming fluid into a dry particulate by spraying in a hot medium. Spray drying highly depends on feed rate, inlet drying air temperature.

Drum drying is a drying technique in which product is kept on revolving drum and heat transfer takes place to the material by conduction. Drum drying is a type of air drying only. The product starts dehydrating when the temperature reaches the boiling point of water. Drum drying is adequate for the products with high viscosity and that can tolerate high temperatures.

Fluidized bed drying involves levitation of particles in an upward flowing gas stream, typically hot air. Due to fluidization intimate contact occurs between the solid particulates and hot air. Heat transfer occurs by convection and heat transfer occurs at faster rate. Drying occurs rapidly in fluidized bed drying. This drying method depends on characteristics of particle and carrier gas.

Microwave drying is a non conventional method of drying and only recently has come in use for food industry. Drying time of products is greatly reduced due to dielectric heating with microwaves and microwave pretreatment of the product followed by conventional drying can greatly reduce the drying time and also can produce a dehydrated product with moderate operating cost. The parameters for microwave drying are dielectric power and exposure time to MW radiations.

Vacuum drying is a drying method in which drying is performed at low pressure. It is based on the fact that boiling point of water can be lowered as the pressure reduces, therefore drying occurs at faster rate. Vacuum drying is ideal for heat sensitive food materials, very low loss of bioactive compounds occurs, as drying takes place in vacuum therefore oxidation of product can be avoided. Vacuum drying is also ideal in situations where a solvent must be recovered or where materials must be dried to very low levels of moisture. Drying of product depends on the pressure (3-6 kPa) and temperature (50-100°C)

Osmotic dehydration is a drying method which is not widely used and the use depends on the desired qualities of the product. This method can result in moisture removal up to 50%. The dehydration occurs due to the flow of water from the product to a concentrated solution and if desired, simultaneously solute transfer from the solution into the product can take place. Therefore, osmotic dehydration is more of a pretreatment technique in which removal of water

and direct formulation of product through impregnation occurs followed by any other drying method. The osmotic dehydration techniques preserves flavor, nutritional characteristics and prevents microbial spoilage. The drying parameters are solute concentration, process temperature, sample size, speed of agitation and time of immersion.

Freeze-drying also called lyophilization is based on the fact that, when the product is in frozen state, by reducing the pressure and providing enough heat can cause the sublimation of ice into vapor state without passing through liquid state and can cause dehydration of the product. Freeze drying is a well known method for producing high value dehydrated foods (Dalglish, 1990). In freeze drying no heat damage occurs and the food structure remains the same (Lin and others 1998). The main parameter of freeze drying is the applied vacuum, which should be corresponding to specific temperature at which ice sublimates. Disadvantages associated with freeze drying are longer time is required to reach the desired moisture content as well as causes high cost of operation (Litvin and others 1998).

Recently, vacuum-microwave drying has been reported by many researchers in being successful to preserve chemical and textural attributes of food products. Vacuum-microwave drying is combination of two drying methods which when used together causes a synergistic effect by considerably reducing the drying time and involves only few minutes. Vacuum provides benefits of faster mass transfer and minimizing the oxidation of the product and by the use of microwave, need of high temperature eliminates. The vacuum microwave dried cranberries were found to have better red color and softer texture as compared to hot air dried cranberries (Yongsawatdigul and Gunasekaran 1996). It was also reported by Petrucci and Clary (1989) that the contents of vitamin A, vitamin C, thiamine, riboflavin and niacin in dried grapes were largely preserved during vacuum microwave drying. Use of microwave for drying causes

uneven drying, textural damages due to fast drying. Microwave drying also involves high operation cost. Drying of pomace at high temperature, may cause significant loss in polyphenol content as well as antioxidant activity and radical scavenging activity (Larrauri and others 1998).

Vacuum belt drying

There is not much literature present on vacuum belt drying. Vacuum belt drying is a continuous drying process involving vacuum drying in combination of moderate to high temperatures. In 1995, Shevaug and Hallinan patented vacuum belt press dryer (patent no. 5426864), a continuous belt dryer for dehydration of substances with high moisture content. In vacuum belt dryer a teflon belt runs over three heating plate and one cooling zone. There is one heat zone above the teflon belt that covers the three heating plates and transfers heat by radiation and heat transfer occurs by conduction from the three heating zones. This entire assembly is positioned in a vacuum tunnel. These four different heating zones give an advantage of setting up different temperatures according to the requirement. Like, when the moisture content of the product is high, then the high temperature can be set and as the product loses moisture, the conveyor belt can take the product to another zone with lower temperature that is just sufficient to enhance moisture loss. Therefore, avoids unnecessary heating which can minimize damage due to heat and as the process takes place in vacuum, no oxidation of product takes place and also due to vacuum mass transfer takes place at a faster rate and therefore drying takes place in shorter duration and also due to heat transfer by conduction and radiation, heating is even throughout the product.

Nindo and others (2003) demonstrated that the antioxidant activity of the dried asparagus was significantly affected by different drying methods. Freeze drying and refractance window methods showed the highest retention of antioxidants. Stralsjo and others (2003) showed that the

folate content of rosehip berries was significantly affected by drying time and temperature. In a study by YanYang and others (2004) retention of chlorophyll and ascorbic acid in cabbage was higher in samples dried by microwave vacuum in comparison to air dried samples. Piga and others (2003) reported that high drying temperatures decreased the level of anthocyanins, flavonoids and ascorbic acid in plums. Standley and others (2001) concluded that the processing conditions significantly affect the polyphenolic content of tea leaves and also influence their antimutagenic activity. A study conducted by Lohachoompol (2004) demonstrated that the anthocyanin concentration reduced by 41% in blueberries when dried although the antioxidant activity of the fruit did not change. Pan and others (2003) reported that osmotic dehydration pretreatment, followed by drying greatly improves the retention of the nutritional values of the product. Osmotic dehydration of strawberries followed by microwave drying resulted in a product quality comparable to freeze drying in a much shorter time (Venkatachalapathy and Raghavan, 1999). Unjka and others (2004) in a comparative study reported that quality of microwave vacuum dried cranberries was better than the microwave convective dried cranberries. Tambunan (2001) reported that the quality of freeze dried herbs were slightly lower than the fresh but still the quality remained considerably higher than oven dried samples at 35-40°C.

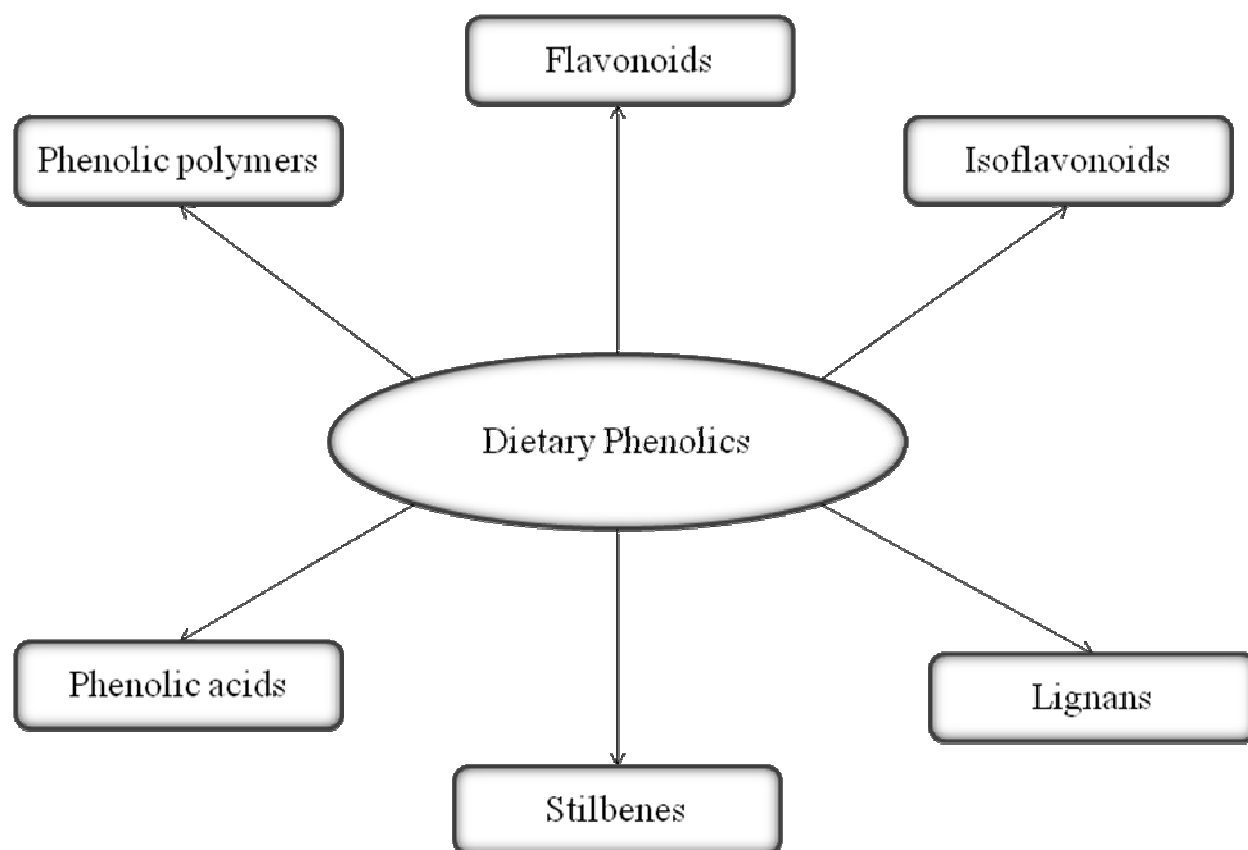


Figure 2.1: Classification of dietary phenolics.

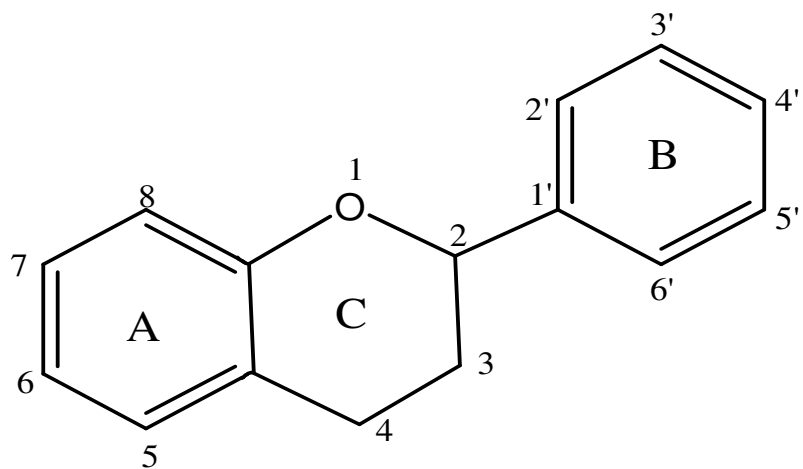
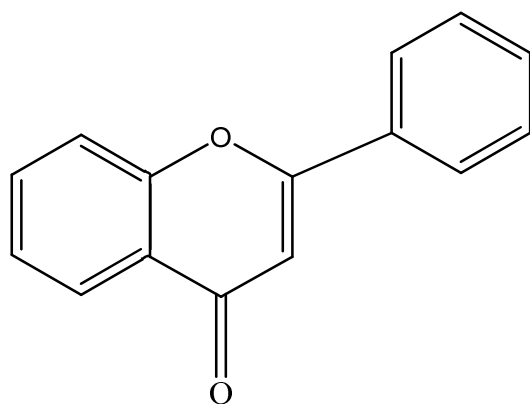
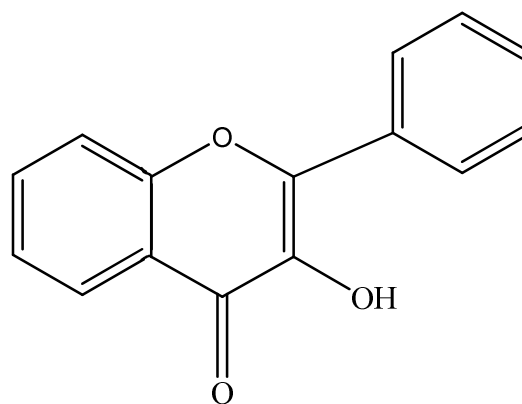


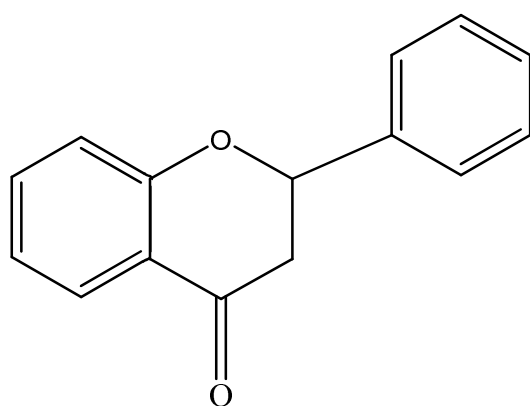
Figure 2.2: Basic structure of flavonoids (C₆-C₃-C₆).



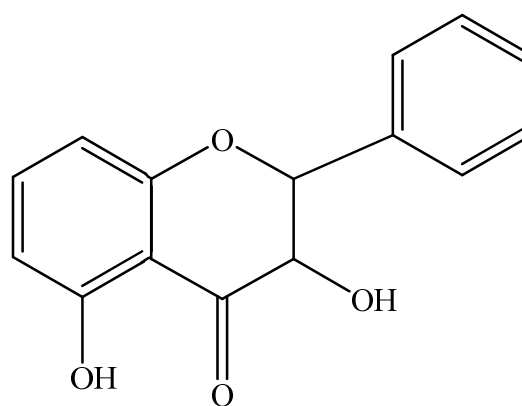
Flavones



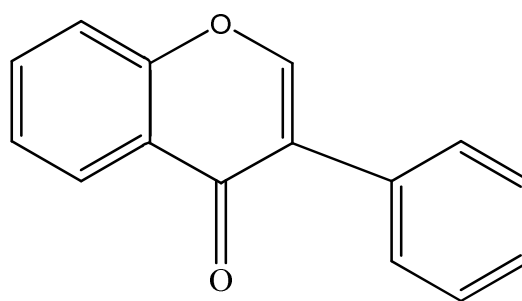
Flavonol



Flavanone

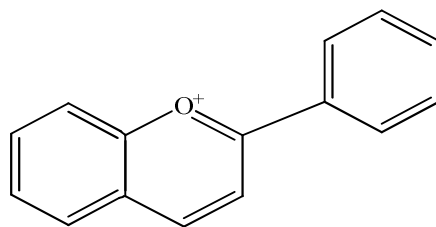


Flavanonol



Isoflavone

Figure2.3: Structure of common flavonoids.



Anthocyanidin (flavylium cation)

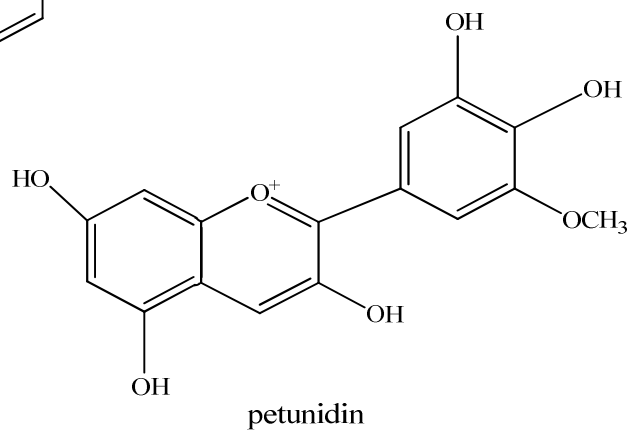
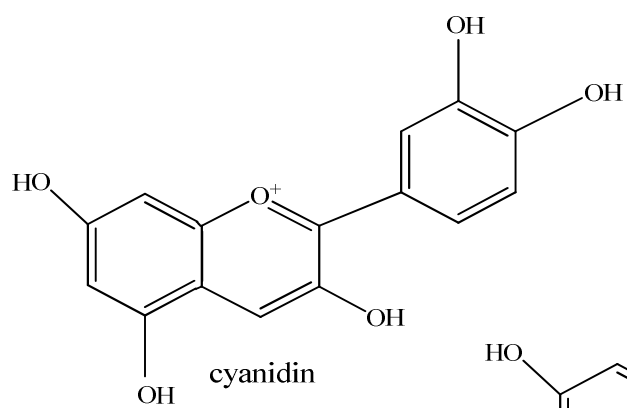
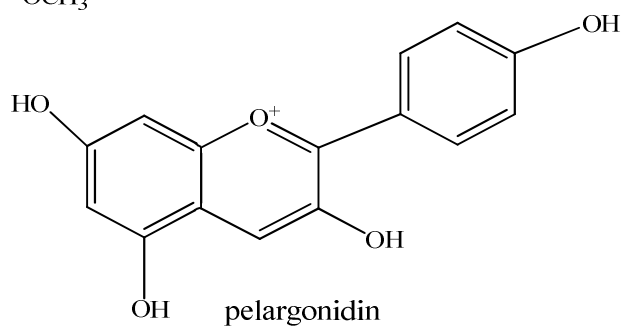
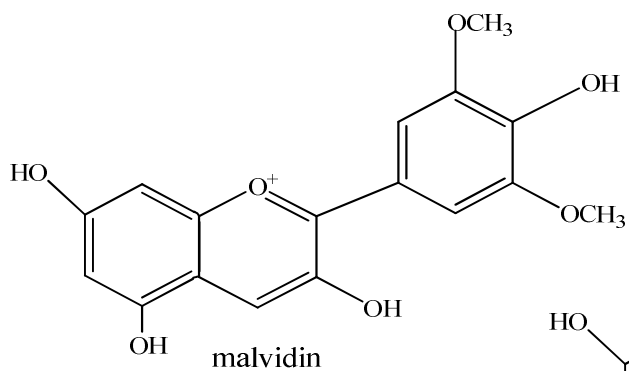


Figure 2.4: Structure of anthocyanidin, cyanidin, malvidin, petunidin, and pelargonidin.

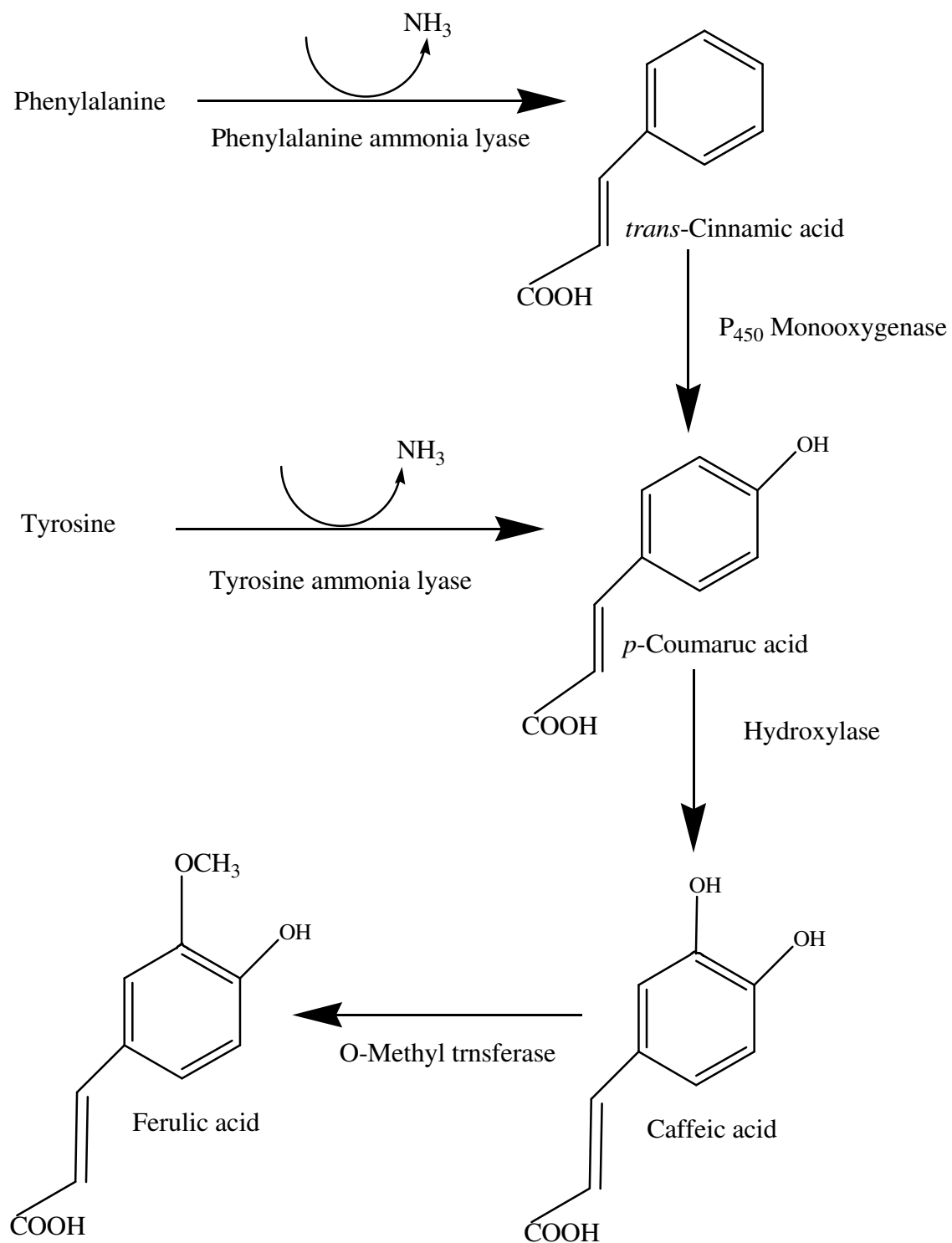


Figure 2.5: Biosynthesis of *p*-coumaric acid and phenolic acids from phenylalanine.

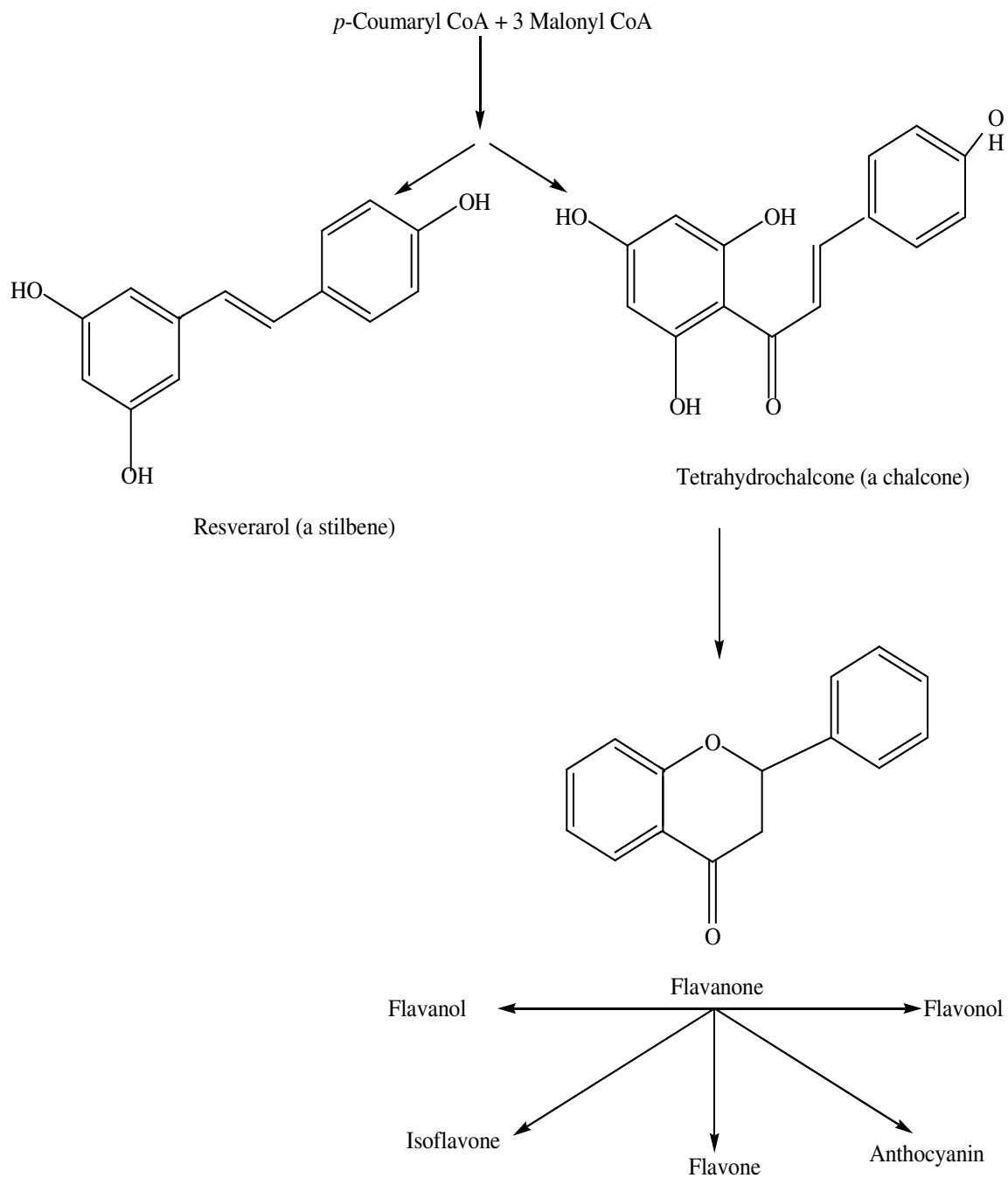


Figure 2.6: Biosynthesis of flavonoids and stilbenes.

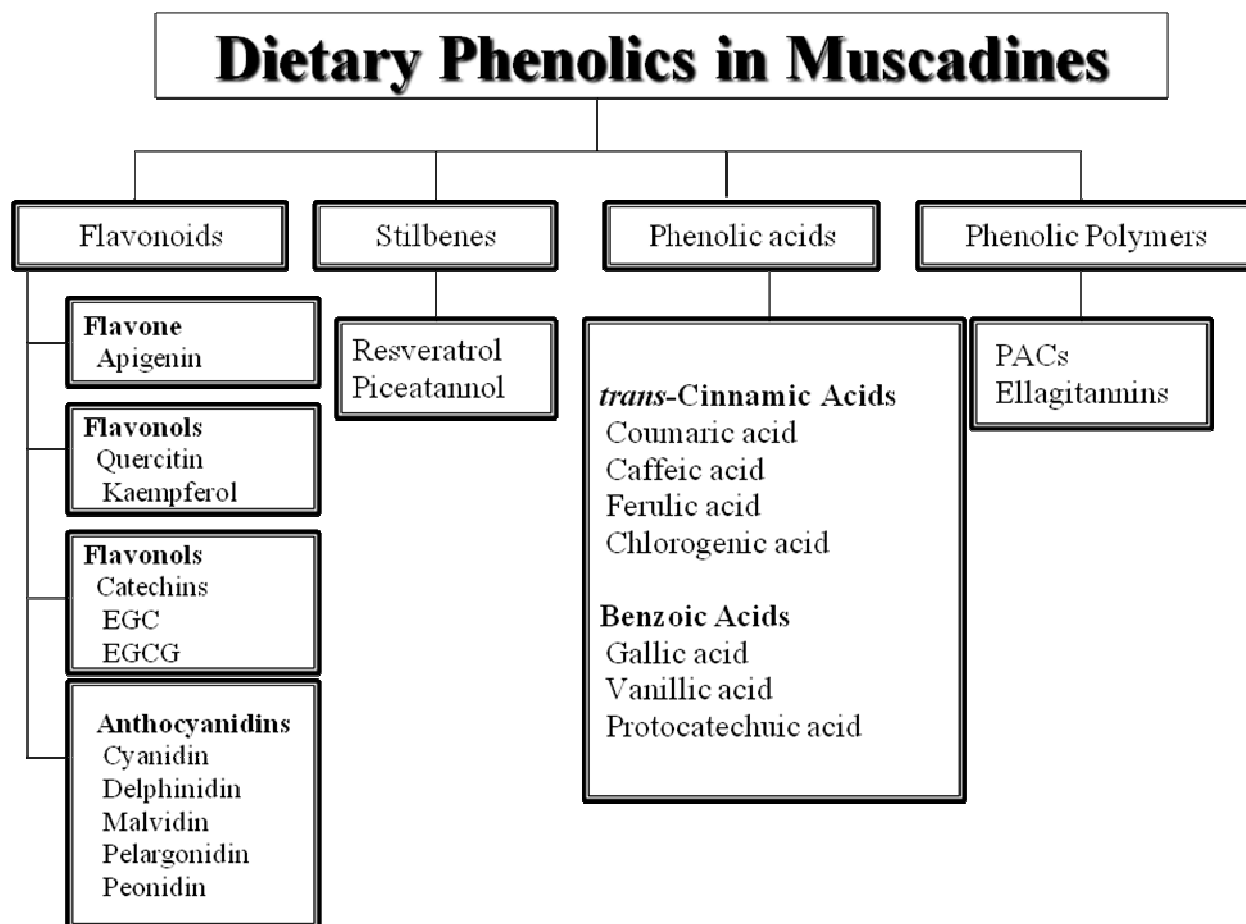


Figure 2.7: Dietary phenolics in muscadine. EGC – epigallocatechin; EGCG – epigallocatechin-3-gallate; PACs – proanthocyanidins.

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CHAPTER 3
**EVALUATION OF DRYING TECHNOLOGIES FOR MUSCADINE POMACE TO
PRODUCE AN ANTIOXIDANT RICH FUNCTIONAL FOOD INGREDIENT**

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Abstract

The objective of this study was to evaluate vacuum belt drying (VBD), freeze drying (FD), and hot air drying (HA) for processing of muscadine pomace and to study the effect of material thickness on the quality of dehydrated pomace in terms of retention of total phenolics content (TPC), antioxidant activity (AA), drying time requirement, water activity (a_w) and moisture content (MC). Four different temperature zones were applied to the product under vacuum in VBD. Muscadine pomace discs with two thicknesses 4 ± 0.5 mm and 2 ± 0.3 mm were dried for 16 different time-temperature combinations for VBD (60 and 90 min; 60°C - 120°C), 12 different time-temperature and air velocity combinations for HA (180 and 240 min; 70°C and 80°C ; 0.2, 0.4, and 0.6 m/s) and one treatment for FD. In lyophilized samples, TPC and AA were found to be 582.5 and 607.5 $\mu\text{mol GAE/g}$ dry weight (DW) and 2.21 and 2.29 mmol $\text{Fe}^{2+}\text{eq/g}$ DW for 2 ± 0.3 mm and 4 ± 0.5 mm thickness, respectively. Treatment TV2 (VBD) resulted in significantly ($p < 0.05$) higher TPC as compared to the lyophilized sample and also no significant ($p > 0.05$) difference was observed in the antioxidant activity of these two methods for the 2 mm thickness. TPC and AA of TV1 and TH2 for the 2 mm thickness were not found to be significantly ($p > 0.05$) different from those values for freeze-dried samples. For the 4 mm thickness sample, the total phenolics content and antioxidant activity for TV9, TV1, TV8, TV7, TV3, and TV15 were found not to be significantly ($p > 0.05$) different from lyophilized samples. These results indicate that VBD is a promising drying technique and can yield high nutritional value products in less time as compared to FD.

Index words: Muscadine pomace, vacuum belt drying, freeze drying, hot air drying, total phenolics content, antioxidant activity.

Introduction

In the last few decades, the demand for nutritious and healthy foods has increased considerably. Consumers are becoming more aware about the relationship between a healthy diet and lower risk of chronic diseases, like coronary heart disease, cancer, atherosclerosis, and Alzheimer's disease etc. A growing body of evidence suggests that secondary plant metabolites play a critical role in human health. Polyphenolic compounds are considered to be very effective in lowering the risk of many diseases due to their action as antioxidants and a wide range of pharmacological properties, like platelet aggregation inhibitory activity, anticarcinogenic activity, and anti-ulcer activity. Antioxidant activity in organisms defends against Reactive Oxygen Species (ROS) produced as by-product of metabolism and can be of endogenous or of dietary origin

In 2005, over 4 million metric tons of grapes were processed in the USA for wine production, and 56 to 80 thousand metric tons of pomace were generated (Uribe and Restrepo, 2006). Grape pomace is the waste left after processing of wine or juice, and consists mainly of skins, seeds and pulp. Grape seeds and skins are good sources of polyphenolic compounds, especially condensed and hydrolyzable tannins. Grape seeds and skins contain many phenolic acids and flavonoids in addition to anthocyanins (Palma and Taylor, 1999; Souquet and others 1996).

Muscadines (*Vitis rotundifolia*) are an integral part of the southeastern US agriculture. They are well adapted to the warm, humid climate and can be found growing in the wild from Delaware to the Gulf of Mexico and westward from Missouri to Texas. Muscadine berries are borne in loose clusters of 3 to 40, and the fruits are round, 2.5 to 3.75 cm in diameter with thick skins and 5 to 6 oblong seeds per cluster. They are also characterized by a distinguished flavor

and aroma. Muscadines are consumed fresh or used in the processing of wine, juice, jelly, and jam (Olien and Hegwood, 1990). Studies have reported that muscadines are a rich source of phytochemicals like gallic acid, catechins, epicatechin, ellagic acid, myricetin, quercetin, kaempferol, resveratrol, and anthocyanin amongst others, and are distinguishable from most other types of grapes due to the presence of high concentrations of ellagic acid and anthocyanins (*i.e.*, 3,5-diglucosides of delphinidin, cyanidin, petunidin, peonidin, and malvidin) (Lee and Talcott, 2002; Pastrana-Bonilla and others 2003). Muscadines are also known to be an excellent source of dietary fiber. Therefore, consumption of muscadine provides health benefits of dietary fiber in addition to phenolic compounds. Striegler and others (2005) reported that the muscadine industry is currently experiencing its fastest growth in decades, and the future looks excellent due to exploitation of this rich nutritional profile. Muscadine seeds and skins are also a reservoir of polyphenolic compounds. Pastrana-Bonilla and others (2003) reported that the major phenolics present in the skins of muscadines are ellagic acid, myricetin, quercetin, kaempferol, and *trans*-resveratrol with the average values 16.5, 8.4, 1.8, 0.6, and 0.1 mg/100 g of fresh weight (FW), respectively. The major phenolics present in the seeds were gallic acid, (+)-catechin, and (-)-epicatechin with respective average values of 6.9, 558 and 1299 mg/100 g of FW. Average total phenolics content in the seeds and skins were 2179 and 375 mg/g gallic acid equivalent respectively, and antioxidant capacity was 281.3 and 12.8 μ M TEAC/g of FW, respectively. Due to these high concentrations of phenolic compounds and antioxidant activity, muscadines possess many health benefits like lowering blood glucose levels (Banini and others 2006), anti-inflammatory activity of muscadine skin powder (Greenspan and others 2005), anti-carcinogenic, and apoptosis activity of muscadines skin (Yi and others 2005). Presence of high concentrations of phenolic compounds and numerous health benefits makes muscadine seeds and

skins highly desirable for the production of functional food ingredients or nutraceuticals which otherwise after wine making or juice production are considered as waste. Muscadine seeds, skins, and pulp left as by-products after production of wine or juice is called pomace (Moure and others 2001). By-products of wine and juice production are characterized by considerably high contents of phenolic compounds. Pomace recovered after juice extraction or wine production serves as a cheap source of polyphenolics, which can be used as food supplements or in phytochemical production (Alonso and others 2002). Makris and others (2007) reported that wine industry by-products are a very rich source of antioxidant polyphenols as compared with other agri-food solid wastes. Muscadine pomace is highly perishable due to high water activity and moisture content, and therefore dehydration techniques are required to preserve this valuable product.

The a_w of a food is defined as the ratio between the vapor pressure of the food itself when in a completely undisturbed balance with the surrounding air media, and the vapor pressure of distilled water under identical conditions. It is the indicator for microbiologically safe food products; usually food products with water activities below 0.5 are considered risk free of microbiological proliferation. Drying of grape pomace at high temperatures may cause a significant degradation of polyphenols and can also affect antioxidant activity and free-radical scavenging activity (Larrauri and others 1997; Larrauri and others 1998). The two commonly used methods for drying are conventional air drying and freeze drying. Usually air drying is favored due to its low operating cost and comparatively less drying time required than freeze drying. In conventional air drying, high temperatures are required which adversely affects the texture, color, flavor, and nutritional value of products (Schadle and others 1983; Yang and Atallah, 1985). Freeze drying is a more gentle technique which does not require exposure of the

product to high temperatures. It reduces the loss of volatiles and textural changes, and is therefore often employed to dehydrate high-value products (Flink, 1975). The main disadvantage with freeze drying is its high operation cost and the long drying period. Vacuum belt drying (VBD), on the other hand, is a newer method for dehydration. So far VBD has been employed to dehydrate fruits, fruit juice powders, and herbs. Vacuum belt dryers are best suited for slurries. VBD is a continuous method and products dried are of high quality. The drying requires a shorter time and operating costs are low (Wang and others 2007). In VBD due to vacuum, mass transfer is accelerated and hence, drying occurs at a quicker rate. Moreover due to the absence of air while drying, oxidation is prevented and can result in products with high nutritional quality. Limited research has been carried out on VBD to evaluate the volatiles, aroma, and flavor compounds profile of fruits after drying as compared to other conventional drying technologies. Wang and others (2007) reported that VBD resulted in a better volatile profile of banana chips than air drying. No literature is available concerning the effect of vacuum belt drying on the phenolic content and antioxidant capacity as compared to freeze drying and conventional air drying. There is also very scarce data at hand about the effect of different drying technologies on the muscadine pomace. Therefore, the objectives of this research were the following:

1. To compare the efficacy of different time-temperature combinations for vacuum belt drying, and time-temperature as well as air velocity combinations for air drying of muscadine pomace at retaining of polyphenolic compounds with antioxidant activity.
2. To evaluate the impact of vacuum belt dried, hot air dried and lyophilized muscadine pomace samples on the water activity (a_w), moisture content (MC), total phenolics content (TPC), and antioxidant activity (AA).

3. To investigate the impact of pomace disc thickness during FD, VBD, and HAD on a_w , MC, TPC, and AA.

Materials and methods

Chemicals and reagent

All solvents used were of ACS grade, unless otherwise specified. Folin & Ciocalteu's phenol reagent, gallic acid, sodium carbonate, and fungal pectinase were purchased from Sigma Chemical Co. (St. Louis, MO). Ferrous sulfate heptahydrate and Whatman No.1 filter paper were obtained from VWR International (Suwanee, GA). 2,4,6-Tris(2-pyridyl)-*s*-triazine (TPTZ) was acquired from Acros Organics (Geel, Belgium) and ferric chloride hexahydrate was bought from Alfa Aesar (Wardhill, MA).

Juice extraction and pomace collection

Muscadines of the Noble cultivar were collected from Paulk Vineyard (Wray, GA) at the time of optimum harvest in early September 2008. Muscadines were processed within 24 h of harvest and until that time were held under refrigerated conditions. The muscadines were crushed in a grinder (Model M046, Hobart Manufacturing Company, Troy, OH) with an end plate having 1.59 mm diameter holes, and were collected in a plastic container with a food-grade polyethylene liner. Crushed muscadines were then transferred to a steam kettle (TWP 20, Legion Equipment Co., Dallas, PA) to attain a temperature of 35-40°C. Once this temperature range was reached, 3.7 ml/kg of pectinase enzyme (activity 10392 units/ml) was added to increase juice recovery. After holding for 20 min at the set temperature, the muscadine slurry was collected from the steam kettle and was fed into the hopper of the hydraulic fruit press (flückiger Hydraulic CH5646 sins, Bucher, Dottingen, Switzerland). The muscadine slurry was pressurized to 1500 kPa until the juice stopped coming out of the outlet. Juice was collected in a

plastic container with a food-grade polyethylene liner. Pomace was recovered from the hopper, packaged in oxygen-barrier Cryovac bags, vacuum sealed with a Polar 2-40 (Henkelman Vacuum System, s-Hertongenbosch, The Netherlands), and then stored at -30°C until further used. Figure 3.1 depicts the process flow diagram for the sample preparation.

Sample preparation

Before the experiment, the pomace was thawed at 22°C (room temperature) for 12 h. Thawed pomace was ground in a Univex mixer grinder (Model MG8912, Salem, NH) to achieve homogeneity by comminuting the seeds and skins. Pomace was then placed in wax paper according to the number of treatments. Wax paper containing pomace was passed through a roller pin machine (Model S-18-BM04458, Moline Machiner Ltd., Duluth, MN) to achieve two thicknesses of 2 ± 0.3 mm and 4 ± 0.5 mm. Then, each pomace sheet was cut into a disc by a circular cutter of 8.9-cm diameter. Pomace was cut in a disc of uniform thickness and shape to determine the effects of thickness on drying. The mass of the disc was dependent on the thickness: for the 4 ± 0.5 mm thick disc, the mass was 26 ± 1 g and for the disc with 2 ± 0.3 mm thickness, the mass was 14 ± 1 g.

Vacuum belt dryer

A laboratory-scale vacuum belt dryer (Zwag, CH-5312 Zchokke Wartman Ltd. Bucher, Dottingen, Switzerland) was employed for carrying out the experiments. A schematic diagram of vacuum belt dryer is illustrated in Figure 3.2. A vacuum belt dryer is a semi-continuous system consisting of housing with a conveyer belt that runs with the product over three heating plates and a cooling plate. Above the conveyer belt there is additional heating plate that runs continuous over the other three, and radiates energy to the product. The other three heating zones transfer heat to the product by conduction. Different temperature combinations were set

for all heating zones and different speeds of the conveyer belt were employed according to the desired drying time by operating the touch screen LCD control panel. Vacuum was maintained in the system by a vacuum pump which gave of 3 to 8 kPa absolute pressure.

Hot air dryer

Hot air drying was performed in a tray dryer (armfield, tray dryer). The system consisted of a heating filament, fan, and drying tray on which the product was placed. Air velocity and temperature were controlled by two separate knobs. Air velocity and temperature were measured by a handheld Anemometer (Fisher) and a Fluke 52II thermometer (Fluke Corporation, WA, USA), respectively. A weighing balance was attached to the drying tray to observe the rate of moisture evaporation.

Drying treatments

All of the three batches were exposed to the same set of drying treatment to ensure the repeatability of the experiments.

Vacuum belt drying process

In the vacuum belt dryer, the three zones were set at different temperatures and the fourth was set at the same temperature as that of zone 3. Temperatures were set from low to high as the product moved from through the system. The vacuum was set at an absolute pressure in the range of 3 to 8 kPa. Temperatures were set at eight different combinations ranging from 60 to 120°C. Two different belt speeds were used to give residence times of 90 and 60 min for each temperature combination. For each temperature combination, there were four variations and overall 32 combinations of treatments. The different combinations are shown in Table 3.1.

Hot air drying process

The hot air drying process was performed at two different temperatures of 70°C and 80°C; three air velocities of 0.2 m/s, 0.4 m/s, and 0.6 m/s; and two different residence time of 240 min and 180 min. Therefore, there were a total of 24 trials; the combinations are as given in Table 3.2.

Freeze drying process

Freeze drying, or lyophilization, was performed using a Freezemobile 25 SL Unitop 600L (Virtis Company, Gardiner, NY) at an absolute pressure of 2 kPa, heating plate temperature of 30°C, and condenser temperature -40°C. The drying time to reduce the moisture content to 1.2-1.8% was determined to be 14-16 h.

Moisture content and water activity

The moisture content is defined as the ratio of the amount of water in the food to the amount of dry solids and is expressed as follows:

$$X_t = (W_t - F_s) / F_s$$

where, W_t is the total weight of the wet material at time t ; F_s is the weight of the dry solids; and X_t is the moisture content expressed as weight of water/weight of dry solids. The moisture content in “dried” pomace samples was determined according to the Association of Official Analytical Chemists (AOAC) Method 934.06 (AOAC 1990) with some modification. Briefly, 5g of ground pomace samples were weighed into a pre-dried aluminum pan and then placed in a vacuum oven (Model G05053-10, Cole Parmer Stable Temp, Cole Parmer Instrument Company, Vernon Hills, IL), set at 70°C and 44 kPa of absolute pressure for 24 h.

The water activity of samples was determined using an Aqualab sensor CX-2 (Decagon Devices Inc, Pullman, WA) set at 25°C.

Extraction of polyphenolic compounds

Extraction of phenolics from dried pomace samples was accomplished with 80% (v/v) acetone. Briefly, dried pomace discs were first ground in a commercial coffee mill (Kitchen aid, St. Joseph, MI). Then, 80% acetone was added to pomace powder in a 250-ml Erlenmeyer flask at an 8:1 (v/w) solvent-to-solids ratio. The flask was placed in a Gyrotory water bath shaker, (Model G76, New Brunswick Scientific Co. Inc., New Brunswick, NJ) set at 40°C for 30 min. After this period, the flask was removed from the water bath and the supernatant was filtered through Whatman No. 1 filter paper into a second Erlenmeyer flask. The residue cake from the filter paper was scrapped off and added back to the first flask (with sediments) to which another portion of 80% (v/v) acetone was added. This procedure was repeated for a total of 3 times, to ensure maximum recovery of phenolics from the sample. The pooled acetonic extract was then transferred to a 250-ml round bottom flask and attached to a Büchi Rotovapor R-210 connected to a V-700 vacuum pump and vacuum controller V-850 (Büchi Cooperation, New Castle, DE) at 40°C and 82 kPa of absolute pressure, to remove the organic solvent. The resultant aqueous residue containing phenolics was poured into a 20-ml aluminum pan and stored overnight at -80°C. The next day, the sample was lyophilized with a set absolute pressure of 94.5 kPa, heating plate temperature of 30°C, and condenser temperature of -40°C for 12 h to obtain the dry phenolics extract.

Total phenolics content

The total phenolics content of lyophilized extracts was determined by a colorimetric assay as described by Swain and Hillis (1959) and modified by Naczk and Shahidi (1989). Briefly, 0.5ml of extract (1:1000 (w/v) dilution in 1:1(v/v) water and methanol) was pipetted into a test tube containing 8 ml of deionized water. Then 0.5 ml of Folin-Ciocalteu's phenol reagent

was added followed by 1 ml of saturated sodium carbonate. The test tube was thoroughly vortexed for 10 s and was left covered at 22°C (*i.e.*, room temperature) for 45 min. Absorbance was read at 750 nm using a FLUOstar Omega 96-well microplate reader (BMG Labtech, Durham, NC). For the construction of the calibration curve, 80 mg of gallic acid was dissolved in a 1 L volumetric flask with 25 ml of methanol and then filled to mark with deionized water. Working solutions of varying concentrations were prepared from this stock solution.

Antioxidant capacity determinations

The antioxidant capacity of extracts was estimated by the FRAP assay as described by Pulido, Bravo, and Saura-Calixto (2000) with some modifications. Briefly, the FRAP reagent was prepared fresh by adding 5 ml of a 10 mmol/L TPTZ solution in 40 mmol/L HCl and 5 ml of 20 mmol/L $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and 50 ml of 0.3 mol/l acetate buffer at pH 3.6. The FRAP reagent was kept at 37°C in a water bath (Isotemp Model 205, Fisher Scientific, Suwanee, GA) until further use. Aqueous solutions of known Fe(II) concentrations in the range of 100 to 2400 $\mu\text{mol/L}$ ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) were used to construct the calibration curve. The test sample used was of 1:1000(w/v) dilution in (1:1 (v/v)) water/methanol solution. Eighteen hundred μL of the 37°C FRAP reagent were mixed with 180 μL of deionized water and 60 μL of the test sample or blank.

Absorbance readings of samples were recorded at 595 nm every 20 s for 30 min using the FLUOstar Omega 96-well microplate reader set at a constant temperature of 37°C.

Experimental design

Muscadines were divided into three batches to replicate the experiment for the processing conditions. Each batch was treated by the same set of drying treatments, extraction procedure, and the same standards were employed for each assay. Determinations of total phenolic content and antioxidant activity were performed twice to check for repeatability of the results.

Statistical analysis

Data was analyzed using the Statistical Analysis System software (SAS[®]) 9.1 version. A two-way ANOVA was performed. Treatment and thickness were considered as two factors. F test was performed to check the effect of thickness on the treatments and interaction. To verify the effect of treatment on the response factors (water activity (a_w), moisture content (MC), total phenolics content (TPC) and antioxidant activity (AA), multiple comparison was done using protected LSD method.

Results and discussion

Effect of thickness and treatment on water activity and moisture content

The moisture content of untreated muscadine pomace was determined to range from 62 to 68%, and water activity (a_w) ranged from 0.97 to 0.98. The two thicknesses of drying layers were found to have a significant effect on a_w (p value < 0.05). The a_w for 2 mm thickness varied between 0.1 to 0.28 for VBD samples, 0.18 to 0.36 for HAD samples, but for freeze dried samples, a_w was found to be 0.16. Higher water activities were observed for discs of 4 mm for all treatments. The a_w ranged from 0.12 to 0.83 in VBD, 0.19 to 0.48 for HAD, and 0.18 for the lyophilized sample (Table 3.3). The lowest a_w was observed in TV5 (2 mm). Results indicate that with the VBD technique lower a_w was obtained in comparison to HAD and FD for both thicknesses.

Moisture content (MC) of dried products was also significantly affected by disc thickness and treatment. The MC ranged from 0.57 to 5.69% for 2 mm thickness pomace disc. In 4 mm thickness disc, the MC was observed from 1.21 to 41.61%.

Large variation in data signifies the impact of treatment on a_w and moisture content. Intensity of heat treatment resulted in lower a_w and moisture content. Moreover, it can also be

concluded from the observation that the same treatment with different thickness discs resulted in different water activities and moisture contents. From the statistical analysis it can be concluded that the treatments significantly ($p < 0.05$) affected a_w and MC.

The interaction of thickness and treatment (thickness*treatment) was also found to be significant ($p < 0.05$) for both a_w and MC.

This observation was expected as many studies show that a_w and MC of a product are significantly affected by the surface area exposed, mass of the product, temperature, air velocity, vacuum, and initial temperature of the product (Liu and others 2009). Velic and others (2004) reported that with increase in air flow velocity higher heat transfer to the product was observed. Higher a_w and MC was observed for the 4 mm thickness due to the fact that in VBD heat transfer to the product occurs by conduction from the heat plate located below conveyer belt and by radiation from the heat plate positioned above product. In both cases, moisture starts evaporating from the area of contact and the heat energy penetrates to the interior of the product. Mass transfer occurs in capillary action depending on the intensity of the temperature. Often, the exposure to high temperature causes the first layer or the immediate contact surface to dry at faster rate than the interior of the product and this causes the “case-hardening”, that is the inner and center of the product remains high in MC and outer surface dries. In the samples of 2 mm thickness lower MC was observed for the same treatment. The conduction and radiation from the heating plates takes place at a faster rate to all the particles as the moisture has to travel small distance for evaporation. In 2 mm thickness, larger surface area is exposed for a given volume of the product as compared to 4 mm thick discs. Similar observation were found for the HAD, where heat transfer to the product was due to convection from hot air. Figure 3.3 shows the drying curve for the samples TH1 and TH2 where lowest MC was observed for TH1 (2 mm) and

TH2 (4 mm) showed the highest moisture content. This graph elucidates that in 4 mm thickness sample, the moisture evaporation took place at a slower rate than the sample of 2 mm thickness. Results found in the current study were similar to other researches. César Kapseu (2007) concluded that the highest drying rate was observed in kernel paste for 4 mm thickness followed by 8 mm thickness. Similar results were observed by Sankat and others (1996), where the authors observed that the increase in drying temperature and decrease in thickness of the banana slices resulted in higher drying rate. The authors also concluded that air drying above 70°C in banana slices may result in case hardening. Ertekin and Yaldiz (2004) reported that there was 104% and about 294% increase in drying time when thickness of the eggplant slice was increased from 0.635 cm to 1.27 and 2.54 cm, respectively. Also the drying time increased to 38.44 h from 4.39 h when the eggplant was dried at 30°C instead of 70°C. Akpinar and others (2003) reported that in convective drying of potato slices of thickness 12.5 mm and 8 mm, 460–740 min and 280–520 min of drying time was required respectively, to reach 11% moisture content. This indicates that thinner slices were dried at a faster rate.

The MC and a_w found for each treatment is shown in Tables 3.4 and 3.5. All of the treatments for 2 mm thickness disc resulted in MC below 8%. Some of the treatments with 4 mm thickness resulted in exceptionally high MC up to 30% but the treatments with 90 min drying time resulted in MC below 10%.

Moisture sorption isotherms for 2 mm thick and 4 mm thick dried pomace discs are shown in figure 3.4 and 3.5 respectively, at temperature of 22°C. Moisture content and water activity have very complex relation but generally increase with one another and follows a non linear trend. In this study that a_w and MC followed a clear exponential trend for 4 mm thick disc

but the relation between a_w and MC in 2 mm thickness pomace discs was not very clear although it also followed an exponential trend.

Effect of thickness and treatment on the total phenolic content (TPC) and antioxidant activity (AA) of muscadine pomace

For the polyphenolic compound extraction, considering the MC of the dried samples, the ratio of acetone: water (v/v) (extraction solvent) was modified but no significant difference was found after modification. Therefore, the same extraction solvent was used.

All the values are dry weight basis and reported as per gram extract.

Total phenolics content (TPC)

Thickness was found to possess no significant effect ($p > 0.05$) on the TPC of the dried samples. Nevertheless, the TPC data indicates that within similar treatments, majority of the times disc with 4 mm thickness demonstrated better TPC retention than 2 mm thickness disc. This observation can be valid due to the fact that polyphenolic compounds are heat sensitive (Mejia-Meza and others 2008; Wojdylo and others 2009). In discs, with 4 mm thickness heat transfer takes place at a slower rate and therefore polyphenolic compounds are affected less than the discs with 2 mm thickness. In the latter, rapid heat transfer takes place, increasing the probability of destruction of polyphenolics with time.

The TPC for the muscadine pomace was found to be 613 $\mu\text{mol GA eq/g extract}$. Freeze dried samples showed slightly lower values of TPC (583 and 608 $\mu\text{mol GA eq/g extract}$ for 2 mm and 4 mm thickness samples respectively) than the fresh pomace. This observation was in line with many others studies done on TPC of untreated fruit compared to freeze drying. Asami and others (2003) reported that highest levels of TPC were consistently found in unprocessed fruit followed by freeze dried sample and air dried samples of marionberry, strawberry and corn.

Similarly, Wojdylo and others (2009) observed a slight decrease in TPC of freeze dried strawberry (Kent cultivar) as compared to fresh fruit. Mejia-Meza and others (2008) also observed a slightly lower retention of TPC as compared to fresh blueberry. The possible reason for these lower values can be degradation of light sensitive polyphenolic compounds as the sample for freeze drying is exposed to light and UV rays for longer time than raw sample. In freeze drying samples are exposed to low temperatures and the whole process takes place in vacuum pressure. Since, there was no significant effect of thickness on TPC of samples, so further evaluation of drying techniques for different thickness sample were compared separately. In 2 mm thickness sample TPC of TV2, TV1 and TH2 were not significantly ($p > 0.05$) different from TF1. TV2 gave higher values of TPC (642 $\mu\text{mol GA eq/g}$) than TF1. TPC of TH5 was found to be 429 $\mu\text{mol GA eq/g}$, which was lowest among all the drying treatments. TV12 gave the lowest TPC value of 453 $\mu\text{mol GA eq/g}$, for VBD samples (Table 3.4).

In 4 mm thick discs larger number of samples were found to be not significantly different ($p > 0.05$) from TF1 in TPC and those were TV9, TV1, TV8, TV7, TV3 and TV15 (data shown in Table 3.5). None of the hot air dried samples were found to be comparable to TF1 in TPC retention. Similar to observation for TPC of 2 mm thickness sample, in 4 mm thick samples lowest TPC was found in HAD sample, TH8 (449 $\mu\text{mol GA eq/g}$).

Different drying treatments resulted in varying TPC of the dried product. In general, the highest total phenolic content was observed for FD, followed by VBD (most of the treatments) and lowest phenolic content preservation was observed in HAD sample. These results are in agreement with other research findings, suggesting that polyphenols are heat liable and prolonged heat treatment causes a damage of polyphenolic compounds (Mejia-Meza et al., 2008; Lin and others 1998). Julkunen-Tiitto and Sorsa, (2001) observed a destruction tannins and

flavonoids due to drying treatments. They also found that glycosides after decomposition formed the corresponding aglycones. Similar result was reported by Chan and others (2009) that upon thermal drying there was a major loss of TPC of ginger leaves. Michalczyk and others (2009) concluded that due to long exposure time and high temperature polyphenol and anthocyanin content in bilberries was significantly reduced. de Ancos and others (2000) suggested that anthocyanin and other polyphenolics compounds may degrade depending upon many factors other than just heat treatment like activity of polyphenol oxidase, organic acid content, sugar concentration and pH. During long thermal treatments polyphenol oxidase gets longer time to oxidize the polyphenolics present and causes reduction in TPC. In freeze drying, product is exposed to maximum temperature of 30°C under vacuum condition. Freezing prior to freeze drying lead to formation of ice crystals in tissues. Creation of ice crystals can lead to puncturing of cell structures and cell wall which may release the polyphenolic components in matrix and makes them easier to extract (Asami and others 2003). These factors results in highest retention of polyphenolic content. In VBD, dehydration of the sample takes place in vacuum, which eliminates the risk of loss of polyphenolics by air oxidation, and also enhances the heat penetration providing a constant internal temperature, resulting in faster drying (60 or 90 min). Kwok and others (2004) suggested that due to constant internal temperature, a better retention of anthocyanins can be observed. Reduced drying time does not cause significant damage to polyphenolic compounds. In hot air drying chances of degradation of polyphenolic compounds increases due to the fact that processing takes place in an open system. Moreover, high temperatures for longer time duration results in higher destruction of polyphenolic compound (Wojdylo and others 2009). Yousif and others (2000) reported that higher susceptibility of oxidation was observed in air dried oregano as compared to vacuum microwave drying and

freeze drying. In air drying due to the presence of heat and oxygen, enzymatic activity of polyphenol oxidase is favored.

This observation of high TPC in TV2 TH2, TV9, TV1, TV8, TV7, TV3 and TV15 can be possibly due to two reasons (a) that the temperature was not very high and exposure was for a short duration of time. Therefore, polyphenolic compounds were preserved in drying. Many of the researchers have reported that at low temperature like below 70°C the polyphenolic components of fruits are preserved. Nindo and others (2003) reported that at 60°C in heated air or microwave spouted bed drying method, high phenolic compounds were retained. Similarly, Larrauri and other (1997) in their study on effect drying temperature on red grape pomace concluded that dehydration temperature around 60°C resulted in no significant change of polyphenolic compounds as compared to freeze dried pomace. (b) In fruits and vegetables phenolic polyphenolic compounds concentration is higher in outer parts of cell rather than vacuoles (Haard and Chism, 1976), Olien and Hegwood, (1990) suggested that since phenolic acids are mainly attached to the carbohydrates and proteins of the cell, so possibly during thermal processing release of polyphenolic compounds occur due to breakdown of cellular constituents and bonds. Therefore, the released polyphenolic compounds become easily available for extraction. Kim and others (2006) reported that higher TPC values were observed for whole grape seed extract at thermal treatment of 150°C for 40 min and powdered grape seed extract at 100°C for 10 min as compared to non treated control samples. A increase in TPC of citrus peel was observed upon heating treatment of 150°C for 60 min as compared to control sample (Piga and others 2003). Chang and others (2006) reported a significant increase in TPC of processed tomatoes as compared to fresh, indicating that drying treatments resulted in an increase in TPC. Although disruption cell may also result in release of oxidative and hydrolytic

enzymes, that are capable of oxidizing polyphenolics present in sample but exposure to high temperature can simultaneously result in denaturation of these enzymes thereby protecting polyphenolic compounds (Wojdylo and others 2009).

Antioxidant activity

The results for antioxidant activity of dried muscadine pomace discs suggest that thickness of the disc had no significant effect ($p > 0.05$). Interaction of thickness and treatment was also not significantly different for antioxidant activity. The data obtained for treatment thickness Vs antioxidant activity still suggests that out of 29 treatments, in 21 treatments higher antioxidant activity was found higher for 4 mm thick samples, therefore it can be expected that due to low heat transfer the antioxidant activity of disc with 4 mm thickness was preserved under same treatment condition with lesser thickness. These results were expected due to several findings by researchers which advocate that temperature can cause decrease in antioxidant activity of the product and also at high temperature destruction of polyphenolics occurs which can eventually result in reduction of antioxidant activity (Katsube and others 2009).

Antioxidant activity of the raw muscadine pomace was determined to be 2.32 mmol Fe^{2+} eq/g extract. Freeze dried (TF1) samples with thickness 2 and 4 mm showed the antioxidant activity of 2.22 and 2.29 mmol Fe^{2+} eq/g, respectively. A very slight decrease in antioxidant activity of TF sample was observed as compared to unprocessed pomace. These results are in accordance with results of Kwok and others (2004), concluding that after freeze drying the antioxidant activity was decreased as compared to fresh Saskatoon berries. Wojdylo and others (2009) reported that lower antioxidant activity of freeze dried strawberries were found for both varieties (Kent, Elsanta) as compared to fresh strawberries.

In assessment of different drying treatments for 2 mm thick discs, TV1, TV2, TH11, TH10, TV10, TV6, TV5, TH12, TV14, TV8, TH6, TV3, TV15, TH1, TV7, TH9, TV4 and TH4 were found to significantly not different ($p > 0.05$) from TF1. TV1, TV2 and TH11 showed to have higher antioxidant activity than freeze dried sample (data shown in Tables 3.1 and 3.2). The lowest antioxidant activity was found in TH5 ($1.62 \text{ mmol Fe}^{2+}\text{eq/g}$). In 4 mm thick samples, 24 treatments showed to be not significantly ($p > 0.05$) different from TF1 in respect to antioxidant activity. The treatments were TV9, TV7, TV3, TV1, TH10, TV5, TH5, TV4, TH3, TV11, TV8, TV13, TH12, TV10, TH11, TV15, TH9, TV12, TV2, TH4, TV6, TV16 and TH8, out of which TV9, TV7, TV3 demonstrated higher AA than lyophilized samples.

In respect to retention of TPC and AA, treatments TV1, TV2 and TH2 for 2 mm thickness and for 4 mm thickness treatments TV9, TV1, TV8, TV7, TV3 and TV 15 resulted in no significant ($p > 0.05$) difference from lyophilized sample. Moreover it can be observed that treatment TV1 with any of the thickness was found to be not significantly different from lyophilized samples.

It is well known that polyphenolic compound due to their hydrogen electron donating characteristics behave as strong antioxidant. Larger number of treatments resulted in being significantly not different from freeze drying in respect to AA as compared to the results of total phenolic content, where only around 8 treatments were found comparable to lyophilized samples. The results of this study are in agreement with other researches about effect of drying temperature and technique on antioxidant activity. Schmidt and others (2006) proposed that antioxidants are usually not damaged during drying process. Chang and others (2006) reported that high antioxidant activity was observed for hot air dried tomato samples than fresh ones. Many other products like tomato juice and sweet corn have shown an increase of antioxidant

activity as compared to untreated samples (Dewanto and others 2002). Similar was observed by Piga and others (2003), that after hot air drying antioxidant activity prunes was increased as compared to fresh plums. The increase in antioxidant activity can be possible due to formation of non nutrient antioxidants. Although, during drying process the natural antioxidants of a sample can be destroyed but the overall antioxidant properties of food could be enhanced by development of new antioxidant during processing (Nicoli and others 1997). This behavior can be due to two reasons (a) at high temperature stabilization, formation of new compounds as a result of Maillard reaction. These compounds are called meladoins or MRPs, have high antioxidant activity and often, work by chain breaking mechanism, moreover the antioxidant capacity of MRPs do not compensate for phenolic compound loss (Manzocco and others 2000; Yilmaz and Toledo, 2005; Morales and Jimenez-Perez, 2001) (b) during polyphenols oxidation products formed in intermediate stages have shown to posses greater antioxidant activity than initially present, although these intermediate compounds are temporary (Manzocco et al., 2000; Cheigh and others 1995).

Conclusions

Muscadine pomace discs, dehydrated by different treatments and techniques (FD, VBD, and HAD), resulted in different MC, a_w , TPC, and AA. With certain treatment conditions in VBD, the drying time was reduced to 60 or 90 min from the 14-16 h of freeze drying to give products of similar TPC, AA, a_w , and MC. Treatments TV1, TV2, TV3, TV7, TV8, TV9, TV15, and TH2 have shown a comparable total phenolics content and antioxidant activity to the lyophilized sample. TV1, TV9, and TV3 were found not to be significantly ($p > 0.05$) different from freeze-dried samples in any of the studied aspects. Nevertheless, treatment TV2, TV7, TV8, TV15, and TH2 can also be utilized for the production of antioxidant-rich functional food

ingredients although both the a_w and MC were not as low as the FD samples, but was below 0.4 and 8 to 7% respectively. From these findings, it can be concluded that vacuum belt drying under the previously mentioned treatment conditions was capable of preserving polyphenolics and antioxidant activity at a higher level than HAD treatment and was equivalent to that by FD. It will not be wise to consider all the treatments that gave comparable antioxidant activity to freeze drying, as explained earlier that antioxidation capability may not be permanent as well as MRPs production may result in undesirable physical and chemical changes. VBD treatment can prove to be very advantageous, as the drying time was considerably reduced to get a high nutritional value dry product with comparable a_w , MC, TPC and AA and also due to decrease in drying time the operating cost for this equipment will be low, moreover, this is a continuous process which can result in higher yield in shorter time.

Muscadine pomace is rich in polyphenolic content and antioxidant activity. Processing with right technology can result in a cheap functional food ingredient or nutraceutical and can help in combating many chronic diseases by being a part of normal diet. Utilization of muscadine pomace will also result in elimination and utilization of winery and juice processing by-product.

More research is needed in this area to have a better understanding of the ongoing phenomena during the drying process that results in these observations. A analysis of to confirm formation of MRPs can be one of the future direction Sensory evaluation of the VBD and FD dried product can also help in assessing the acceptability of these products.

Table 3.1: Treatment conditions for vacuum belt drying

TREATMENT	THICKNESS (mm)	ZONE 1 (°C)	ZONE 2 (°C)	ZONE 3 (°C)	ZONE 4 (°C)	TIME (min)
TV1	4	60	80	100	100	90
TV1	2	60	80	100	100	90
TV2	4	60	80	100	100	60
TV2	2	60	80	100	100	60
TV3	4	60	90	120	120	90
TV3	2	60	90	120	120	90
TV4	4	60	90	120	120	60
TV4	2	60	90	120	120	60
TV5	4	70	95	120	120	90
TV5	2	70	95	120	120	90
TV6	4	70	95	120	120	60
TV6	2	70	95	120	120	60
TV7	4	70	90	110	110	90
TV7	2	70	90	110	110	90
TV8	4	70	90	110	110	60
TV8	2	70	90	110	110	60
TV9	4	80	90	100	100	90
TV9	2	80	90	100	100	90
TV10	4	80	90	100	100	60
TV10	2	80	90	100	100	60
TV11	4	80	95	110	110	90
TV11	2	80	95	110	110	90
TV12	4	80	95	110	110	60
TV12	2	80	95	110	110	60
TV13	4	90	100	110	110	90
TV13	2	90	100	110	110	90
TV14	4	90	100	110	110	60
TV14	2	90	100	110	110	60
TV15	4	90	105	120	120	90
TV15	2	90	105	120	120	90
TV16	4	90	105	120	120	60
TV16	2	90	105	120	120	60

Table 3.2: Treatment conditions for hot air drying

TREATMENT	THICKNESS (mm)	TEMPERATURE (°C)	AIR VELOCITY m/s	TIME (min)
TH1	4	70	0.6	240
TH1	2	70	0.6	240
TH2	4	70	0.6	180
TH2	2	70	0.6	180
TH3	4	70	0.4	240
TH3	2	70	0.4	240
TH4	4	70	0.4	180
TH4	2	70	0.4	180
TH5	4	70	0.2	240
TH5	2	70	0.2	240
TH6	4	70	0.2	180
TH6	2	70	0.2	180
TH7	4	80	0.6	240
TH7	2	80	0.6	240
TH8	4	80	0.6	180
TH8	2	80	0.6	180
TH9	4	80	0.4	240
TH9	2	80	0.4	240
TH10	4	80	0.4	180
TH10	2	80	0.4	180
TH11	4	80	0.2	240
TH11	2	80	0.2	240
TH12	4	80	0.2	180
TH12	2	80	0.2	180

Table 3.3: Water activity and moisture content range for different drying treatments for different thickness pomace discs.

Samples	2 mm disc		4 mm disc	
	a_w	MC	a_w	MC
FD	0.16	1.76%	0.18	2%
VBD	0.1-0.28	0.57-3.70%	0.12-0.83	1.21-41.6%
HAD	0.18-0.36	2.13-5.69%	0.19-0.48	3.6-9.05%

Table3.4: Treatments Vs mean of water activity (a_w), moisture content (MC), total phenolics content (TPC), antioxidant activity (AA) for 4mm thick muscadine pomace disc.

TREATMENTS	a_w	MC (%)	TPC ($\mu\text{molGAE/gm ext}$)	AA ($\text{mmolFe}^{2+}\text{E/gmext}$)
TF1	0.18 ^m	2.01 ^l	607.53 ^a	2.30 ^{a,b,c,d}
TV1	0.28 ^{h,i,j}	3.49 ^{j,k}	577.93 ^a	2.29 ^{a,b,c,d}
TV2	0.87 ^a	41.6 ^{1,a}	468.05 ^m	2.02 ^{c,d,e,f}
TV3	0.17 ^m	1.53 ^{l,m}	564.84 ^a	2.36 ^{a,b,c}
TV4	0.74 ^{a,b}	17.10 ^c	537.50 ^{c,g,d}	2.20 ^{a,b,c,d,e}
TV5	0.12 ⁿ	1.21 ^m	555.28 ^{c,d}	2.26 ^{a,b,c,d}
TV6	0.78 ^{a,b}	20.34 ^{b,c}	486.21 ^{m,l}	1.93 ^{d,e,f}
TV7	0.23 ^{l,k}	1.86 ^l	566.80 ^a	2.52 ^{a,b}
TV8	0.83 ^{a,b}	29.95 ^b	570.84 ^a	2.17 ^{a,b,c,d,e}
TV9	0.19 ^{l,m}	3.70 ^{j,k}	603.93 ^a	2.54 ^a
TV10	0.77 ^{a,b}	17.43 ^c	513.58 ^{c,d,g,h}	2.15 ^{b,c,d,e}
TV11	0.23 ^{j,k}	3.54 ^{j,k}	547.94 ^{c,d}	2.17 ^{a,b,c,d,e}
TV12	0.74 ^{a,b}	18.33 ^c	502.29 ^{g,h}	2.08 ^{c,d,e,f}
TV13	0.30 ^{g,h,i}	4.33 ^{h,l,j}	549.68 ^{c,d}	2.16 ^{a,b,c,d,e}
TV14	0.7 ^{b,c}	14.90 ^{c,d}	494.34 ^{g,h,l}	1.86 ^{e,f}
TV15	0.19 ^{l,m}	2.82 ^k	559.57 ^a	2.14 ^{b,c,d,e}
TV16	0.6 ^c	11.19 ^{d,e}	514.68 ^{c,d,h,g}	1.93 ^{d,e,f}
TH1	0.31 ^{g,h,i}	4.80 ^{g,h,l,j}	465.52 ^{m,l}	1.71 ^f
TH2	0.48 ^d	9.05 ^{e,f}	488.94 ^{h,l}	1.84 ^{e,f}
TH3	0.43 ^{d,e}	5.44 ^{g,h}	535.72 ^{c,d,h,g}	2.18 ^{a,b,c,d,e}
TH4	0.43 ^{d,e}	6.59 ^{f,g}	501.15 ^{h,g}	2.02 ^{c,d,e,f}
TH5	0.29 ^{h,i}	3.6 ^{l,j,k}	508.58 ^{d,h,g}	2.20 ^{a,b,c,d,e}
TH6	0.47 ^d	8.86 ^{e,f}	489.09 ^{h,l}	1.82 ^{e,f}
TH7	0.26 ^{i,j,k}	3.92 ^{i,j,k}	449.31 ^{m,l}	1.84 ^{f,e}
TH8	0.33 ^{f,g,h}	5.65 ^{g,h}	438.35 ^m	1.91 ^{d,e,f}
TH9	0.39 ^{e,f}	4.81 ^{g,h,l,j}	513.44 ^{c,d,h,g}	2.14 ^{b,c,d,e}
TH10	0.30 ^{h,i}	5.2 ^{l,g,h,i}	534.28 ^{c,d,h,g}	2.29 ^{a,b,c,d}
TH11	0.28 ^{i,j,k}	4.79 ^{i,h,j}	547.49 ^{c,d}	2.14 ^{b,c,d,e}
TH12	0.36 ^{e,f,g}	6.09 ^{g,h}	535.70 ^{c,d,h,g}	2.15 ^{b,c,d,e}

Note: mean with same alphabet are not significantly different from other means of respective column

Table3.5: Treatments Vs mean of water activity (a_w), moisture content (MC), total phenolics content (TPC), antioxidant activity (AA) for 2mm thick muscadine pomace disc.

TREATMENTS	a_w	MC (%)	TPC ($\mu\text{molGAE/gmext}$)	AA ($\text{mmolFe}^{2+}\text{E/gmext}$)
TF1	0.16 ^{h,l,j}	1.76 ^{g,h,i}	582.52 ^b	2.21 ^a
TV1	0.19 ^{f,h,g}	1.65 ^{h,l,j}	599.89 ^b	2.38 ^a
TV2	0.26 ^{b,c,d}	3.70 ^b	642.10 ^b	2.27 ^{a,b}
TV3	0.13 ^{j,k,l}	1.10 ^{k,l}	532.10 ^{d,e,f}	2.02 ^{b,c,d,e,g}
TV4	0.16 ^{h,l,j}	1.17 ^{j,k,l}	471.17 ^{i,j}	1.96 ^{c,d,e,f,g,h,i}
TV5	0.10 ^m	0.57 ^m	523.21 ^{e,f}	2.11 ^{a,b,c,d}
TV6	0.19 ^{f,g,h}	1.87 ^{g,h,i}	508.74 ^{f,g,h}	2.11 ^{a,b,c,d}
TV7	0.13 ^{k,l}	0.96 ^l	528.93 ^{e,f}	1.98 ^{b,c,d,e,f,g,h}
TV8	0.28 ^b	2.92 ^{b,c,d,e,f}	538.69 ^{e,d,f}	2.04 ^{b,c,d,e,f,g}
TV9	0.15 ^{i,j,k}	1.90 ^{g,h,i}	467.80 ^{i,j}	1.74 ^{g,h,l,j}
TV10	0.26 ^{b,c}	3.41 ^{b,c,d}	548.93 ^{e,d}	2.13 ^{a,b,c}
TV11	0.12 ^{k,l}	1.51 ^{j,k,l}	478.80 ^{h,l,j}	1.81 ^{d,e,f,g,h,i}
TV12	0.17 ^{h,i}	2.50 ^{c,d,e,f,g}	453.44 ^{j,k}	1.66 ^{i,j}
TV13	0.13 ^{k,l}	1.01 ^{k,l}	490.41 ^{g,h,i}	1.79 ^{e,f,g,h,l,j}
TV14	0.22 ^{d,e,f,g}	2.48 ^{d,e,f,g,h}	518.26 ^{e,g,h}	2.08 ^{a,b,c,d,e,f}
TV15	0.12 ^{l,m}	0.91 ^l	521.11 ^{e,f,g}	2.00 ^{b,c,d,e,f,g,h}
TV16	1.88 ^{j,k,l}	1.87 ^{g,h,i}	464.90 ^{i,j}	1.6 ^{h,l,j}
TH1	0.19 ^{f,g,h}	2.13 ^{f,g,h}	538.14 ^{e,d,f}	1.99 ^{b,c,d,e,f,g,h}
TH2	0.24 ^{b,c,d,e}	3.55 ^{b,c}	561.68 ^{c,d}	2.21 ^{a,b,c}
TH3	0.26 ^{b,c,d,e}	3.88 ^b	479.70 ^{h,i,j}	1.78 ^{f,g,h,l,j}
TH4	0.36 ^a	5.69 ^a	479.29 ^{h,l,j}	1.93 ^{c,d,e,f,g,h,l,j}
TH5	0.19 ^{f,g,h}	3.47 ^{b,c,d,e}	429.35 ^k	1.63 ^j
TH6	0.21 ^{d,e,f,g}	4.24 ^{a,b}	523.18 ^{e,f}	2.03 ^{b,c,d,e,f,g}
TH7	0.18 ^{g,h,i}	2.26 ^{f,g,h}	452.72 ^{j,k}	1.75 ^{g,h,l,j}
TH8	0.25 ^{b,c,d,e}	2.95 ^{b,c,d,e,f}	469.69 ^{i,j}	1.77 ^{g,h,l,j}
TH9	0.22 ^{i,j,k}	2.39 ^{d,e,f,g,h}	466.07 ^{i,j}	1.98 ^{b,c,d,e,f,g,h}
TH10	0.21 ^{e,f,g}	3.11 ^{b,c,d,e,f}	541.15 ^{d,e}	2.15 ^{a,b,c}
TH11	0.20 ^{f,g,h}	2.3 ^{e,f,g,h}	533.54 ^{d,e,f}	2.22 ^{a,b,c}
TH12	0.21 ^{d,e,f,g}	3.1 ^{b,c,d,e,f}	547.47 ^{d,e}	2.10 ^{a,b,c,d,e}

Note: mean with same alphabet are not significantly different from other means of respective column

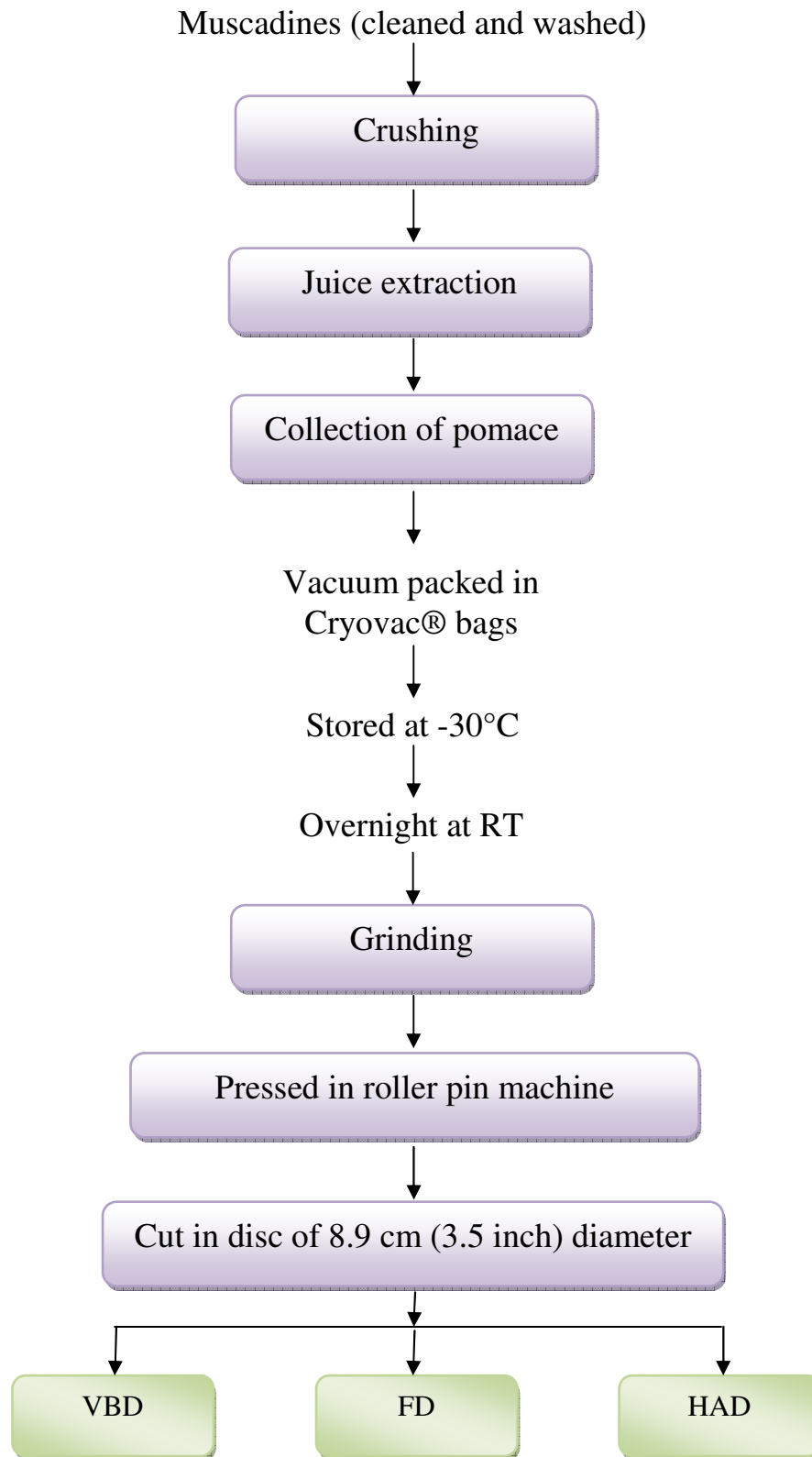


Figure 3.1: Process flow diagram of the experiment.

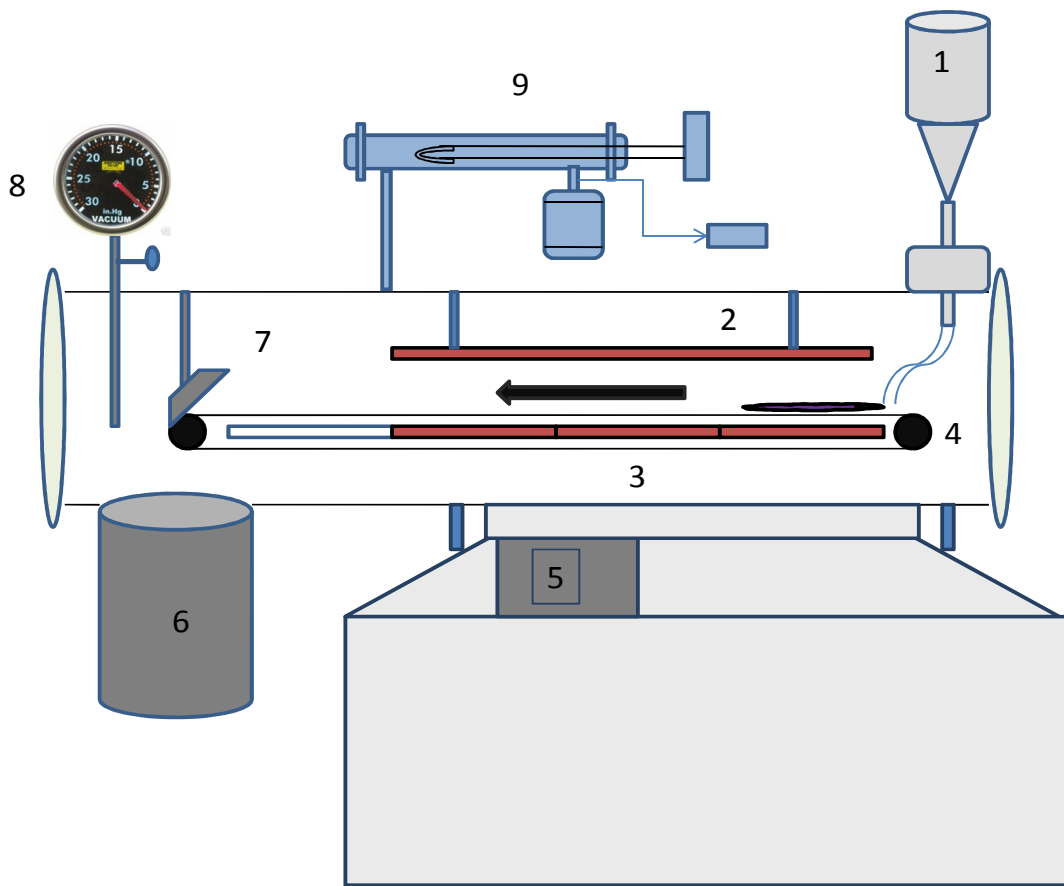


Figure 3.2: Schematic of Vacuum belt dryer. 1-feed tank, 2-radiating heating plate, 3-conducting heating plate, 4-conveyor belt, 5-control panel, 6- collecting tank, 7-scraper, 8-vacuum gauge.

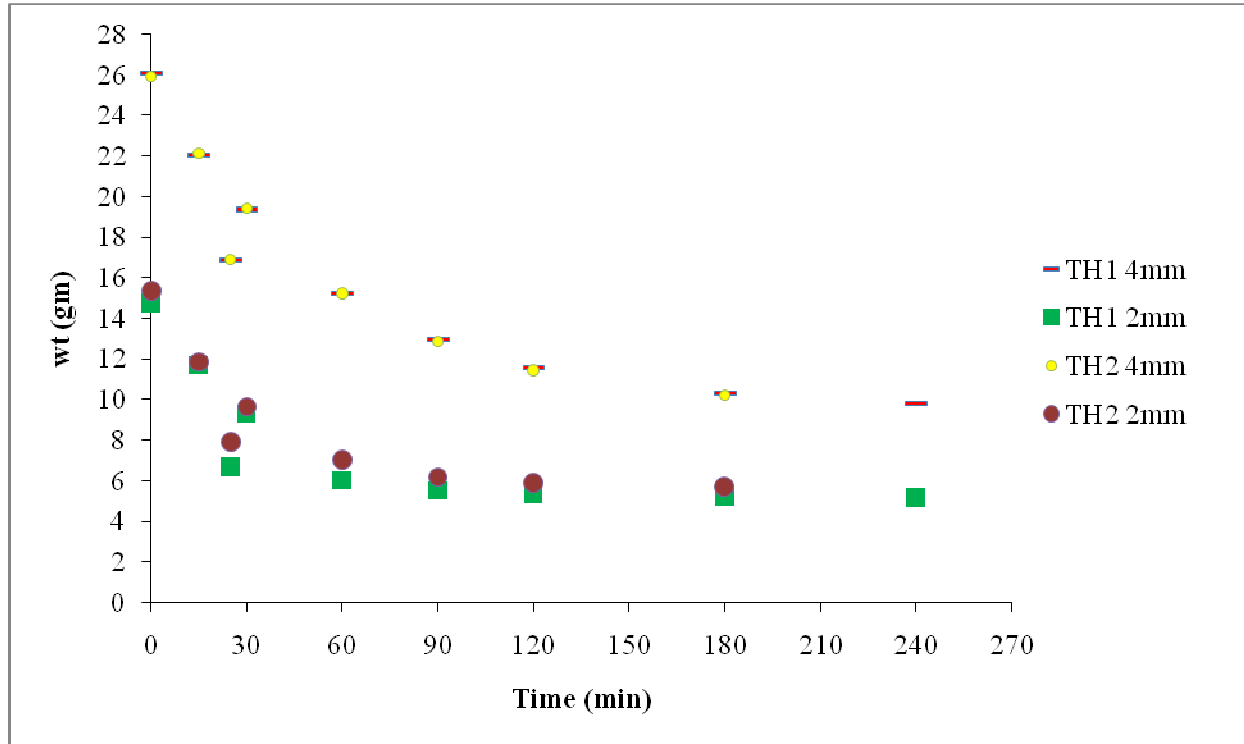


Figure 3.3: Drying curve for TH1 and TH2 for 2 and 4mm thick muscadine pomace discs.

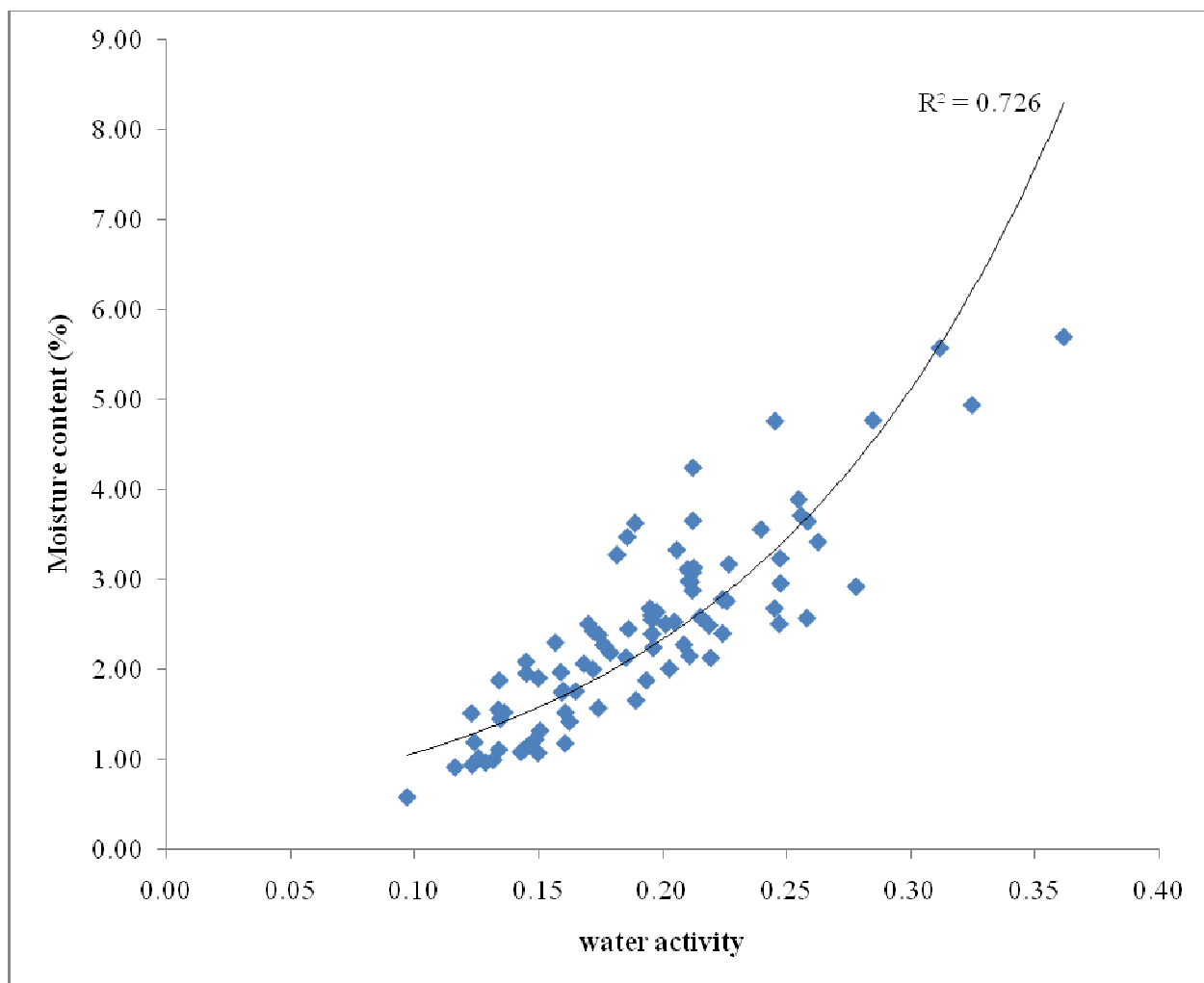


Figure 3.4: Moisture sorption isotherm for 2mm thick pomace discs.

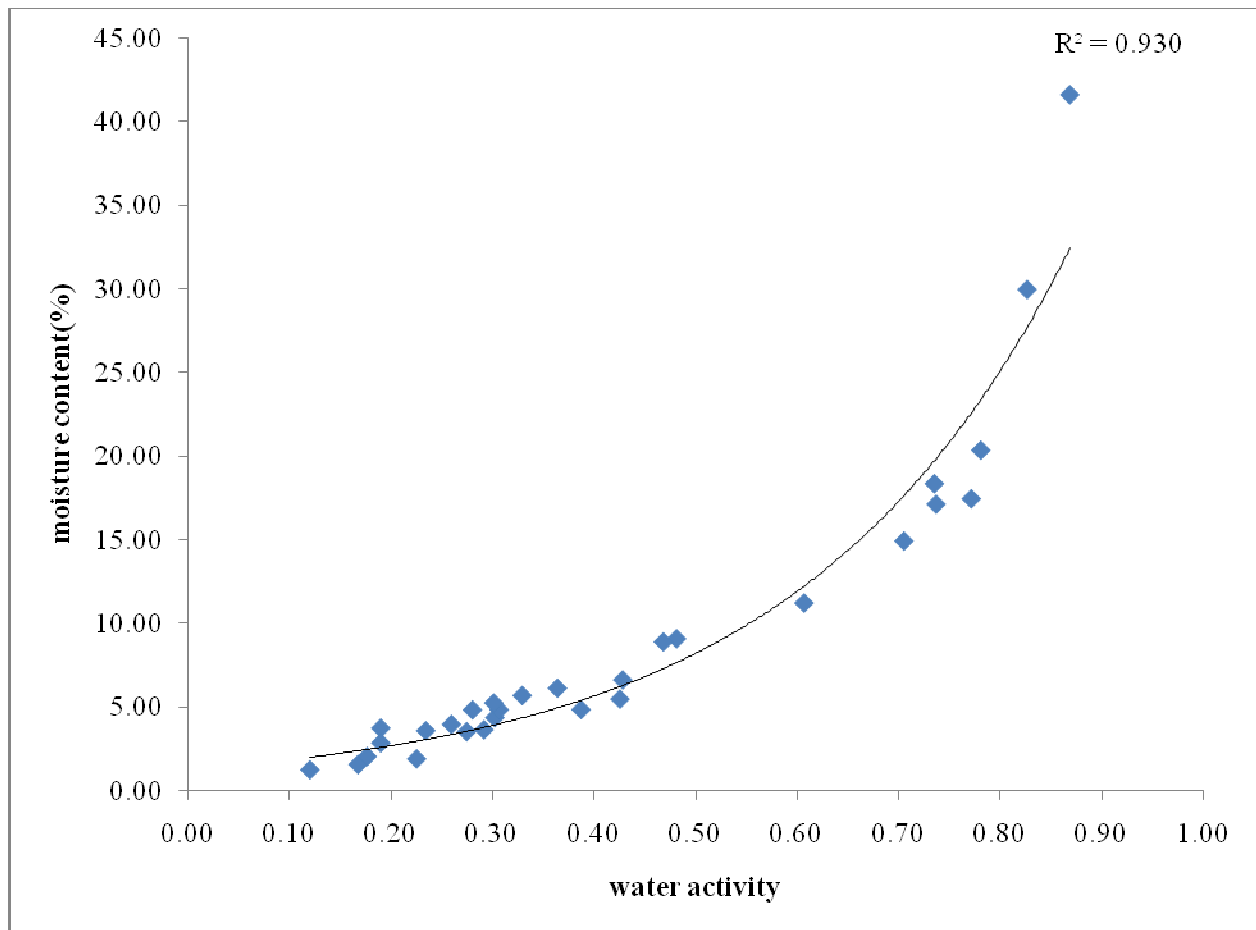


Figure 3.5: Moisture sorption isotherm for 4mm thick pomace disc.

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CHAPTER 4

SUMMARY AND CONCLUSIONS

The three drying technologies were compared and contrasted for drying of muscadine pomace to produce an antioxidant-rich functional food ingredient/nutraceutical. Vacuum belt drying, hot air drying were compared with freeze drying in respect to 4 parameters, water activity, moisture content, total phenolics content and antioxidant activity. Different time-temperature and time-temperature-air velocity combinations were compared for VBD and HAD, respectively. Effect of thickness (2 mm, and 4 mm) on drying, and retention of polyphenolic compounds as well as antioxidant activity was also compared for all the drying treatments. A significant ($p < 0.05$) effect of thickness was found on water activity and moisture content of dried product. Interaction of thickness and treatment was also found to be significant ($p < 0.05$) for moisture content and water activity. No significant ($p > 0.05$) effect of thickness on total phenolics content and antioxidant activity of dried muscadine pomace was observed. Moisture content and water activity below 8% and 0.4 respectively, was found for most of the treatments, few of the exceptions where pomace discs with 4mm thickness.

In respect to retention of TPC and AA, treatments TV1, TV2, and TH2 with 2 mm thickness and for 4 mm thickness treatments TV9, TV1, TV8, TV7, TV3, and TV 15 resulted in no significant difference from lyophilized sample with 2 and 4mm thickness., Moreover it was observed that treatment TV1 with any of the thickness was found to be not significantly different from freeze dried samples.

From this research it can be concluded that vacuum belt drying is a promising technique for preservation of nutritional components present in raw material. In 90 min drying time

moisture content was reduced from 65% to below 5% and a_w from 0.97 to below 0.4, for both thicknesses and therefore, it can be compared to freeze drying and can result in nutritionally rich product in a very less drying time, which can eventually result in reduction of operating cost.

More research is needed in the area of vacuum belt drying, to have a better scientific understanding of these observations. The study on effect of drying technique on quantity and quality on each individual polyphenolic compounds can also elucidate the observations.

APPENDIX A

Bar graphs with Standard deviations

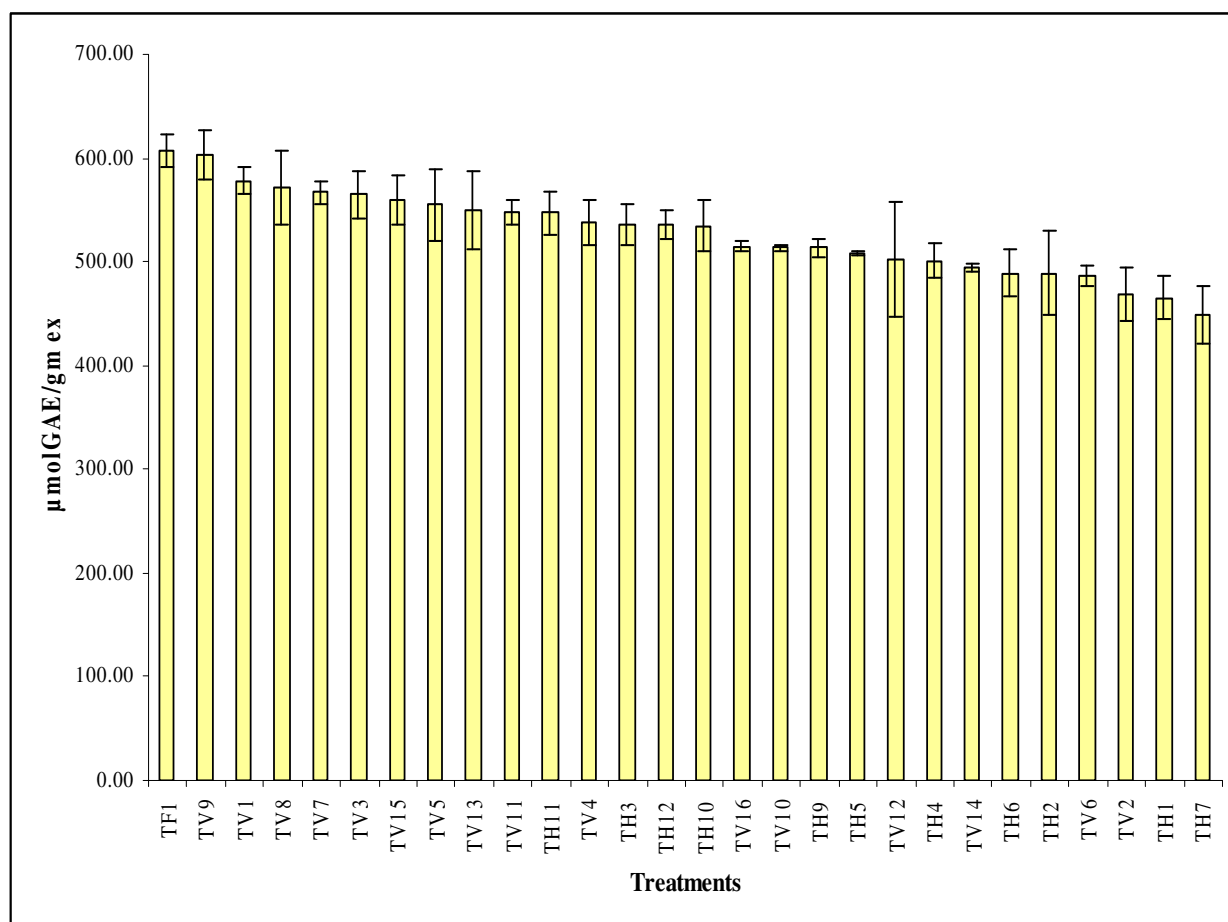


Figure A.1: Bar graph showing means total phenolics content Vs treatment for 4mm thick muscadine pomace disc.

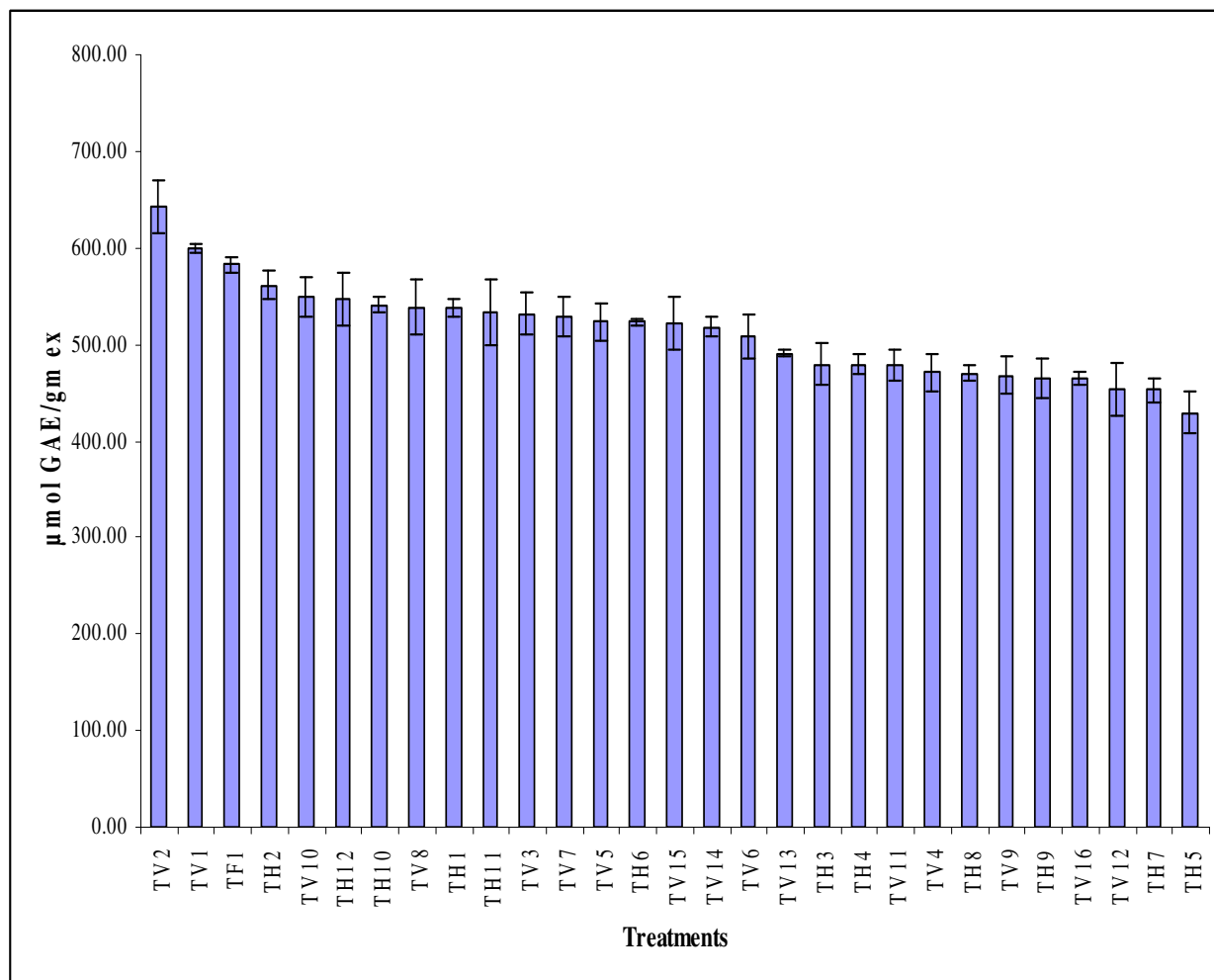


Figure A.2: Bar graph showing means total phenolics content Vs treatment for 2mm thick muscadine pomace disc.

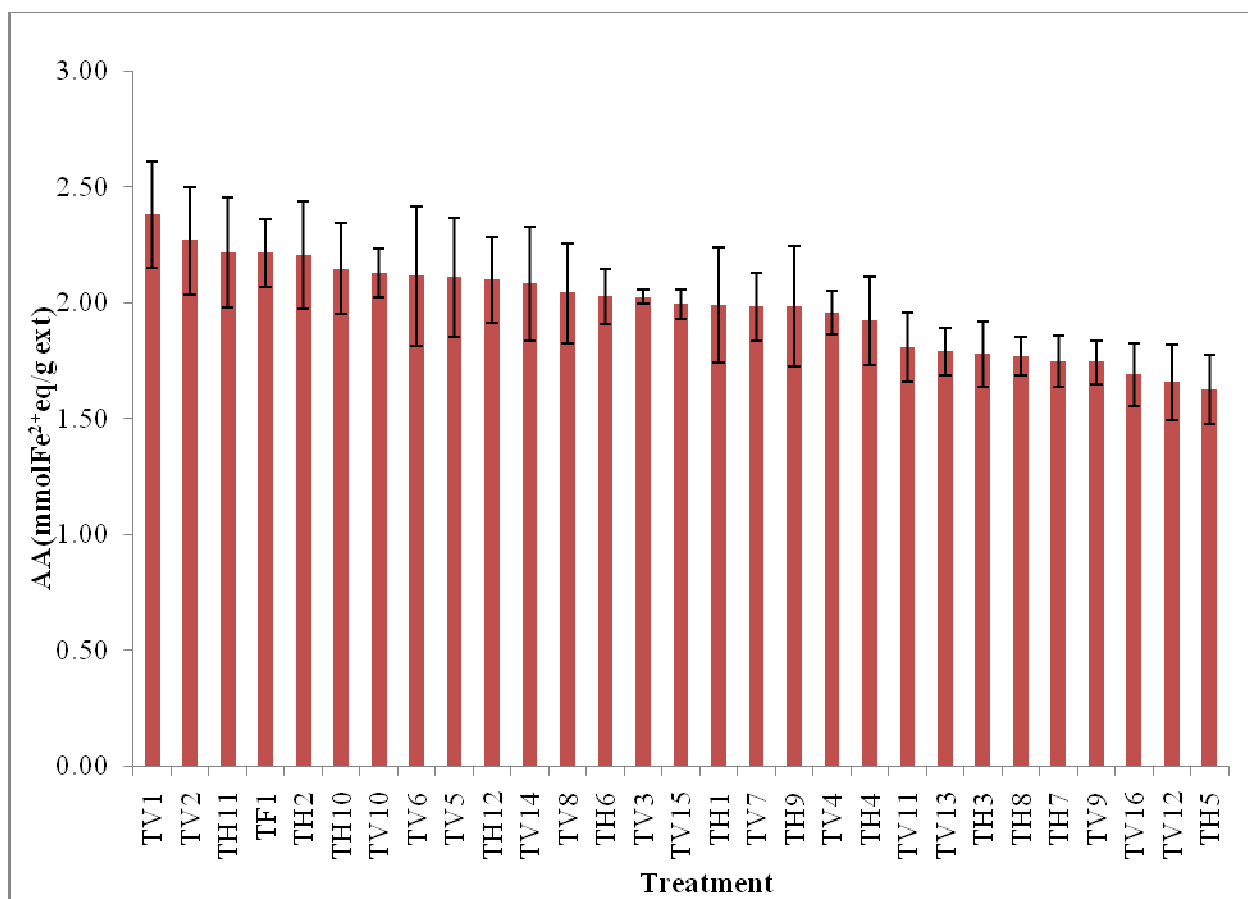


Figure A.3: Bar graph showing mean antioxidant activity Vs treatment for 2mm thick muscadine pomace disc.

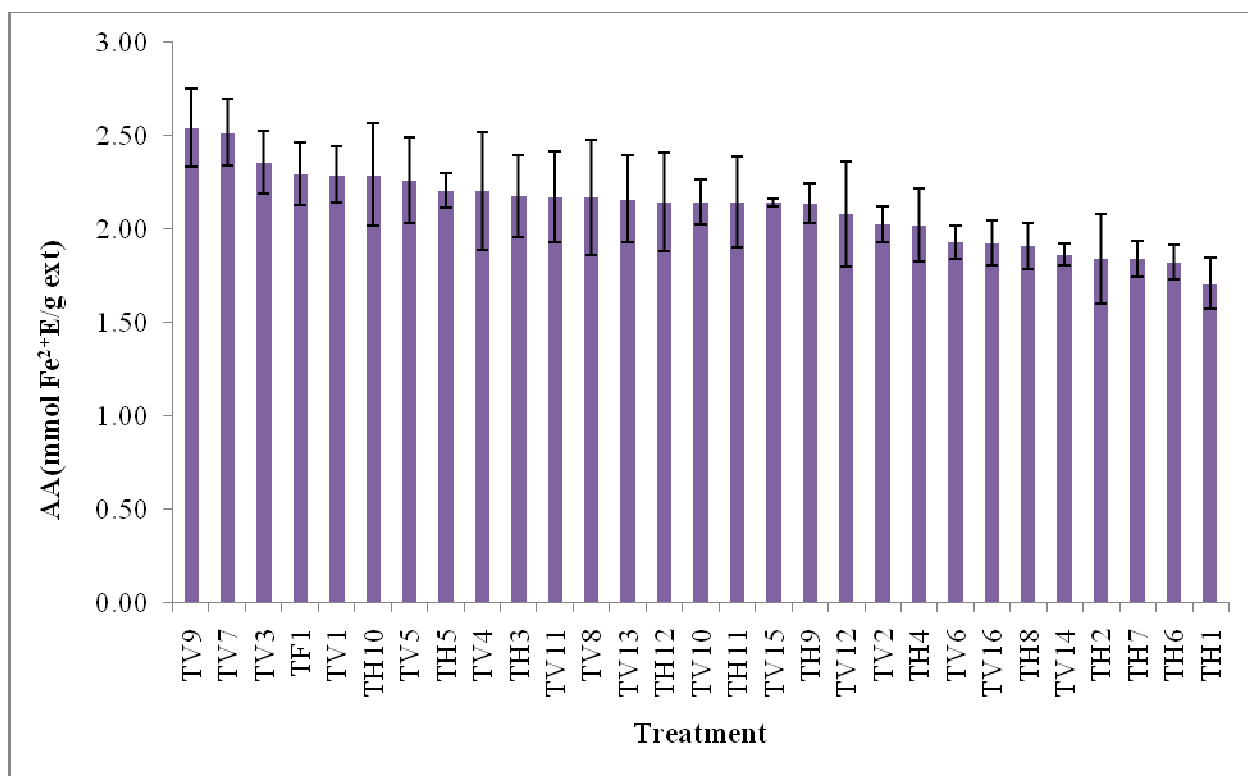


Figure A.4: Bar graph showing mean antioxidant activity Vs treatment for 4mm thick muscadine pomace disc