

**EFFECT OF VARIOUS DRYING TECHNIQUES ON QUALITY  
PROPERTIES OF ORGANIC BLUEBERRY FRUIT**

**M.Sc. Thesis**

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**Food Engineering  
Gaziantep University**

**Supervisor**

**Prof. Dr. Medeni MASKAN**

**Co-Supervisor**

**Assoc. Prof. Dr. K. Bülent BELİBAĞLI**

**by**

**Gülşah GÖK**

**December 2019**



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Name of the Student : Gülşah GÖK

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Approval of the Graduate School of Natural and Applied Sciences.

Prof. Dr. Ahmet Necmeddin YAZICI  
Director

I certify that this thesis satisfies all the requirements as a thesis for the degree of  
Master of Science.

Prof. Dr. Medeni MASKAN  
Head of Department

This is to certify that we have read this thesis and that in our consensus/majority  
opinion it is fully adequate, in scope and quality, as a thesis for the degree of Master  
of Science.

Assoc. Prof. Dr. K. Bülent BELİBAĞLI  
Co-Supervisor

Prof. Dr. Medeni MASKAN  
Supervisor

Examining Committee Members

Prof. Dr. Medeni MASKAN

Prof. Dr. Fahrettin GÖĞÜŞ

Asst. Prof. Dr. Hidayet SAĞLAM

\_\_\_\_\_

\_\_\_\_\_

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**Gülşah GÖK**

## **ABSTRACT**

### **EFFECT OF VARIOUS DRYING TECHNIQUES ON QUALITY PROPERTIES OF ORGANIC BLUEBERRY FRUIT**

**GÖK, Gülşah**

**M.Sc. in Food Engineering**

**Supervisor: Prof. Dr. Medeni MASKAN**

**Co-Supervisor: Assoc. Prof. Dr. K. Bülent BELİBAĞLI**

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The effects of convectional air and microwave-convectional (hybrid) drying methods, and ultrasound and osmotic dehydration pretreatment on drying characteristics and some quality parameters of organic blueberry were investigated. Convectional drying was performed at 60, 70, and 80 °C temperatures. Hybrid drying was performed at 150, 180 and 210 W power levels at 60 °C constant air temperature. Blueberries were exposed to a simultaneous ultrasound and osmotic dehydration pretreatment. An organic apple juice concentrate (70 °Brix) was used as the osmotic medium. The ultrasound pretreatment extended the drying process. Hybrid drying resulted in shorter drying times compared to air drying. Increasing temperature in air drying and microwave power in hybrid drying increased the drying rates. The best fitting model for all drying conditions was determined as Midilli et al. model. Higher total phenolics content (TPC) and antioxidant activity (AA) levels were obtained by the hybrid drying compared to hot air method. The highest TPC and AA values were obtained at 180 W hybrid drying, while the lowest values were obtained at 70 °C air drying. Application of ultrasound and osmotic dehydration treatment led to a significant decrease in TPC and AA. Color values were generally similar to each other. As a conclusion, hybrid drying can be considered as an alternative dehydration method for the drying of blueberries.

**Key Words:** Organic Blueberry, Drying, Ultrasound, Phenolics Content, Microwave

## ÖZET

### ÇEŞİTLİ KURUTMA TEKNİKLERİNİN ORGANİK MAVİ YEMİŞ MEYVESİNİN KALİTE ÖZELLİKLERİ ÜZERİNE ETKİSİ

**GÖK, Gülşah**

**Yüksek Lisans Tezi, Gıda Mühendisliği**

**Danışman: Prof. Dr. Medeni MASKAN**

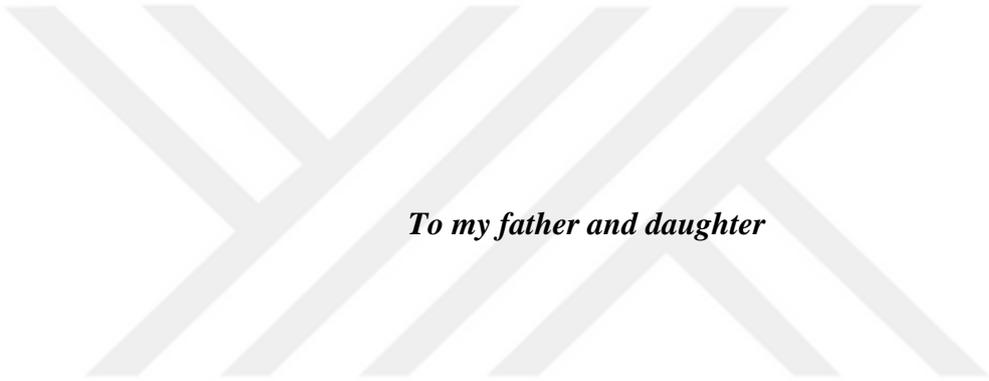
**İkinci Danışman: Dr. Öğr. Üyesi K. Bülent BELİBAĞLI**

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Konveksiyonel (havalı) ve mikrodalga-konveksiyonel (hibrit) kurutma yöntemlerinin ve ultrason ve ozmotik dehidrasyon önışleminin, organik yabanmersininin kurutma özellikleri ve bazı kalite parametreleri üzerindeki etkileri araştırıldı. Konveksiyonel kurutma 60, 70 ve 80 °C sıcaklıklarında yapıldı. Hibrit kurutma 60 °C sabit hava sıcaklığında, 150, 180 ve 210 W güç seviyelerinde uygulandı. Yabanmersinleri eşzamanlı bir ultrason ve ozmotik dehidrasyon önışlemine tabi tutuldu. Ozmotik ortam olarak organik elma suyu konsantresi (70 °Briks) kullanıldı. Ultrason önışlemi kurutma sürecini uzattı. Hibrit kurutma havalı kurutmaya göre daha kısa kurutma süreleri ile sonuçlandı. Havalı kurutmada sıcaklığın, hibrit kurutmada mikrodalga gücünün artması kurutma hızlarını artırdı. Tüm kurutma koşullarına en iyi uyan model Midilli et al. olarak belirlendi. Hibrit kurutmada, havalı kurutma yöntemine göre daha yüksek toplam fenolik madde içeriği (TPC) ve antioksidant aktivite (AA) değerleri elde edildi. En düşük TPC ve AA değerleri havalı kurutmada 70 °C sıcaklıkta elde edilirken, en yüksek değerler 180 W hibrit kurutmada elde edildi. Ultrason ve ozmotik dehidrasyon işleminin uygulanması TPC ve AA değerlerini anlamlı ölçüde düşürdü. Renk değerleri genel olarak birbirine benzer bulundu. Sonuç olarak, hibrit kurutma yabanmersininin kurutulması için alternatif bir dehidrasyon yöntemi olarak değerlendirilebilir.

**Anahtar Kelimeler:** Organik Yabanmersini, Kurutma, Ultrason, Fenolik İçeriği, Mikrodalga



*To my father and daughter*

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## LIST OF ABBREVIATIONS

<b>AA</b>	Antioxidant Activity
<b>ANOVA</b>	Analysis of Variance
<b>AOAC</b>	Association of Official Analytical Chemists
<b>dm</b>	Dry matter
<b>FAO</b>	Food Agriculture Organization
<b>FW</b>	Fresh Weight
<b>GAE</b>	Gallic Acid Equivalent
<b>H&amp;P</b>	Handerson and Pabis Model
<b>Logar</b>	Logarithmic Model
<b>LSD</b>	Least Significant Differences
<b>mod. Log</b>	Modified Logistic Model
<b>RMSE</b>	Root Mean Square Error
<b>TPC</b>	Total Phenolic Content

## CHAPTER 1

### INTRODUCTION

Recently, foods with not only nutritional benefits but also with health improving properties and disease prevention potential are popular among consumers. Fruits provide bioactive compounds and nutrients offering important health benefits (Chen and Martynenko, 2018). Among fruits, berries such as raspberries, blackberries, strawberries and blueberries, are considered to be very important in terms of their antioxidant capacity (Araujo-Díaz et al., 2017). Blueberry, which is the main concern of this study, is a North American fruit which belongs to the family *Ericaceae* and genus *Vaccinium*. Polyphenolic compounds and especially anthocyanins in blueberry provide antioxidant effects (Sinha et al., 2012). Blueberry is called a “longevity fruit” because it possesses high antioxidant capacity, having an important role in decreasing the risk of several diseases (Reque et al., 2014) such as cancer, cardiovascular disease or Alzheimer's disease (Mohideen et al., 2015). The functional properties of blueberry make it commercially important. In addition to high phenolic and anthocyanins content, blueberry contains vitamins and essential minerals. It is anticarcinogenic, anti-inflammatory, antihypertensive, and hypoglycemic (Fonseca et al., 2018).

Blueberry is a seasonal fruit with a short harvesting period and a short storage life (six days under refrigeration conditions). Therefore, they are preserved by dehydration, canning or freezing technologies (Stojanovic and Silva, 2007). Blueberry processing should maintain the bio-active ingredients, which are important for the consumer acceptability, as much as possible. These are very sensitive elements, easily destroyed by any mechanical, physical or chemical treatment. For that reason, the processing requires an extensive knowledge to be able to find suitable methods and conditions. Drying is an agreeable processing method performed by conventional and modern techniques, or combination of these (Michalska and Łysiak, 2015). The purpose is to decrease the water activity and prolong the shelf life (Reque et al., 2016).

Drying equipment and conditions are determined according to the characteristics and drying behaviour of the food material in addition to final product requirements (George et al., 2004). Most of the dryers used in the industry are convective type in which hot air is used to transfer the heat. Drying is considered as an energy intensive operation and conventional drying operations may cause poor product quality, therefore an important technological progress in drying techniques, equipments and pretreatment methods has been made lately (Moses et al., 2014). Ultrasound assisted and microwave assisted drying techniques are among the common ones.

Blueberries contain a waxy skin layer which hinders the moisture transfer and consequently the drying process. Chemical dipping, mechanical and thermal treatments are used to overcome this difficulty (George et al., 2004). There are a number of studies concerning this problem (Azoubel and Murr, 2016; Grabowski and Marcotte, 2003; Vásquez-Parra et al., 2013). Solutions of potassium carbonate with olive oil or fatty acid ethyl esters (e.g. ethyl oleate) are used for chemical dipping most frequently. These combinations cause synergistic reactions to alter the structure of the skin and decrease the moisture impermeability (Vásquez-Parra et al., 2013).

Hot air drying is the most commonly used method for fruit preservation (Zielinska et al., 2015) because the process is comparatively low-cost (Zielinska and Michalska, 2016). However, it has several drawbacks such as long drying times and undesirable changes in color, texture and nutritional content (Zielinska et al., 2015). Besides, drying time may be even longer in the case of waxy skinned fruits such as blueberry (Zielinska and Michalska, 2016). Use of elevated temperatures may help increasing the moisture diffusivity, but in that case the product quality may be compromised (Zielinska et al., 2015).

Microwave drying where the heating is volumetric can be an alternative to speed up the process (Zielinska and Michalska, 2016). Yet it may not be advantageous when it is applied by itself due to the non-uniform heating. The samples may be overheated and may suffer from textural damage (Horuz et al., 2017). Combination methods can be a solution to this problem and commonly, microwave drying is combined with hot air drying (hybrid drying). Here, water is removed from the inside of the material by microwaves and hot air helps removing the water on the surface. Hence, the drying rate is increased and better quality product can be obtained (Horuz et al., 2017).

Microwave-assisted hot air drying of various fruits has been studied by several authors (Contreras et al., 2008; Horuz et al., 2017; İzli et al., 2014; Maskan, 2001).

Total drying time can be shortened by means of some pretreatment applications. By this way, the initial moisture content of the material may be decreased or the surface structure of the material may be altered to ease the water movement (Fernandes et al., 2008 b). In osmotic dehydration pretreatment, the fruits are partially dehydrated and they are further dried with a proper drying method. Osmotic dehydration is carried out by soaking the fruits in a concentrated liquid medium for a specific time. After this step, usually the fruits are dried by air drying method; however, microwave or microwave-air combination methods can be applied as well. It has been indicated that the drying rate is accelerated and products with better quality are obtained by these techniques (Zhang et al., 2006).

Ultrasound treatment is another pretreatment method for fruit drying. It has been a popular subject to contribute the drying process and product quality (Mothibe et al., 2011). Ultrasound alters the properties of foods by inducing cavitation and sponge effects (Nowacka et al., 2018). Application of ultrasound can promote the mass transfer rate of osmotic dehydration where this rate is usually low (Emam-djomeh et al., 2018). In ultrasound-assisted osmotic dehydration, the ultrasound medium is an osmotic solution. The mass transfer is improved by means of the microscopic channels produced inside the capillaries of the material (Mothibe et al., 2011). The influence of osmotic dehydration, ultrasound and their combination has been shown to have different effects on different fruits (Fernandes et al., 2009). The discrepancy in results of ultrasound and osmosis pretreatment for different fruits requires more investigations on this matter.

Concerning the importance of blueberry consumption and the difficulties in the blueberry drying process, there are several purposes of this study: (1) to produce an organic blueberry product dried with suitable and effective pretreatment and drying techniques, which retains bio-active ingredients and nutritional value as much as possible; (2) to examine the effects of chemical dipping and ultrasound-assisted osmotic dehydration pretreatments on drying characteristics and product quality in terms of total phenolics content, antioxidant capacity, color, and overall appearance;

and (3) to compare conventional air drying and microwave-assisted air (hybrid) drying methods for blueberry drying.



## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Blueberry

Most of the fruits belonging to the genus *Vaccinium* contain high amount of phytonutrients. *Vaccinium* plants are characteristically short to medium height, multi-year bushes that generally bear highly pigmented berries. There are more than 500 species in this genus consisting of blueberries, cranberries, lingonberries, bilberries and huckleberries (Terry, 2011).

Blueberries (Figure 2.1) are popular fruits that have health improving nutrients and high economic value. They contain a great deal of antioxidants such as anthocyanins which contribute health and provide protection against many illnesses like memory loss, cancer, cardiovascular disease, urinary disease, vision problems and aging (Shi et al., 2008 a). In addition to the health promoting properties of blueberries, the sweet taste, aromatic flavor, high dietary fiber, and low fat content also make this fruit noticeable (Zhao, 2007).

Blueberries (*Vaccinium corymbosum* L.) are sensitive fruits with a favorable taste (Celejewska et al., 2018). The fruit typically has blue, blue-black, or purple color. It has a wax layer on the surface called “bloom”, so the berry may seem light-colored (Zhao, 2007).

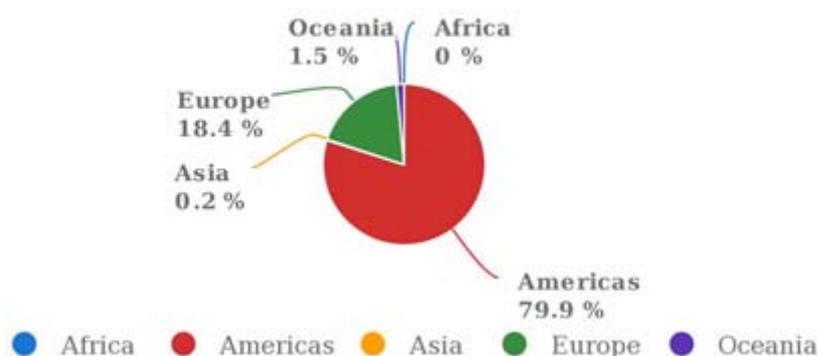


**Figure 2.1** Picture of blueberry

Blueberry grows well on acidic and sandy soil with high organic matter. It requires a soil pH between 4.5 and 5.0, and an adequate supply of moisture during growth period (Sinha et al., 2012). The plant blossoms in spring and the fruit development varies according to the cultivar, weather conditions and plant vigour. The sugar content increases during fruit development and it reaches to nearly 15% at full growth. Berry gets bigger in size even after its color becomes blue mostly because of water uptake (Zhao, 2007).

Blueberry (*Vaccinium* sp., family *Ericaceae*) has nearly 400 species (Zhao, 2007). Highbush (*V. corymbosum* L.), lowbush (*V. angustifolium* Aiton) and rabbiteye (*V. virgatum* Aiton) blueberries are commercially important species (Terry, 2011).

The most harvested blueberry is highbush blueberry, which accounts for over three fourths of the world's blueberry production (Andrew D., 2017). Northern highbush blueberry gives high-quality fruit and it is resistant to lower temperatures, therefore it is the most common blueberry species in the world (Michalska and Lysiak, 2015). Northern highbush blueberries are widespread in the northern parts of the world. Typical cultivars are Bluecrop, Jersey, Bluejay, Rubel, Elliott, Duke, Bluejay, Earliblue, Reka, Draper, Aurora, and Liberty. Southern highbush type is generally grown in warmer parts of the Northern and Southern Hemispheres (Zhao, 2007). Production of blueberry by region can be seen from Figure 2.2.



**Figure 2.2** Production of blueberry by region in 2019 (FAO)

The main blueberry production in Turkey takes place in the Black Sea and Marmara regions for northern highbush types. Highbush and rabbiteye blueberries are frequently cultivated in the southern part of Turkey and western part of Black Sea Region (Çelik, 2016).

Turkey is an important country in berry cultivation. Although wild blueberries used to be consumed particularly in the Black Sea Region, breeding studies resulted in growing of cultivated berries in large areas (Koca and Karadeniz, 2009). It is expected that commercial blueberry production will increase significantly in Turkey (Kazan et al., 2016). Production of blueberries is shown in Table 2.1.

**Table 2.1** Blueberry production in Turkey (TUIK, 2018)

<b>Year</b>	<b>Area (Decare)</b>	<b>Production (Tons)</b>
2013	485	170
2014	525	180
2015	533	180
2016	588	185
2017	582	225
2018	990	375

Blueberries can be obtained only in the growing season and they have a short storage life, therefore some preservation techniques are used to make the fruit available for much longer periods (Giovanelli et al., 2012).

Blueberries are commercially available in several forms, commonly as fresh or frozen products (Lohachoompol et al., 2004). Processed blueberries are used as ingredients in bakery, dairy, and convenience foods. Dried berries are used as ingredients in ready-to-eat cereals and many snack food products (Sinha et al., 2012). The berries can be converted to products such as dry berries, jams, jellies, juices, canned berries, and purees in order to extend their commercial life (Howard et al., 2010).

### **2.1.1 Nutritional Composition of Blueberry**

Berries generally contain plenty of sugars such as glucose and fructose, but they do not offer high calories (Table 2.2). They are low in fat, and rich in dietary fiber. They contain organic acids (citric, malic, tartaric, oxalic, fumaric acids). Certain minerals are also found in berries in trace quantities (Hidalgo and Almajano, 2017). Chemical composition of berries varies according to the cultivar, growing location, environmental conditions, maturity level, and harvest and storage conditions (Zhao, 2007).

**Table 2.2** Nutrient values of blueberries (USDA, 2018)

<b>Nutrient</b>	<b>Unit</b>	<b>Value per 100 g</b>
Water	g	84.21
Energy	kcal	57
Protein	g	0.74
Total lipid	g	0.33
Carbohydrate	g	14.49
Fiber, total dietary	g	2.4
Sugars, total	g	9.96
Calcium, Ca	mg	6
Magnesium, Mg	mg	6
Phosphorus, P	mg	12
Potassium, K	mg	77
Vitamin C, total ascorbic acid	mg	9.7
Vitamin A, IU	IU	54

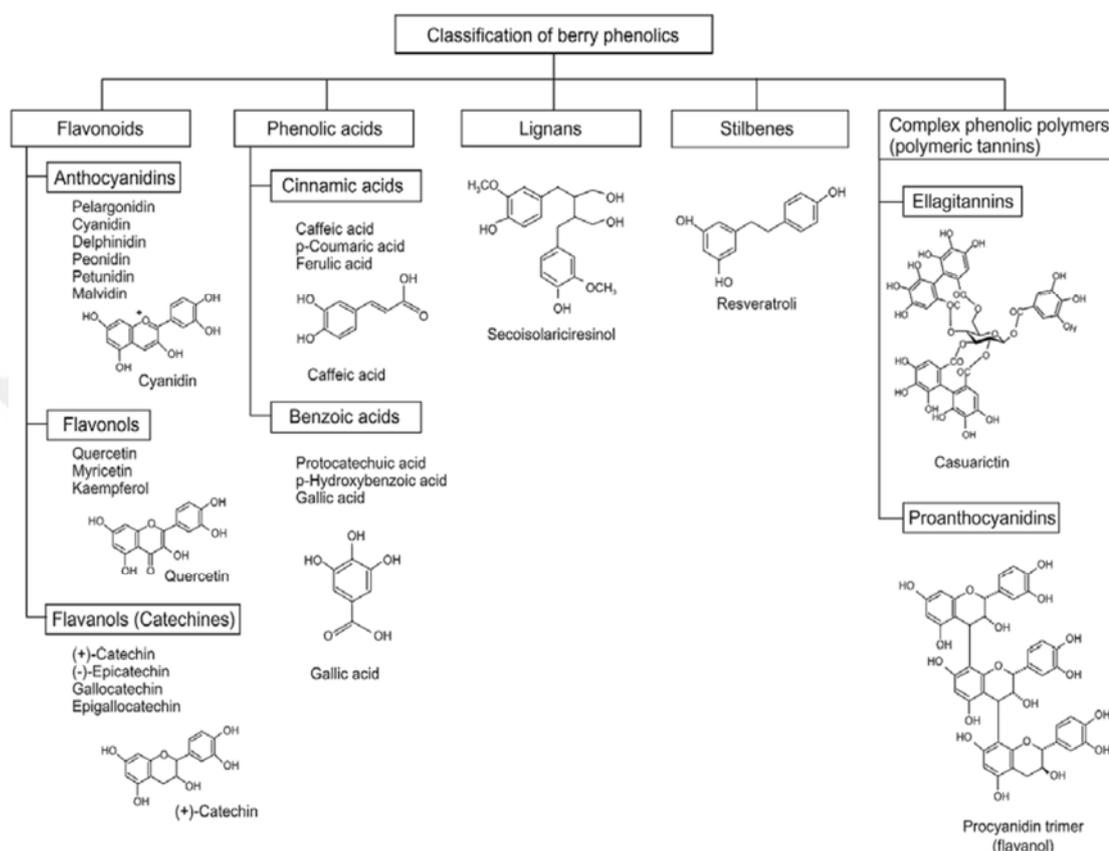
### 2.1.2 Bioactive Compounds in Blueberry

Phytochemicals are plant based bioactive compounds which may prevent chronic diseases. Berries contain high amount of phytochemicals, particularly phenolics compounds (Zhao, 2007). Numerous studies suggested that these compounds exhibit antioxidant, anti-hypertensive, anti-diabetic, anti-leukemia, anti-obesity, anti-inflammatory, and anti-microbial activity. They also present neuroactive properties potentially preventing cancer and stroke (Değirmencioğlu et al., 2017). Berry fruits are identified with high levels of antioxidants, a group of secondary metabolites. Acting as free radical terminators, phenolic antioxidants provide protection against oxidation which is stimulated by environmental factors, such as light, oxygen, and microbial activity (Hidalgo and Almajano, 2017).

Classification of berry phenolics is shown in Figure 2.3. Flavonoids and phenolics acids are two important elements of the classification of phenolic antioxidants (Hidalgo and Almajano, 2017).

Main flavonoid classes in fruits are anthocyanidins, flavonols, and flavanols (Rice-Evans et al., 1997). Anthocyanins are the form of anthocyanidins paired to sugars. They give blue, purple, and red colors to plant tissues. 3-glucosides of the anthocyanidins are the major anthocyanins found in red fruits. Most of the berries contain cyanidin-3-glucosid (Hidalgo and Almajano, 2017).

Phenolic acids are classified into two groups: hydroxybenzoic acid derivatives and hydroxycinnamic acid derivatives (Hidalgo and Almajano, 2017). Many hydroxybenzoic and hydroxycinnamic acid derivatives are found in berries (Zhao, 2007).



**Figure 2.3** Classification of berry phenolics (Puupponen-Pimia et al., 2005)

Blueberry is called the “longevity fruit” because of its health benefits and most of the health properties of the fruit are related with the phenolics composition. Therefore, phenolics and flavonoids content as well as the overall antioxidant capacity are the determiners of the fruit quality in blueberries (Goncalves et al., 2015).

Phenolics content and antioxidant activity of blueberries are determined by several factors. Composition and content of phenolics in blueberries are reported to be affected by the cultivar, the growing season and the growing location (Dragovic-Uzelac et al., 2010).

Blueberries comprise considerable amount of anthocyanins, flavonols, flavonons, proanthocyanidins, and phenolic acids (Değirmencioğlu et al., 2017).

Stevenson and Scalzo, (2012) determined the total phenolics content of blueberry between 175 and 929 mg/100 g fruit. According to the studies, total phenolic contents of highbush blueberries range between 106 -435 mg/100 g fresh weight (FW). The ranges of total phenolics for lowbush and rabbiteye types were given as 295 - 495 mg/100 g FW and 231 - 961 mg/100 g FW, respectively. Anthocyanins, procyanidins, and the hydroxycinnamate chlorogenic acid are abundant in blueberry fruit (Zhao, 2007).

Practically, one can say that the outer layer is the richest part of the blueberry, because almost all of the anthocyanins present in the outer layer. It contains most of the phenolics compounds, while the flesh and seeds contain only a small amount of these compounds (Michalska and Lysiak, 2015).

### **2.1.3 Characteristics of Blueberry Skin**

Blueberry has a blue-black skin but it looks blue-white, because the skin is covered with a cuticular wax called “bloom” (Chu et al., 2018). A hydrophobic surface of cuticle and epicuticular wax covers the single layer of blueberry epidermis. This surface has a buffer function protecting the fruit from environmental factors such as insects, infections, and desiccation. However, it also limits the transfer of water and chemical substances, which is important in dehydration of the fruit; therefore it economically affects the blueberry processing (Michalska and Lysiak, 2015).

The cuticle is composed of two parts: cutin and wax. Cutin is a biopolymer matrix of hydroxy- and hydroxyepoxy-fatty acids. Waxes present both in the cutin and on the top of it as a layer. Various plant extracts of waxes contain very long chain aliphatic compounds (e.g. fatty acids, aldehydes, ketones, primary and secondary alcohols, alkanes and alkyl esters) (Szakiel et al., 2012) and cyclic compounds (e.g. triterpenoids and steroids) (Chu et al., 2017).

The structure and characteristics of the outer layer significantly affects the water transfer from the inside out or vice versa. Since the polyphenolic compounds are generally present under the epidermis, the contents of bioactive compounds are also influenced during processing (Michalska and Lysiak, 2015).

Cuticular wax composition varies even between different organs of the same plant and it depends on the maturity level, geographic location and environmental conditions (Szakiel et al., 2012).

Chu et al., (2017) investigated the wax composition of different blueberry cultivars and found that triterpenoids and  $\beta$ -diketones dominate the cuticular wax. According to their results, the wax layer was composed of 64.2% of triterpenoids, 16.4% of  $\beta$ -diketones, 4.3% of aldehydes, 3.2% of primary alcohols, 2.8% of fatty acids, 1.3% of alkanes, and 7.8% of unidentified compounds, on average.

## **2.2 Processing of Blueberry**

### **2.2.1 Chemical Dipping Pretreatment**

Some fruits have a waxy skin reducing permeability and hindering the water loss. Therefore, some chemical and physical pretreatments are applied to the fruit before drying process in order to increase the drying rate, while preserving its quality properties (Carranza-Concha et al., 2012). Thick and waxy skin of blueberry slows down the drying rate and prolonged exposure to temperature may negatively affect the quality parameters (Giovanelli et al. 2012).

Fruits with waxy skin such as blueberries, grapes, cherries, plums, apricots and tomatoes were prescribed to be pretreated with chemical and physical methods to increase the drying rate (Vasquez-Parra et al., 2013).

Physical pretreatments generally involve blanching, skin puncturing and surface abrasion (Vasquez-Parra et al., 2013). Physical treatments such as abrasion or puncturing the skin may be effective in decreasing the drying time, but leakage and loss of internal mass is a problem. Blanching also improves the skin permeability, contributing water loss and solid gain during osmotic dehydration of the fruit. However, water soluble materials may be lost and heat sensitive nutrients may be affected by the high temperature (Ketata et al., 2013).

Alkaline or acid solutions of oleate esters are used for dipping in chemical pretreatments. Cracks formed on the surface of the fruit makes drying easier in alkaline dipping, but high temperature or long immersing time may affect texture and taste (Ketata et al., 2013).

A dipping solution of olive oil and potassium carbonate ( $K_2CO_3$ ) is the most frequently used combination for the dehydration of whole fruits. Fatty acid ethyl esters such as ethyl oleate are also commonly combined with potassium carbonate. The waxy skin texture is modified due to the synergistic reaction of these combinations and therefore, the skin impermeability for water transport is decreased (Vasquez-Parra et al., 2013).

Application of various pretreatments to the drying of black grapes was studied by Doymaz (2006). Black grapes were pretreated with a potassium carbonate solution (potas), an ethyl oleate plus potassium carbonate solution, and an ethyl oleate plus sodium carbonate solution for 1 min immersion time. All dipping treatments decreased the drying time. The author demonstrated that ethyl oleate plus potassium carbonate solution was more effective in the removal of waxy layer on the skin and improvement of drying rates (Doymaz, 2006).

Effect of ethyl oleate dipping pretreatment on blueberry drying was studied by An et al. (2019). Blueberries were treated with an alkali emulsion of ethyl oleate and the drying rate was significantly improved. The authors also showed that preservation of nutritional contents was improved by dipping (An et al., 2019). Alkaline ethyl oleate solution was also used in the chemical dipping of goldenberries. The drying time of pretreated berries was shorter than that of the untreated samples (Ozdemir et al., 2016).

Effect of dipping pretreatment using hot sodium hydroxide solution was investigated and it was observed that the drying rate and moisture diffusivity of the blueberries were increased (Shi et al., 2008 b).

Vásquez-Parra et al. (2013) investigated various chemical and physical pretreatments (blanching) on the convective drying of cape gooseberry fruits. According to their results, oil type and dipping time did not significantly affect the moisture loss, while oil concentration had a significant effect. Pretreatment with olive oil (9.48%) and  $K_2CO_3$  (4.74%) resulted in the lowest content of moisture, the highest content of vitamin C and the highest rehydration capacity. However, the biggest changes in color and chroma were also obtained for this emulsion. The authors presented the solution of olive oil and potassium carbonate as the best pretreatment option.

### 2.2.2 Osmotic Dehydration

In osmotic dehydration, a cellular material is submerged into an osmotic solution and water is transferred from the material to the osmotic solution (Ratti, 2008). Here, the sugar in the osmotic solution and the water in the material are in a countercurrent movement. The water flows out while the sugar is flowing into the fruit (Zhao, 2007). The hypertonic solution has a higher osmotic pressure and this pressure is the driving force for water diffusion (Sagar and Kumar, 2010).

There are several osmotic agents, sucrose and sodium chloride being the most popular ones. Other agents include lactose, maltodextrin, glucose, and glycerin (Ratti, 2008). Important factors influencing the performance of osmotic dehydration are the type and concentration of the osmotic agent, the ratio of the solution to the fruit, temperature, some physical properties of the fruit such as shape and size, and the existence of agitation (Zhao, 2007).

Osmotic dehydration, a pretreatment, removes moisture from the materials only to a certain extent and this removal is not enough to accept the product as dried. Therefore the process can be considered as a partial dehydration. Energy requirement of the process is low which is an advantage (Sagar and Kumar, 2010).

Treating fruits with sugar solutions or juices can be performed in combined drying methods. The purpose is to lower the moisture content and at the same time increase the solute content. Texture, colour and flavour of dried fruits can be improved by pretreating the fruits osmotically which decreases water content at low temperature and transport some functional solutes into the tissues. The moisture content can be further lowered by drying (Giovanelli et al., 2012). The materials are submerged into a hypertonic solution for a specific time in osmo-dehydration. Use of sugar for this purpose provides avoiding polyphenoloxidase activity and preserving volatile compounds (Zhang et al., 2006). Osmotic dehydration as a pretreatment may have favorable effects on dried foods. The structure may be more flexible and less dense. The amount of some fruit acids are decreased and sugar content is increased, which may positively effect the acceptability of the product (Stojanovic and Silva, 2007).

Osmotic dehydration has been extensively applied prior to air drying method, but it has also been combined with microwave drying and microwave convective drying. It

has been suggested that combining microwave or microwave convection drying with osmo-dehydration has enhanced the drying rate and the product quality was better preserved in comparison with air drying (Zhang et al., 2006). Osmotic dehydration followed by convection and microwave assisted convection drying methods were applied to blueberries pretreated with ethyl oleate and sodium hydroxide. It was suggested that, the results of osmotic dehydration and microwave assisted dehydration of pretreated berries were comparable to freeze drying (Venkatachalapathy and Raghavan, 1998).

In recent years, substances such as vitamins, minerals and prebiotics are incorporated to osmodehydration in order to enrich the products (Kowalska et al., 2017). Juice concentrates that are rich in biologically active components have been preferred as osmotic agents with the aim of adding value (Samborska et al., 2019). Although osmotic dehydration is most frequently performed using solutions of sucrose and salt, these substances are associated with adverse health effects (Lech et al., 2017).

Using fruit juice concentrates may also be economical when overproducing fruit (Samborska et al., 2019). For example, apple concentrate is considered as a comparatively cheap, natural alternative to sucrose, which has a negative perception among consumers (Nowicka et al., 2015).

Few studies have been carried out using fruit juice concentrates as osmotic agents in fruit dehydration, including blueberry (Akharume et al., 2016), yellow melon (Chambi et al., 2016), sour cherry (Konopacka et al., 2008; Nowicka et al., 2015), and kiwifruit (Escriche et al., 2000).

Use of apple juice concentrate in the osmotic dehydration of blueberries was investigated by Akharume et al. (2016). The authors indicated that fruit juice concentrates, in this case apple juice concentrate, can be an alternative to granular sugar.

### **2.2.3 Ultrasound Application**

Using ultrasound as a pretreatment for fruits before drying has become popular among researchers lately. The purpose is to increase the drying rate and improve the quality of the end product (Mothibe et al., 2011). Ultrasound can be defined as an energy produced by sound waves at a frequency range between 16 kHz and 100

MHz. This range is beyond the human ear threshold of hearing (Nowacka et al., 2018).

There are two main applications of ultrasound in the food industry. High-frequency ultrasound is in the megahertz range. It has low energy and is mostly employed in quality monitoring of foods. Low-frequency ultrasound, also called power ultrasound is in the kilohertz range. It has high energy and it is generally employed in the modification of the food structure (Mothibe et al., 2011).

The mass transfer kinetics can be improved to accelerate processes, which can be achieved by using pressure waves such as sonic or high intensity ultrasound (Mulet et al., 2003). Ultrasound has two important effects to alter the properties of the food, which are “sponge effect” and cavitation (Nowacka et al., 2018). When a material is exposed to the ultrasound, rapid compressions and expansions of the material occurs repeatedly provided by the ultrasonic waves. This condition is known as “sponge effect” as it resembles squeezing and releasing a sponge continuously (Mulet et al., 2003). The surface tension keeps the water inside the capillaries of the fruit. The mechanical forces produced by ultrasonic effect can be greater than the surface tension leading to the formation of microscopic channels. The moisture removal from the fruit is facilitated by this way (Fernandes et al., 2008 b). Other than that, ultrasound can cause cavitation in liquid systems. Bubbles are formed and collapsed in the liquid, causing a localized pressure that can favor moisture removal (Fernandes et al., 2008 a).

Application of ultrasound can be performed either using ultrasonic probes or water baths. The basic components of the power ultrasound systems are a generator, a transducer, and a coupler (Mothibe et al., 2011). An ultrasonic bath has a mettalic trunk. There are piezoelectric transducers at the bottom. The vibrations are transmitted to the whole body and then the liquid medium from the transducers. Consistent results may be difficult to get due to the irregular ultrasonic field in the bath. This is because the reflections on the air and liquid interfaces generate stationary waves. Several replicates of the application are necessary to obtain significant results (Mulet et al., 2003). Table 2.3 shows the effects of ultrasound on different drying methods.

**Table 2.3** Effects of ultrasound on different drying methods (Mothibe et al., 2011)

Drying methods	Ultrasound effects	Remarks
Hot air drying	Water effective diffusivity is increased, drying time is greatly reduced	Ultrasonic pretreatment produces microchannels inside the fruit structure and modifies the structures of materials exposed to it
Freeze drying	Freezing rate is significantly improved drying time is reduced	Ultrasound enhances the nucleation rate in freezing by producing a large number of nucleation sites in water inside the fruits
Microwave drying	Drying time is decreased, rehydration capability and texture are improved	Ruptured cell membrane structures make it easier for the water to be removed and tightly bounded water possibly becomes free because of the cavitation
Infrared drying	Hardness of samples is increased, drying rate is significantly shortened with increase of ultrasonic amplitude	Microscopic channels enable the release of moisture from the samples during drying

An important advantage of ultrasound treatments may be the retention of functional compounds because the application temperatures are low (near ambient conditions). Therefore, sensitive components are not exposed to thermal degradation (Romero and Yopez, 2015).

The ultrasound pretreatment prior to hot air drying can give well observable results due to the significant increase in effective water diffusivity. This means a considerable decrease in drying time, which in turn will decrease processing costs. Several studies have been performed applying ultrasound before hot air drying. With the application of ultrasound prior to hot air drying, the decrease in the drying time can be associated to the reduction of the initial moisture content of the material as well as the increase in the effective water diffusivity (Mothibe et al., 2011). On the other hand, the ultrasound application may not be significantly effective for the nonconventional drying techniques employing improved heating systems as in the case of microwaves where the drying is already rapid. So, it is considered that pretreating fruits with ultrasound before such drying techniques does not shorten the processing time. Still, it has been reported that ultrasound-assisted osmotic pretreatment decreased the drying time of the sea cucumber microwave freeze drying

by approximately 2 hours. This effect has been reported by some other studies for the microwave freeze drying of some fruits (Mothibe et al., 2011).

Several studies have been conducted to investigate the effect of ultrasound prior to drying on many fruits such as banana, pineapple, apple, papaya, Malay apple, cucumber and, edamame. Studies revealed that ultrasound has different effects on different fruits. For example, some fruits take up water while others release water during the application. An important parameter to take into consideration is the ratio of fruit to the liquid ultrasonic medium. This ratio is mostly given between 3:1 to 1:4 on wet basis. When distilled water is used as the liquid medium, water soluble materials are transferred to the water from the fruit and water diffuses into the fruit from the medium. The osmotic pressure phenomenon can be benefited from to improve the mass transfer, if an osmotic solution is used as the liquid medium instead of the distilled water. In this case, water migrates into the osmotic medium and solids move into the fruit (Mothibe et al., 2011).

#### **2.2.4 Ultrasound and Osmotic Dehydration**

A considerable loss of bioactive components is an important disadvantage of traditional processes and pretreatments such as osmotic pre-dehydration. The interest in non-thermal technologies has been increasing due to the demand of consumers for low-processed foods. The new techniques in food processing are important in terms of quality and shelf life of products, particularly for the forest fruits. Ultrasound is one of the most outstanding techniques in food technology (Nowacka et al., 2018). On the other hand, the traditional processes are still popular as they can be used as pretreatments in combined techniques. Ultrasound-assisted osmotic dehydration has been practiced as an alternative and innovative pretreatment technique for drying processes (Garcia-Noguera et al., 2010). Water transfer from the food material to the osmotic medium is enhanced by ultrasound at high sugar concentrations leading to significant reduction in osmotic dehydration times (Stojanovic and Silva, 2006).

During osmotic dehydration, water is being transferred from the food into the osmotic solution and the solutes in the osmotic solution move into the food material. In the ultrasound-assisted osmotic dehydration method, the ultrasonic medium is an osmotic solution. Here, ultrasound contributes both water removal and sugar gain. This condition has been presented by several studies. Solute gain was found to be

higher when the ultrasound was implemented (Mothibe et al., 2011). Ultrasound treatment during osmotic dehydration can improve the rate of mass transfer in blueberries (Stojanovic and Silva, 2006). Ultrasound-assisted osmotic dehydration has been studied for the drying of many fruits and vegetables and the effect of ultrasound was different for different fruits and vegetables (Fernandes et al., 2008 a).

When the initial moisture content of the fruit is high and the final moisture level is needed to be very low, ultrasound-assisted osmotic dehydration may not be practical. The reason may be related to the effect of ultrasound causing high sugar gain in the fruit. The sugar tends to concentrate on the surface and hinder the water removal. This sugar may also be tightly bound to the fruit water which also makes the water movement difficult. The application of ultrasound-assisted osmotic dehydration could not decrease the total processing time in the hot air drying of pineapple, where 80% of the initial water content needed to be removed (Mothibe et al., 2011).

In a study where the influence of ultrasound-assisted osmotic dehydration pretreatment on the air drying of strawberry was investigated, it was demonstrated by the authors that the combination of ultrasound energy with osmotic dehydration prior to air drying decreased the drying time and increased the effective water diffusivity (Amami et al., 2017). Optimum conditions were determined as 20.5 min pretreatment time, 47.5° osmotic solution of commercial sugar and 31°C temperature. It was also observed that the color losses were minimized when the pretreatment applied for a short time.

A similar result was obtained by Garcia-Noguera et al. (2010) for air dried strawberries. The authors indicated that the total processing time was decreased and the effective water diffusivity was improved by ultrasound-assisted osmotic dehydration, while osmotic dehydration alone increased the total processing time. They also suggested that use of higher concentrations of sucrose for longer pretreatment times might have blocked the microchannels produced by ultrasonic waves with soluble solids; and therefore, the sugar mass transfer and water diffusion during air drying were decreased.

In a modeling and optimization study of ultrasound assisted osmotic dehydration applied to cranberry (Emam-djomeh et al., 2018), it was shown that the ultrasound process decreased the osmotic dehydration time from 9 hours to 40 minutes.

Increasing ultrasound frequency increased the water loss, while decreasing the solid gain.

The ultrasound treatment during osmotic dehydration led to lower contents of bioactive compounds for cranberry samples (Nowacka et al., 2018). According to their results, the effect of ultrasonic waves on cranberries varied depending on the type of bioactive component. For example, while 60 min pretreatment time resulted in a significant decrease in vitamin C content, type of solution and pretreatment times gave statistically same contents of total polyphenols for whole berries.

Garcia-Noguera et al. (2014) studied the influence of ultrasound-assisted osmotic dehydration on the color of strawberries dried with freeze drying method. The authors concluded that this pretreatment improved the color of strawberries. More reddish and vivid color was obtained.

### **2.2.5 Drying Applications**

Dehydration is one of the oldest techniques to preserve foodstuff. The technique has been widely used in the food industry and it is a common research subject (Ratti, 2008). Drying can be defined as decreasing the moisture content of a food material to a specific level with the purpose of limiting microbial and enzymatic actions. Drying applications have an important place in food science (Mothibe et al., 2011).

In drying, water is removed from the food via evaporation or sublimation. Here, the driving force for the water movement is the heat transfer. The aim is to reduce the water activity and thus extend the shelf life of the food material (Zhao, 2007). When drying is performed under controlled conditions, it is named as dehydration (Ratti, 2008).

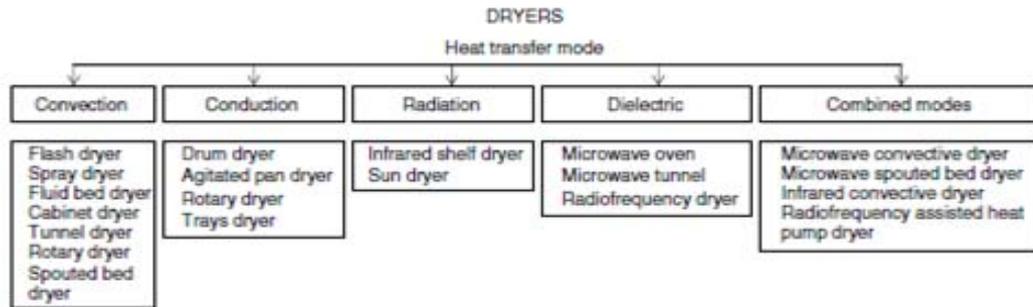
Drying is mostly practiced for perishable foods and foods containing high moisture. In addition to preservation, the drying method provides wider product range and reduced costs for packaging, handling, and storage. Besides, some products may be available for consumers after their growing season (Moses et al., 2014). Dried fruits are consumed as snacks or they are added into several other food products such as cakes and cereals (Mothibe et al., 2011).

By reducing the water activity with drying, microbial growth and deteriorative reactions can be avoided. According to the purpose of the process, the detrimental effects of the drying may be advantageous or disadvantageous (Rahman, 2007).

Drying rate curves can be examined in several parts. These parts arise from the internal drying mechanism and the changes going on during drying (Sinha et al., 2012). Generally, drying curves are formed by plotting the drying rate versus drying time or moisture content. The main parts of the drying curve are: (1) transient period, which is an initial stage where the product gets heated, (2) constant rate period, where the moisture removal is easy, and (3) falling rate period, during which the moisture removal is difficult and the rate is decreasing (Rahman, 2007). The average moisture content measured when the surface gets dry is known as the critical moisture content. The drying rate decreases after this critical point (Sinha et al., 2012).

Both heat transfer and mass transfer take place in a drying process. So, we can say there are two resistances (Rahman, 2007). The moisture continuously moves to the surface of the material being dried during the constant rate period and the surface remains wet (Sinha et al., 2012). Therefore, it is considered that there is no mass transfer resistance in the constant rate period and external heat transfer governs the drying. During the falling rate period, the mass transfer resistance takes over. If the constant rate period is not observed, that means internal mass transfer controls the process all along (Rahman, 2007).

Dehydration of berry fruits should be performed carefully in order to minimize the loss of important phytonutrients (Zhao, 2007). Various drying methods have been used to produce dried blueberries with good quality. Sun drying, convection oven drying, freeze drying, and microwave drying are some of the examples (Değirmencioglu et al., 2017). A typical classification of dryers is shown in Figure 2.4.



**Figure 2.4** Classification of dryers (Ratti, 2008)

### 2.2.5.1 Conventional Hot Air Drying

Conventional hot air drying can be defined as a process where the food materials are exposed to a steady flow of hot air (Wang et al., 2019). It is a frequently used method for drying of foods such as fruits, vegetables, pasta, coffee, etc. Dehydration is performed by conducting hot air to food materials in a mechanical device. Hot air can be supplied in different directions and the food materials can be static or moving. The process is easily controlled and good quality products are produced. Therefore, this system is commonly used for berry drying (Zhao, 2007).

During drying, the surface moisture is evaporated from the surface of the materials by the external heat. At the same time, the internal moisture moves to the surface to be evaporated. Also, the internal moisture may be evaporated inside the material and then it may be forced to the surface (De Bruijn and Borquez, 2014). Two heat transfer mechanisms take place in hot air drying of foods: convection and conduction. Heat is transferred to the food material from the flowing hot air via convection and conduction occurs by the movement of heat inside the product. The heat transfer coefficient in convection is mostly dependent to the air speed and air flow. On the other hand, the heat transfer by conduction depends on the characteristics of the food material, moisture content, and the temperature gradient inside the material (Zhao, 2007).

The drying rate is determined by several factors. These factors are temperature, humidity, air velocity, direction of air supply (horizontal or vertical), air exchange and fruit properties including characteristics (i.e. structure and composition) and thickness and geometry of the fruit. Generally air temperature and velocity are

directly proportional to the drying rate. Also, when the air humidity is low, the drying rate is higher. An air dryer must exude moist air and new air with lower humidity must enter the dryer, but this condition may lead to heat loss from the system. Sometimes, two- or multistage drying techniques with different drying conditions may be practised (Rahman, 2007).

A simple drier is composed of a feeder, a heater, and a collector. The design of these units is particular to each drying type (Vega-Mercado et al., 2001). Tray, cabinet, tunnel, belt conveyor, and fluidized bed dryers can be used for berry drying (Zhao, 2007). Cabinet and bed-type dryers are considered as the simplest drying methods. Drying occurs in an enclosed chamber, where the food materials are placed on trays and exposed to hot air passing over or through the product (Rahman, 2007).

The quality of the dried product is affected by dehydration conditions. These conditions are determined by the characteristics of the food material (Zhao, 2007). To provide an extended shelf life, the water content of fruits dried by convection should be between 20% and 25% (i.e. 0.33 kg water/kg dry matter) (De Bruijn and Borquez, 2014). Usually the hot air is applied in a temperature range of 50 °C to 110°C, and the air velocity is between 0.1 and 5 m/s (Nemzer et al., 2018). Berries greatly vary in skin thickness, presence of seeds, and contents of water, sugar, and acids. Therefore, the drying behaviour and consequently the product quality depend on the process variables (Zhao, 2007).

Convective hot air drying has important advantages ensuring a wide use in food industry. It is a comparatively low-cost method (Kowalski and Szadzinska, 2014). The application does not need expensive equipment. The operation is simple and ordinary workers can easily perform the procedure. However, it has the disadvantage of being energy intensive; it has low energy efficiency (Wang et al., 2019). Shrinkage is another disadvantage of this method. The moisture removal causes a decrease in volume which leads to tissue collapse (Wojdylo et al., 2014). Additionally, this method can harm heat sensitive materials, so it is important to choose the drying conditions and parameters appropriately (Kowalski and Szadzinska, 2014).

The waxy skin of blueberries causes a low drying rate in hot air drying. This rate decreases with decreasing moisture in the product resulting in a long drying time. The physical properties of the blueberries such as color, volume, porosity, and structure undergo some changes during drying. Also, biological activity of some components may be negatively affected (Zielinska and Michalska, 2016).

There are several studies conducted to investigate the effects of air drying temperatures on some quality characteristics of many fruits (Jessica e al., 2013). A lot of research has been made to study the convective air drying technique on blueberries (Degirmencioglu et al., 2017; Giovanelli et al., 2013; Lopez et al., 2010; Reque et al., 2016; Vega-Galvez et al., 2009)

In a study conducted on fresh, whole blueberries, the heat processing of the fruits at 70, 80 and 90 °C temperatures was evaluated. The samples were dried by convection in a bench-scale tray dryer. Obviously, drying rates increased, and so the drying times decreased with increasing temperature. The authors reported that longer exposure to heat was detrimental to the bioactive materials and antioxidant properties of blueberries (Reque et al., 2016).

In an another work, fresh blueberries were dried in a laboratory-scale convective dryer to study the effect of air temperature on drying kinetics and some quality properties of blueberry such as vitamin C content, antioxidant capacity, phenolics content, color and firmness. Important losses of vitamin C and discoloration due to non-enzymatic browning were observed at all temperatures studied. It was concluded by the authors that product quality was better at high temperatures (80 and 90 °C) compared to lower temperatures (50, 60 and 70 °C) in terms of nutrient property and antioxidant activity, which was associated with long processing times. On the other hand, more browning and less firmness was obtained at high temperatures (Lopez et al., 2010).

#### **2.2.5.2 Microwave Drying**

Microwaves are electromagnetic waves between a frequency of 300 MHz to 300 GHz. These wavelengths account for a range of 1 to 0.001 m. In household type applications and in the industry, the overall frequency range in use is between 915 and 2450 MHz (Mohapatra and Mishra, 2011). The microwaves are created by a

magnetron type vacuum tube (Borquez et al., 2010). The basic components of a microwave heating system are a generator or magnetron, a wave guide and an applicator (Mohapatra and Mishra, 2011). Materials containing water can absorb electromagnetic energy in the above-mentioned frequency range (Maskan, 2000). Bound water molecules are polarized by the electromagnetic field and gets ionized (Mohapatra and Mishra, 2011). This heating system makes use of the polarization mechanism occurring at molecular and atomic levels. Polarized molecules and ions in the product try to align with the alternating electromagnetic field. They rotate millions of times per second resulting in a generation of heat (Vega-Mercado et al., 2001). Microwave heating is defined “volumetric” since the waves can permeate into material and heats it up thoroughly. The entire volume is heated rapidly and uniformly (Maskan, 2000).

Microwave technology finds itself a place in various practices such as baking, cooking, blanching, concentrating, etc. (Ratti, 2008). Recently microwave drying has been a popular method for various food products due to its important benefits compared to conventional heating (Wojdylo et al., 2014). Since water molecules rapidly absorb microwave energy, the evaporation is fast, providing a higher drying rate. Rapid water evaporation causes an outward flux, which not only improves the drying rate, but also helps preventing the shrinkage (Maskan, 2000). Efficient energy use and better control of the process are some other important advantages of microwave energy. Additionally, the operation can be readily started and shut down whenever necessary (Botha et al., 2012). Microwave technology also saves up floor space thanks to its concentrated energy.  $20 \pm 35\%$  of the space needed for conventional heating equipment is enough for microwave heating (Maskan, 2000).

On the other hand, there are important limitations concerning overheating of some regions and loss of bioactive materials (Zielinska and Michalska, 2016). Hot spots may occur due to the non uniform electromagnetic field and nonhomogenous structure of the product (Wojdylo et al., 2014). Therefore, drying material should be continuously moving or rotating in the cavity to prevent the formation of hot spots. During the last stages of drying, the amount of water is low, so the temperature may considerably increase enough to cause scorching. It is comparatively easier to control the product temperature in hot air drying because the product temperature never exceeds air temperature (Zhang et al., 2006).

Although the drying time is reduced, microwave drying may cause low product quality. Higher power usually provides a faster drying, however higher quality requires lower power (George et al., 2004). The product quality may suffer from the rapid water transfer and some unwanted textural changes may occur by “puffing” (Zhang et al., 2006).

High investment costs and limited penetration of microwaves are some other drawbacks of this system. To overcome some of these difficulties, microwave drying can be combined with other drying methods such as convective air drying (Izli et al., 2014).

### **2.2.5.3 Hybrid Drying**

The combination of microwave drying and hot air drying is one of the most frequently used combined technique. In this technique, while microwaves remove water from the inside of the product, hot air facilitates the removal of water on the surface. Therefore microwave-hot air combined (hybrid) drying increases the rate of drying and better preserve the quality (Horuz et al., 2017).

As microwave energy can be combined with convection heating through the whole process, it can also be combined at different stages of drying. When microwave energy is applied at the beginning of the process, the temperature inside the product rapidly rises to evaporate water and the vapor moves from inside out. This provides easy removal of water from the surface by hot air. Application of microwave energy at a point where the rate of drying begins decreasing, allows the internal heat force the moisture at the center of the product to the surface by means of vapor pressure. Microwaves can also be applied in the falling rate period to finish the drying at a faster rate. This way, removal of bound water from the product may be facilitated and the shrinkage of tissue structure may be avoided (Zhang et al., 2006).

In a study in which apple and strawberry samples were dried using air drying and microwave assisted air drying, the combination of microwave energy with convection drying was recommended (Conteras et al., 2008). It was asserted that the drying time was decreased and less color change occurred when higher air temperature and microwave was applied. The drying time was reduced by a

significantly higher extent with microwave application compared to the increase in air temperature.

Horuz et al. (2017) studied hybrid and convective drying of sour cherries. They found that the drying rate was higher in hybrid drying technique compared to air drying. Besides, hybrid drying resulted in higher total phenolics content, antioxidant activity, vitamin C content and rehydration ability.

Beaudry et al. (2004) tried four different drying methods to dry osmotically dehydrated cranberries, and showed that drying time was the shortest in microwave-assisted air drying, compared to freeze drying, vacuum drying and hot air drying methods. Hot air drying resulted in the longest drying time. Additionally, no significant differences were obtained for color and water activity results.

Sunjka et al. (2004) also investigated the microwave-convective drying of osmodehydrated cranberries. According to the study, microwave-vacuum drying was more efficient in terms of energy than microwave-assisted air drying. Color values were similar for both methods.

Effect of microwave-assisted convective drying on the functional quality of raisins was studied by Carranza-Concha et al. (2012). The study showed that microwave assisted convective drying caused greater reduction of ascorbic acid, however it also resulted in higher antioxidant activity. On the other hand, the phenol content increased in dehydrated grapes.

The effects of microwave assisted drying was also investigated for goldenberry (Izli et al., 2014). In the study, goldenberry samples were subjected to convective, microwave, and microwave– convective drying procedures. According to the results, the combination of microwave energy with conventional air drying significantly decreased the drying time. The authors concluded that the hybrid drying method was a proper method for goldenberry in terms of both shortening the drying time and preserving the phenolics and antioxidant capacity.

In an another study, in which the effects of different drying techniques on drying kinetics, colour, total polyphenols, anthocyanins, antioxidant capacity and texture of freeze/thawed blueberries were investigated, the results of convective air drying, microwave vacuum drying and the combination of these two were compared

(Zielinska and Michalska, 2016). Hot air drying at 90 °C combined with microwave vacuum drying was considered suitable for blueberry drying with respect to short drying time and enhanced product quality. It was also noted that the highest total polyphenols was obtained in hot air drying at 90 °C, while hot air drying at 90 °C combined with microwave vacuum drying resulting in the highest content of anthocyanins and the strongest capacity of antioxidant.



## CHAPTER 3

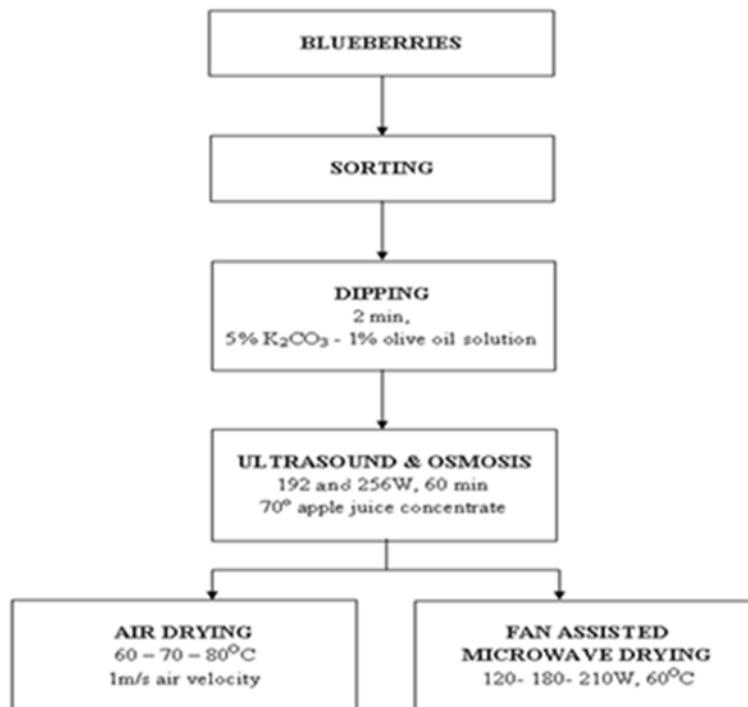
### MATERIALS AND METHODS

#### 3.1 Materials

Organic blueberries (*Vaccinium Corymbosum* L., Bluecrop variety) were obtained from an organic farm in Trabzon, Turkey. Blueberries were stored in a refrigerator at 4 °C until processing. Organic olive oil used for dipping solution was obtained from a local supplier. Organic apple juice concentrate (70 °Brix) was purchased from Targid Tarım ve Gıda Ürünleri Sanayi ve Ticaret A.Ş., Mersin, Turkey. Potassium carbonate ( $K_2CO_3$ ), ethanol, Folin-Ciocalteu reagent, sodium carbonate ( $Na_2CO_3$ ), gallic acid, 1,1-diphenyl-2-picrylhydrazyl (DPPH), and ( $\pm$ )-6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox) were purchased from Sigma-Aldrich.

#### 3.2 Sample Preparation Steps

Dry blueberries were produced according to the flow diagram given in Figure 3.1.



**Figure 3.1** Experimental flow chart

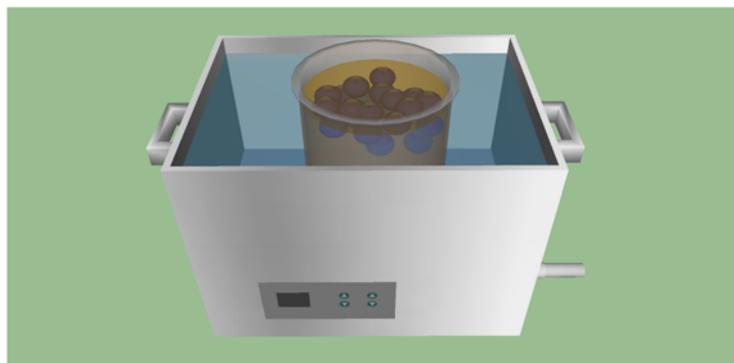
### 3.2.1 Dipping Pretreatment

Ingredients of the dipping solution and their concentrations were determined according to the literature (Doymaz and Pala, 2002; Doymaz, 2006; Vasquez-Parra, 2013) and preliminary works. 1 litre of solution containing 5%  $K_2CO_3$  and 1% olive oil (v/v) was prepared by continuous stirring to form an emulsion. This solution was kept in a refrigerator and it was allowed to reach room temperature before each dipping treatment.

Berries were examined physically and ripe, intact fruits with average size were selected. Sorted and weighed blueberries were immersed into the dipping solution for 2 min at room temperature. After dipping, excess water was removed from the berries with a paper towel. All materials and treatments were chosen to preserve the organic condition of the fruit.

### 3.2.2 Ultrasound and Osmosis Pretreatment

Ultrasound and osmotic dehydration techniques were combined as seen in the Figure 3.2. Berries were weighed and placed in a glass beaker containing organic apple juice concentrate. The ratio of osmotic solution to fruit was maintained at 4:1 (w:w). The beaker was then placed in the ultrasonic bath (Bandelin Sonorex Digiplus, Berlin, Germany) filled with distilled water. The ultrasound instrument specifications were: 160/640 W power and 35 kHz frequency. Berries were exposed to ultrasounds for 60 min at two different power levels (30% and 40% corresponding to 192 W and 256 W, respectively). Temperature of the bath was controlled periodically with a thermometer during the treatment and it was not allowed to exceed 25 °C by using ice blocks when necessary. After the treatment, excess concentrate was removed with a paper towel from the surface of berries.



**Figure 3.2** Ultrasound and osmotic dehydration system

### 3.2.3 Convectonal Air Drying

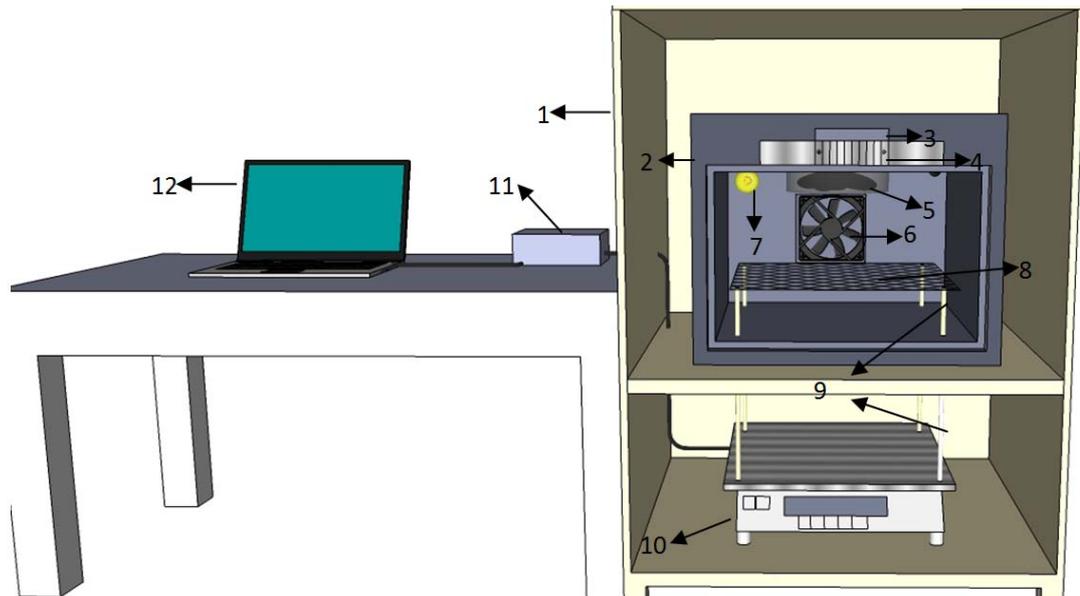
Convectonal drying was performed by using a simple home type dehydrator (Excalibur 4526T, Sacramento, U.S.A) as shown in Figure 3.3. Berries were spread uniformly on a tray and placed into the oven. The air was blown over the berries by the fan mounted on the back of the oven. Air velocity was 1 m/s constant. Drying was carried out at three different temperatures (60, 70 and 80 °C). The tray was removed from the oven periodically to record the moisture loss and put back immediately after weighing. Berries were dried to 20 % final moisture content (wet basis). When the samples reach the desired final weight, they were taken out and allowed to cool at room temperature. Dehydrated samples were packed and sealed in polyethylene bags, and stored at 4 °C in a refrigerator for further analyses.



**Figure 3.3** Picture of a home type convectional dehydrator

### 3.2.4 Hybrid Drying

A programmable air-circulating hybrid domestic microwave oven (Figure 3.4) was used for the dehydration of blueberries (Arçelik KMF 833 1, Turkey). It can simultaneously supply microwaves and air circulation to the samples. The maximum output of the oven is 900 W at 2465 MHz frequency. Microwave emitter is at the top of the oven. Approximately 230 g of whole berries were placed on a tray every time. The berries were spread uniformly on the tray and the tray was put on a perforated polyamide platform inside the oven. The platform was connected to a 0.01 g precision analytical balance under the drying cabinet to record the moisture loss periodically. Berries were dried at three different microwave power levels: 150, 180 and 210 W. Air flow was kept constant at 60 °C. The tray was rotated 90 degrees every 30 min to provide more homogenous heating.



**Figure 3.4** Schematic representation of hybrid oven: (1) Drying cabinet; (2) Hybrid oven; (3) Display; (4) Control buttons; (5) Microwave emitter; (6) Fan; (7) Light; (8) Polyamide tray; (9) Polyamide legs; (10) Analytical balance; (11) Data logger; (12) PC (Horuz et al. 2017).

Drying blueberries at 210 W resulted in excessive dripping of blueberry juice to the bottom of the oven during the preliminary works; therefore, the berries were first dried in the dehydrator at 80 °C for 90 min and then transferred into the microwave oven. This was done for all samples to be dried at 210 W.

Berries were dried to a final moisture content of 20 % (wet basis). After drying, they were allowed to cool at room temperature, packed and sealed in polyethylene bags, and stored at 4 °C in a refrigerator for further analyses.

### 3.3 Modeling of Thin Layer Drying

Five different mathematical models were used to determine the best model that describes the conventional air and hybrid drying of pretreated blueberry. Four of the selected models are commonly used in the modeling of fruit drying (Page, Logarithmic, Midilli et al., and Handerson and Pabis models). Also, a modified Logistic model was tested which has recently been suggested for sour cherry drying (Horuz et al. 2017). The model names and equations are given in Table 3.1. The moisture ratio (*MR*) was calculated using the formula:

$$MR = \frac{M_t - M_e}{M_o - M_e}$$

where,  $M_t$  is the moisture content at any time,  $M_o$  and  $M_e$  are the initial and final moisture contents, respectively.

SigmaPlot software (Systat Software, Inc., San Jose, California, USA) was employed for model fitting. Three parameters were used to evaluate the quality of fit: the correlation coefficient ( $R^2$ ), the reduced chi-square ( $\chi^2$ ), and the root mean square error ( $RMSE$ ).

$$\chi^2 = \frac{\sum_{i=1}^N (MR_{exp,i} - MR_{pre,i})^2}{N - z}$$

$$RMSE = \sqrt{\frac{\sum_{i=1}^N (MR_{exp,i} - MR_{pre,i})^2}{N}}$$

where,  $MR_{exp}$  is the experimental moisture ratio,  $MR_{pre}$  is the predicted moisture ratio,  $N$  is the number of experimental data, and  $z$  is the number of model parameters.

**Table 3.1** Mathematical models used for the drying curves of blueberry

Model name	Model	References
Page	$MR = \exp(-kt^n)$	Igual <i>et al.</i> (2012)
Logarithmic (Logar.)	$MR = a \exp(-kt) + c$	Karaarslan&Tuncer (2008)
Midilli <i>et al.</i> (Midilli)	$MR = a \exp(-kt^n) + bt$	Midilli <i>et al.</i> (2002)
Handerson and Pabis (H&P)	$MR = a \exp(-kt)$	Zielinska&Michalska (2016)
Modified Logistic (mod. Log)	$MR = a/[1 + \exp(-4k(l-t)/a+2)]$	Horuz <i>et al.</i> (2017)

$k$  is the drying rate constant and  $a$ ,  $b$ ,  $c$ ,  $l$ , and  $n$  are equation constants.

### 3.4 Chemical and Physical Analyses

#### 3.4.1 Moisture Content Determination

Moisture contents of the samples were determined by the oven method according to the standard procedure of AOAC (1995). 3-5 g of fruits were weighed and dried in an oven at 105 °C until constant weight. Moisture content was calculated from the weight difference. All measurements were performed in triplicate.

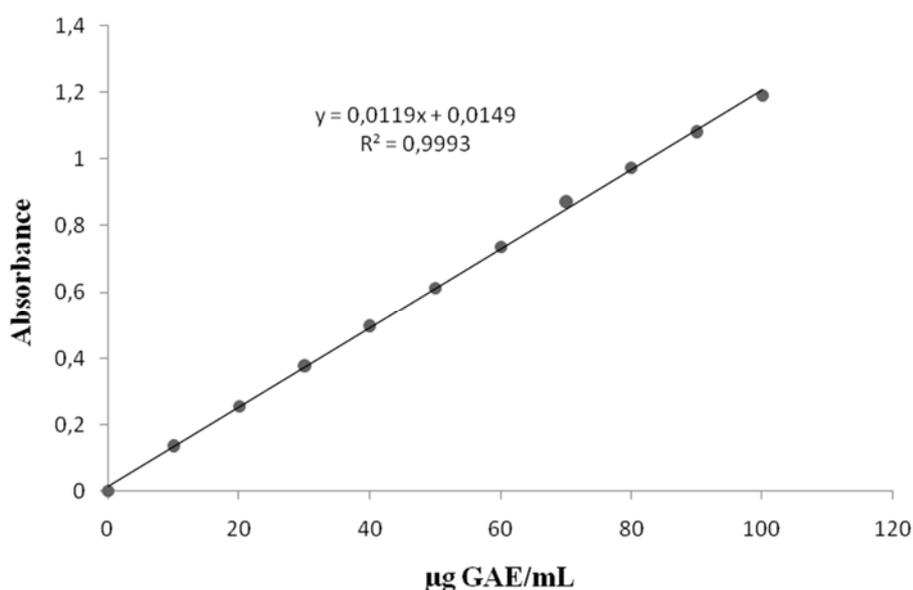
#### 3.4.2 Preperation of Phenolic Extracts

Berry extracts were obtained following the procedures described by (Fu *et al.*, 2011) with some modifications. 2 grams of crushed berries were weighed and mixed with

40 ml of ethanol:water solution (65:35, v:v). The mixtures were covered with stretch film and aluminum foil and placed into a shaker (Heidolph Unimax 1010, Schwabach, Germany) operating at 50 °C. After shaking for 45 min, the extracts were filtered using Whatman No. 41 filter paper. The volumes of extracts were recorded. The extract solutions were placed into amber glass vials and kept at -20 °C until analysis.

### 3.4.3 Total Phenolics Content Analysis

Total phenolics content (TPC) of the samples was determined according to the procedure described by Singleton et al. (1999). 450 µL of 30-fold diluted extract was mixed with 2.25 mL of Folin-Ciocalteu reagent (1:9, v:v) diluted with distilled water. After 3 min, 1.8 mL of Na<sub>2</sub>CO<sub>3</sub> (75 g/L) solution was added and the tubes were left in dark for 2 hours at room temperature. Absorbances were measured at 760 nm using Lambda 25 UV/Vis spectrophotometer (PerkinElmer, Shelton, CT, USA). A linear standard curve (Figure 3.5) was obtained between the gallic acid concentrations of 10-100 µg/mL. All measurements were carried out in triplicate and the results were expressed as mg gallic acid equivalent per 100 g dry matter (mg GAE/100g dm).

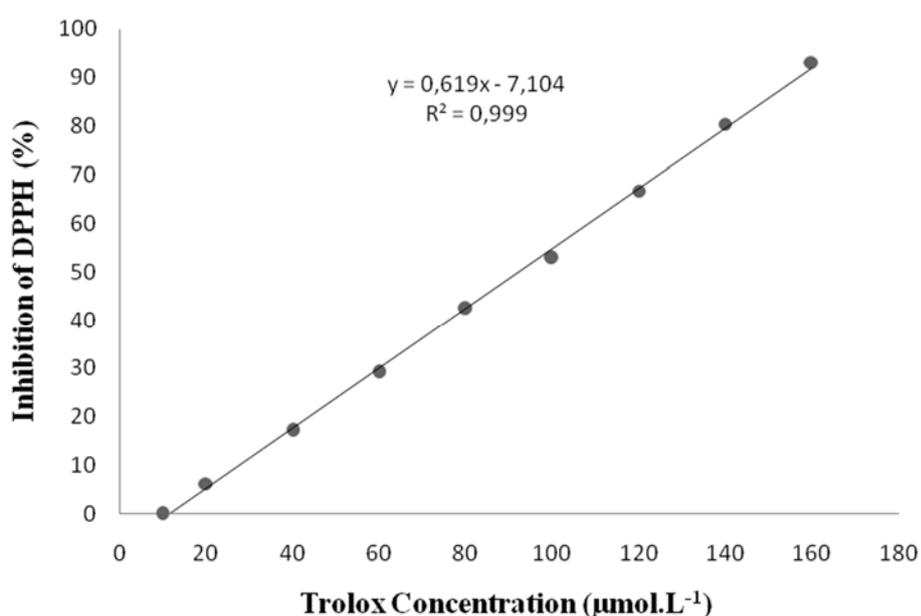


**Figure 3.5** Gallic acid calibration curve

### 3.4.4 Antioxidant Activity Analysis

Antioxidant activities of the samples were measured following the procedure described by Brand-Williams et al. (1995), with slight modifications. 89.7  $\mu\text{M/L}$  of DPPH solution was prepared and its final absorption was adjusted to  $0.800 \pm 0.010$  AU at 517 nm. 500  $\mu\text{L}$  of 65-fold diluted extract or blank (ethanol) was mixed with 2500  $\mu\text{L}$  of the DPPH solution. The samples were left in the dark for 60 min. Absorbance (A) was measured at 517 nm (Optima SP-3000 nano UV-Vis Spectrophotometer, OPTIMA Tokio, Japan). A trolox standard curve (Figure 3.6) was plotted using trolox concentrations 10-160  $\mu\text{M}$ . Results were expressed as micromolar trolox equivalent per g of dry matter ( $\mu\text{mol TE/g dm}$ ). The calculation of percent inhibition for DPPH radical-cavenging activity was made according to the formula:

$$\text{Inhibition of DPPH \%} = [(A_{\text{control}} - A_{\text{sample}}) / (A_{\text{control}})] \times 100$$



**Figure 3.6** Trolox calibration curve

### 3.4.5 Color Measurement

Color parameters were measured using HunterLab ColorFlex A60-1010-615 Model Colorimeter (Hunter Lab, Reston, VA). The sample cup was filled with dried berries and the surface was covered with a dark background to avoid diffraction errors.  $L^*$  (whiteness or brightness / blackness),  $a^*$  (redness / greenness) and  $b^*$  (yellowness /

blueness) values were measured. Three readings were taken by rotating the sample cup after each recording.

### **3.5 Statistical Analysis**

SPSS (IBM SPSS Statistics for Windows, Version 21.0. Armonk, NY, USA) was employed for ANOVA, *t*-test, and Pearson correlation tests. LSD test was used to compare means of the groups. For model fitting and plotting graphs, SigmaPlot version 12.0 (Systat Software, Inc., San Jose, California, USA) was used.



## CHAPTER 4

### RESULTS AND DISCUSSION

#### 4.1 Drying Characteristics of Hybrid and Conventional Air Drying

In this chapter, total phenolics content, antioxidant activity and color parameters of blueberries dried by convectional and hybrid drying techniques will be compared. The fresh blueberries were dried to a final moisture content of 20 % (wb) by both drying processes. The effects of dipping, ultrasound and osmotic pretreatment on the physical-chemical properties and drying kinetics of blueberries will be discussed. On the other hand, the best fitting model for all drying conditions was determined as Midilli et al. Model as shown in section 4.2. Therefore, this model was fitted to the drying data and shown on all the figures of moisture ratio vs time.

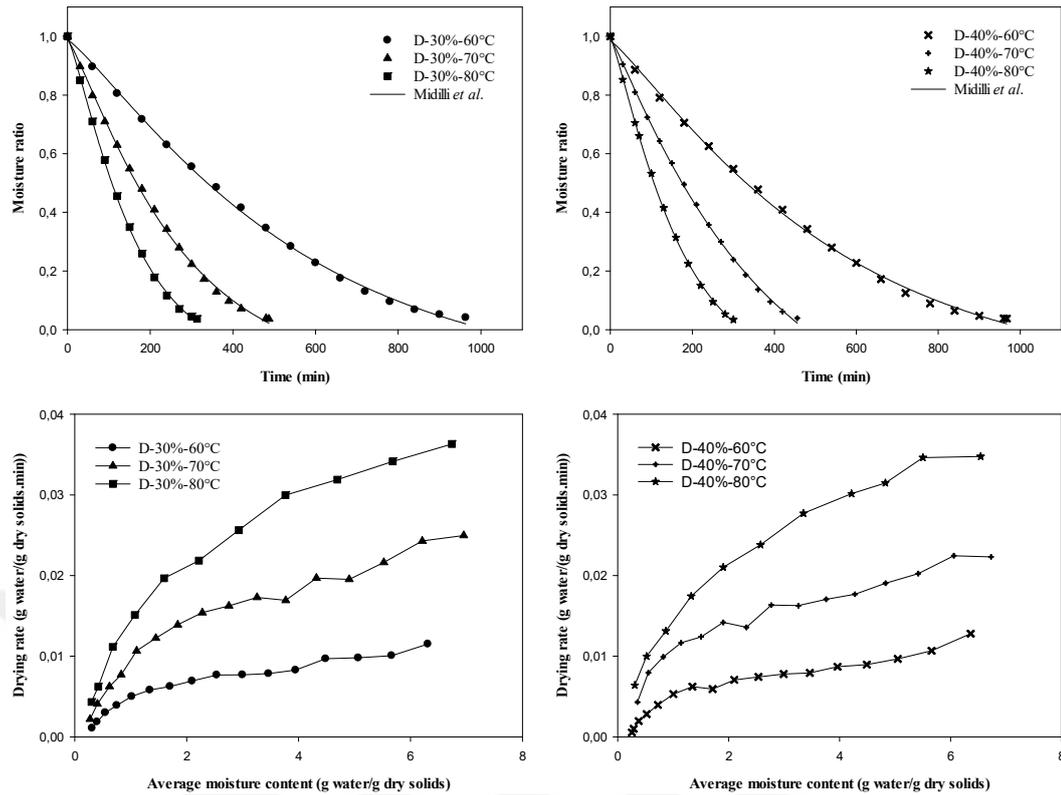
Drying conditions and sample codes used in this text are shown in Table 4.1. Experimental drying times of different drying processes described in Table 4.1 are listed in Table 4.2. The moisture ratio and drying rate curves for air drying of blueberries are given in Figure 4.1. During conventional air drying, hot air at constant velocity was applied to the berries at three different temperatures (60, 70, and 80°C). As expected, the increase in temperature shortened the drying time, and the drying rate was significantly ( $p < 0.05$ ) increased. The effect of temperature on drying time was studied by Vega-Gálvez et al. (2009), and they observed a similar trend for hot air drying of blueberry.

**Table 4.1** Sample codes for the experimental drying conditions

<b>Drying condition</b>	<b>Description</b>
D-30%-150W	Dipping, Ultrasound and osmosis at 30% power level, Hybrid drying at 150W
D-30%-180W	Dipping, Ultrasound and osmosis at 30% power level, Hybrid drying at 180W
D-30%-210W	Dipping, Ultrasound and osmosis at 30% power level, Hybrid drying at 210W
D-40%-150W	Dipping, Ultrasound and osmosis at 40% power level, Hybrid drying at 150W
D-40%-180W	Dipping, Ultrasound and osmosis at 40% power level, Hybrid drying at 180W
D-40%-210W	Dipping, Ultrasound and osmosis at 40% power level, Hybrid drying at 210W
210W	Hybrid drying at 210W
D-210W	Dipping, Hybrid drying at 210W
D-40%(Water)-210W	Dipping, Ultrasound in water at 40% power level, Hybrid drying at 210W
D-30%-60°C	Dipping, Ultrasound and osmosis at 30% power level, air drying at 60°C
D-30%-70°C	Dipping, Ultrasound and osmosis at 30% power level, air drying at 70°C
D-30%-80°C	Dipping, Ultrasound and osmosis at 30% power level, air drying at 80°C
D-40%-60°C	Dipping, Ultrasound and osmosis at 40% power level, air drying at 60°C
D-40%-70°C	Dipping, Ultrasound and osmosis at 40% power level, air drying at 70°C
D-40%-80°C	Dipping, Ultrasound and osmosis at 40% power level, air drying at 80°C
80°C	Air drying at 80°C
D-80°C	Dipping, air drying at 80°C
D-40%(Water)- 80°C	Dipping, Ultrasound in water at 40% power level, air drying at 80°C

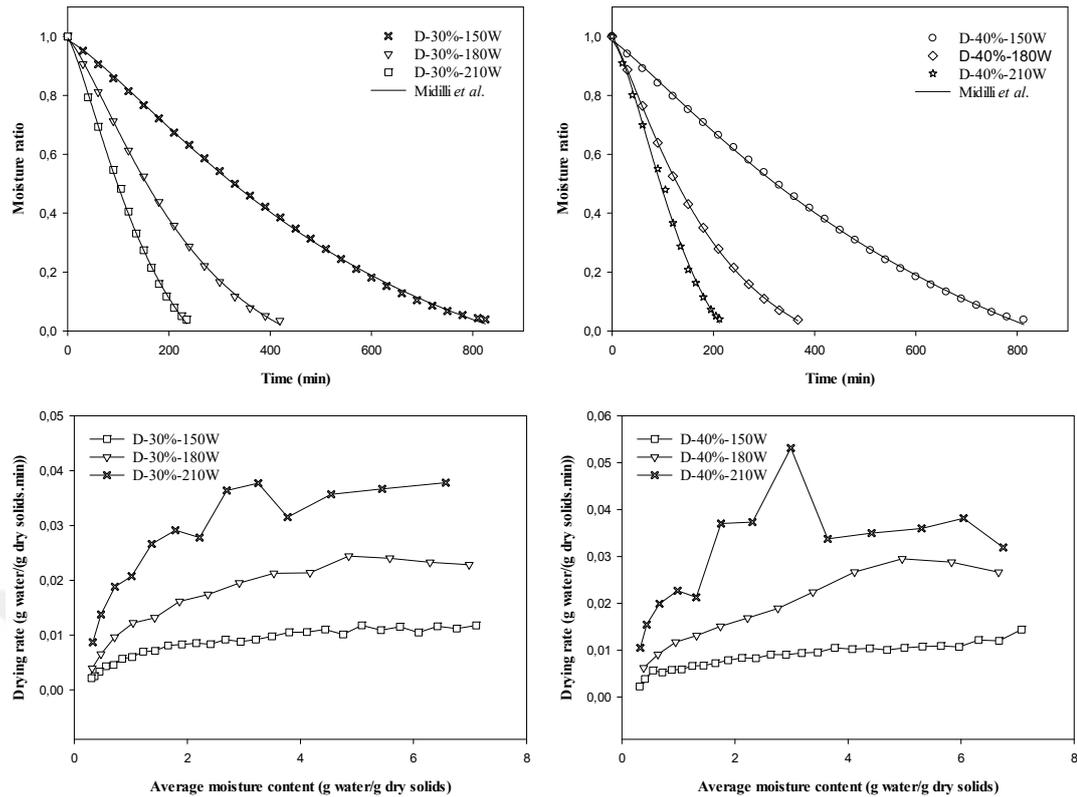
**Table 4.2** Drying times for hybrid and convectional air drying methods at varying conditions.

<b>Drying condition</b>	<b>Drying time (min)</b>	<b>Drying condition</b>	<b>Drying time (min)</b>
D-30%-150W	825	D-30%-60°C	963
D-30%-180W	419	D-30%-70°C	487
D-30%-210W	235	D-30%-80°C	313
D-40%-150W	813	D-40%-60°C	967
D-40%-180W	367	D-40%-70°C	456
D-40%-210W	212	D-40%-80°C	300
210W	242	80°C	315
D-210W	223	D-80°C	270
D-40%(Water)-210W	248	D-40%(Water)-80°C	314



**Figure 4.1** Moisture ratio and drying rate curves for air drying of blueberries

The moisture ratio and drying rate curves for hybrid drying of blueberries are given in Figure 4.2. During hybrid drying, three different power levels (150, 180, and 210W) were applied to the fruit. As the power levels increased, the drying times were decreased, and the drying rates were increased significantly ( $p < 0.05$ ). The fact that increasing microwave power level increases drying rates was observed by several authors (Andres et al., 2004; Maskan, 2000; Piotrowski et al., 2004).



**Figure 4.2** Moisture ratio and drying rate curves for hybrid drying of blueberries

When the drying times of the two drying methods are compared, it can be said that the hybrid drying resulted in shorter drying times than hot air drying. To illustrate, the drying time for D-30%-150W was 825 min, where hot air was applied simultaneously at 60 °C. The drying time for berries dried at 60 °C in dehydrator (D-30%-60°C) was 963 min. For all drying conditions (Table 4.2), the drying times of hybrid drying were shorter than the hot air drying equivalents. Therefore, application of microwaves during hot air drying reduces the drying time. This effect was also observed by Contreras et al. (2008), Horuz et al. (2017), and Izli et al. (2014).

Preliminary studies at 210 W microwave power level showed that fruit juice leaked from cracks formed in the structure of blueberry fruit during drying. Therefore, drying processes at 210W were performed by drying the berries at 80 °C in the dehydrator for the first 90 min and then transferring them to the hybrid oven to finish the drying. Faster drying effect of microwave energy can be seen in the drying rate curves. The drying rates increased at a certain point. This point is the point where the berries are removed from the dehydrator and placed into the microwave oven.

#### **4.1.1 Effect of Dipping Pretreatment on Drying Characteristics**

A chemical dipping treatment was applied to the blueberries in order to remove the waxy bloom layer on the surface and accelerate the drying process. For both hybrid drying and hot air drying methods, drying times of untreated berries were longer than pretreated blueberries. The successful removal of this layer was visually observable.

However, the treatment did not cause a significant difference in terms of drying rate ( $p>0.05$ ). This may be due to the presence of an originally thin and poor wax layer on the surface of the fruit. Also, drying processes carried out at 210 W and 80 °C may have been too short to observe a significant effect on drying rates.

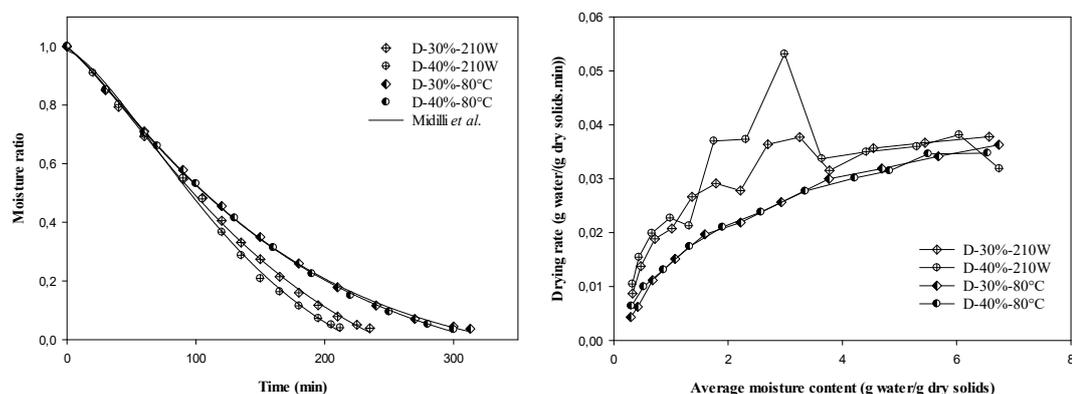
#### **4.1.2 Effect of Ultrasound and Osmotic Dehydration on Drying Characteristics**

An important part of the study was the ultrasound-assisted osmotic dehydration of blueberries prior to drying. It was expected to enhance the drying rates, reduce the overall processing times and contribute to the product quality. Few researches studied the effect of this pretreatment on fruit drying, and favourable results have been reported (Amami et al., 2017; Emam-djomeh et al., 2018; Garcia-Noguera et al., 2010). In the present study, however, combination of ultrasound and osmotic dehydration resulted in longer or similar drying times. While the drying time at 210W microwave drying was 223 min for the berries treated with only dipping (D-210W), drying took 212 min for the berries treated with dipping and ultrasound and osmosis (D-40%-210W). When the 60 min of treatment time is considered, the overall processing time is 49 min longer. For hot air drying at 80 °C, the application of ultrasound and osmosis prolonged the drying time for 30 min. That means 90 min longer processing time. Besides, the drying rates for the aforementioned cases were not statistically significant ( $p>0.05$ ). According to these results one can say that, applying ultrasound in apple juice concentrate did not favor the drying process of blueberries increasing processing time will unnecessarily increase the cost. High sugar gain by the blueberry fruit may be responsible for the negative effect of the ultrasound and osmosis treatment on drying rate. Mothibe et al. (2011) represented this case in their study and indicated that the combination of ultrasound with osmotic dehydration may not be practical for the materials which originally have high water content. They explained that, high amount of sugar incorporated during the treatment may concentrate on the surface or may be tightly bound to the water of the fruit;

therefore it could negatively effect the drying process. Fernandes et al. (2008 a) observed an increase in total processing time in air drying of pineapple when ultrasound was carried out in 70 °Brix osmotic solution. This condition was explained by high sugar incorporation acting as a barrier to water transport. Effect of ultrasound and osmosis on pineapple cell structure was investigated by Fernandes et al. (2009), and they demonstrated that the effective moisture diffusivity was increased with 35 °Brix osmotic solution, but it was reduced with 70 °Brix osmotic solution. It was indicated that, when an osmotic solution with high sugar content was employed during ultrasound pretreatment, saturation of microchannels might have limited the mass transfer. Another explanation may be the formation of an outer sugar layer on the surface of the fruit which means an extra resistance to mass transfer.

#### 4.1.3 Effect of Ultrasound Power Level on Drying Characteristics

Effect of ultrasound power on the drying behavior of dipped blueberries is shown in Figure 4.3. Two ultrasound power levels (30% and 40%) were applied to the blueberries during the ultrasound and osmosis treatment. The drying times were similar when the two power levels are compared, and there were no significant differences between the drying rates of these levels ( $p>0.05$ ).



**Figure 4.3** Moisture ratio and drying rate curves for berries dried at 210 W and 80 °C at 30% and 40% ultrasound power levels

According to these results, effects of the power levels in this study are very close to each other. In order to observe a significant effect of ultrasound on drying rates, higher power levels can be tested. On the other hand, when the Figure 4.3 is examined, it seems that there is a difference between hybrid drying and air drying at

these two power levels. While the curves for 30% and 40% power levels pretty much overlaps for air drying, and differences are observed for hybrid drying. This may be related to the drying mechanism of microwave, where the heating is volumetric. Structural changes of fruit occurring at 40% power might have slightly favored the microwave drying.

#### **4.1.4 Effect of Ultrasound Treatment in Water on Drying Characteristics**

Application of ultrasound to the fruits as a pretreatment alters the surface structure to make water removal easier and consequently enhance drying. The material is exposed to rapid compressions and expansions by the ultrasonic waves. This effect resembles a sponge when it is squeezed and released again and again. Hence, the microscopic channels on the surface of the fruit allow better transport of mass (Amami et al., 2017). Fernandes et al. (2008 a) investigated the effect of ultrasound for the drying of pineapple and observed that ultrasound application provided increased water diffusivity and reduced drying time. However, the effect of ultrasound may differ for different fruits (Mothibe et al., 2011). Some may take up water and the moisture content of the fruit is increased, so it may take longer to reach the desired final moisture content. For example, by the application of ultrasound in water medium, papayas, sapotas, genipaps, and bananas gained water, whereas melons and pineapples lost water (Fernandes and Rodrigues, 2008). The drying time of berries pretreated with only dipping and dried at 80 °C (D-80°C) was 270 min (Table 4.2). This time was 314 min for the berries pretreated with dipping and ultrasound in distilled water and then dried at the same temperature (D-40%(Water)-80°C). Application of ultrasound with distilled water being the ultrasonic medium prolonged the time required to dry the berries. The same behaviour was observed for the hybrid drying at 210 W. When we consider the 60 min of ultrasonic pretreatment time, the overall processing time for blueberries is much longer, therefore ultrasound application in distilled water may not be a practical pretreatment for blueberry drying.

Shorter drying times were obtained with the use of osmotic solution during ultrasound treatment compared to distilled water. To illustrate, the drying time of berries treated with dipping and ultrasound treatment in the apple juice concentrate (D-40%-210W) was 212 min for the hybrid drying at 210 W. When the ultrasound

medium was distilled water (D-40%(Water)-210W), this time was 248 min. This means use of apple juice concentrate instead of distilled water is somewhat better.

#### 4.2 Modeling of Blueberry Drying

Mathematical modeling gives information about drying kinetics of materials and therefore it is an important aspect of drying studies in terms of providing valuable knowledge for engineering applications. In this study, five mathematical models were tested for blueberry drying and the one with the highest  $R^2$  and the lowest  $RMSE$  and  $\chi^2$  was considered the best model (Mirzaee et al., 2010). Statistical results of the models are presented in Table 4.3. Midilli et al. was the best model representing the drying kinetics of blueberry for all drying conditions shown in the Table 4.3.  $R^2$  values varied between 0.9978 and 0.9999 for this model.  $RMSE$  results were between 0.0029 and 0.015, and  $\chi^2$  results were between  $1.20 \times 10^{-5}$  and  $3.16 \times 10^{-4}$ . Aral and Beşe (2016) reported that Midilli et al. model was the best appropriate model for the convective drying of hawthorn fruit. Bingol et al. (2012) investigated the effect of various dipping conditions on grape drying and selected the Midilli et al. model as the best for describing the drying curves for all dipping pretreatments. This model has also been suggested for convective, microwave, and microwave–convective drying of goldenberry by Izli et al. (2014).

Rate constant (k) values varied between 0.0004 and 0.0026 for the Midilli et al. model. k values increased as the temperature and microwave power level increased. Generally, k values of the hybrid drying runs are higher than the k values of the air drying runs.

**Table 4.3** Statistical results for model parameters for varying drying conditions

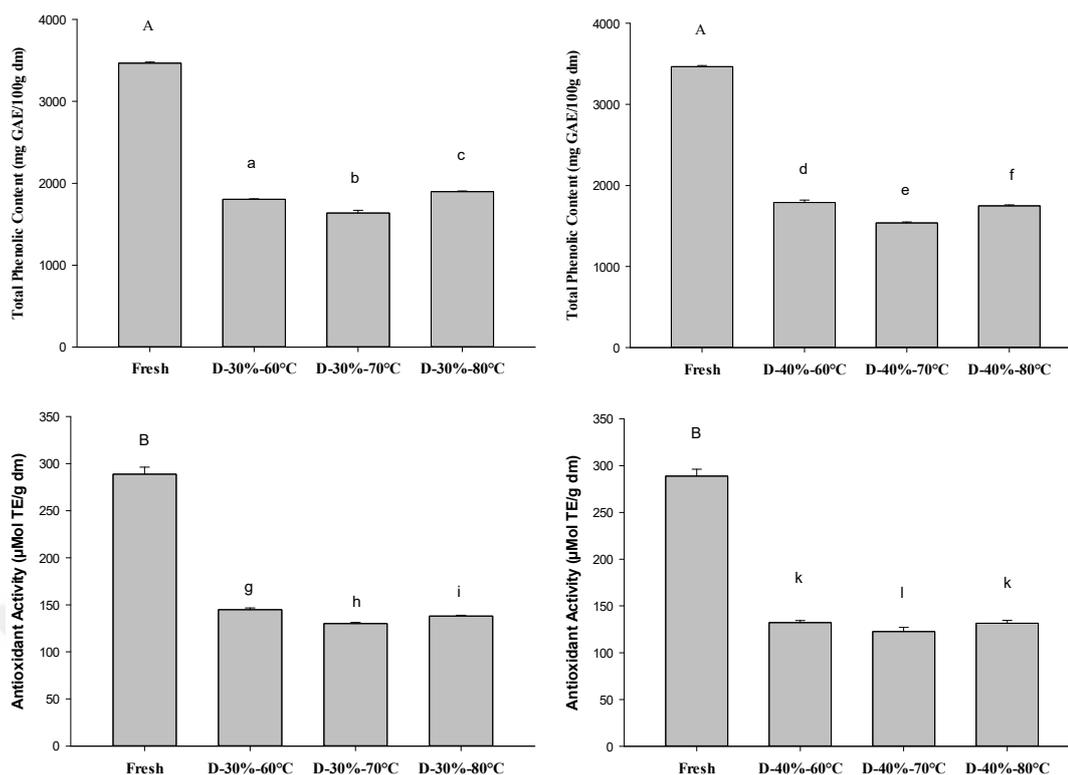
<b>Drying condition</b>		<b>Page</b>	<b>Logar.</b>	<b>Midilli</b>	<b>H&amp;P</b>	<b>mod.Log</b>
D-30%-150W	$R^2$	0.9951	0.9985	0.9995	0.9701	0.9978
	$RMSE$	0.0212	0.0119	0.0068	0.0523	0.0142
	$\chi^2 \times 10^4$	4.81	1.57	0.54	29.37	2.24
	$k \times 10^3$	0.2	1.2	0.4	2.7	1.6
D-30%-180W	$R^2$	0.9973	0.9975	0.9996	0.9732	0.9988
	$RMSE$	0.0164	0.0156	0.0064	0.0514	0.0110
	$\chi^2 \times 10^4$	3.11	3.03	0.56	30.47	1.51
	$k \times 10^3$	0.5	2.9	0.9	5.6	3.4
D-30%-210W	$R^2$	0.9942	0.9974	0.9990	0.9535	0.9965
	$RMSE$	0.0220	0.0148	0.0090	0.0622	0.0170
	$\chi^2 \times 10^4$	5.66	2.79	1.12	45.17	3.70
	$k \times 10^3$	0.6	3.5	1.5	9.2	5.6
D-40%-150W	$R^2$	0.9933	0.9993	0.9996	0.9721	0.9971
	$RMSE$	0.0240	0.0078	0.0062	0.0492	0.0158
	$\chi^2 \times 10^4$	6.21	0.68	0.45	26.02	2.78
	$k \times 10^3$	0.3	1.1	0.7	2.7	1.6
D-40%-180W	$R^2$	0.9984	0.9988	0.9999	0.9839	0.9989
	$RMSE$	0.0122	0.0108	0.0029	0.0391	0.0102
	$\chi^2 \times 10^4$	1.77	1.50	0.12	18.08	1.35
	$k \times 10^3$	1.2	4.0	2.0	6.4	4.0
D-40%-210W	$R^2$	0.9936	0.9947	0.9978	0.9476	0.9970
	$RMSE$	0.0257	0.0233	0.0150	0.0735	0.0177
	$\chi^2 \times 10^4$	7.72	6.91	3.16	63.10	3.97
	$k \times 10^3$	0.3	2.7	2.6	9.9	6.3
D-30%-60°C	$R^2$	0.9957	0.9981	0.9989	0.9790	0.9980
	$RMSE$	0.0200	0.0132	0.0103	0.0442	0.0137
	$\chi^2 \times 10^4$	4.55	2.12	1.39	22.12	2.28
	$k \times 10^3$	0.3	1.4	0.6	2.5	1.5
D-30%-70°C	$R^2$	0.9961	0.9980	0.9989	0.9809	0.9982
	$RMSE$	0.0192	0.0136	0.0103	0.0422	0.0131
	$\chi^2 \times 10^4$	4.16	2.26	1.37	20.18	2.08
	$k \times 10^3$	0.9	3.1	1.4	5.0	3.0
D-30%-80°C	$R^2$	0.9977	0.9980	0.9995	0.9807	0.9989
	$RMSE$	0.0152	0.0141	0.0073	0.0441	0.0104
	$\chi^2 \times 10^4$	2.76	2.66	0.79	23.36	1.43
	$k \times 10^3$	1.4	4.9	2.1	7.9	4.7
D-40%-60°C	$R^2$	0.9945	0.9979	0.9984	0.9788	0.9973
	$RMSE$	0.0227	0.0139	0.0122	0.0446	0.0159
	$\chi^2 \times 10^4$	5.79	2.33	1.92	22.33	3.04
	$k \times 10^3$	0.4	1.5	0.7	2.5	1.5
D-40%-70°C	$R^2$	0.9942	0.9993	0.9995	0.9755	0.9969
	$RMSE$	0.0229	0.0080	0.0070	0.0472	0.0169
	$\chi^2 \times 10^4$	6.01	0.80	0.65	25.48	3.50
	$k \times 10^3$	0.7	2.3	1.7	4.8	2.9
D-40%-80°C	$R^2$	0.9973	0.9987	0.9997	0.9791	0.9985
	$RMSE$	0.0163	0.0113	0.0051	0.0455	0.0121
	$\chi^2 \times 10^4$	3.18	1.71	0.39	24.85	1.94
	$k \times 10^3$	1.4	4.5	2.4	7.9	4.8

### **4.3 Total Phenolics Content (TPC) and Antioxidant Activity (AA) of Blueberry**

#### **4.3.1 Effect of Drying Methods on TPC and AA**

In this study, the total phenolics content of fresh blueberry was determined as  $3466.43 \pm 11.35$  mg GAE/100g dm. This value is in agreement with other studies (An et al., 2019; Castrejon et al., 2008; Somsong et al., 2010; Stojanovic and Silva, 2007). All drying conditions led to significant reductions in TPC ( $p < 0.05$ ) as shown in the Figures 4.4 and 4.5. The lowest TPC loss (32.29%) was obtained from D-30%-180W sample. The highest TPC loss (55.65%) was obtained from hot air drying at 70 °C (D-40%-70°C). During thermal processing, phytochemicals can be lost by disruption of cell structure resulting in leakage of these components or breakdown by chemical reactions induced by enzymes, light and oxygen (Youssef and Mokhtar, 2014). Also, TPC can be lowered by drying processes because polyphenols can bind with other components such as proteins, or chemical changes in polyphenol structures can occur which cannot be determined by methods in use (Jessica et al, 2013).

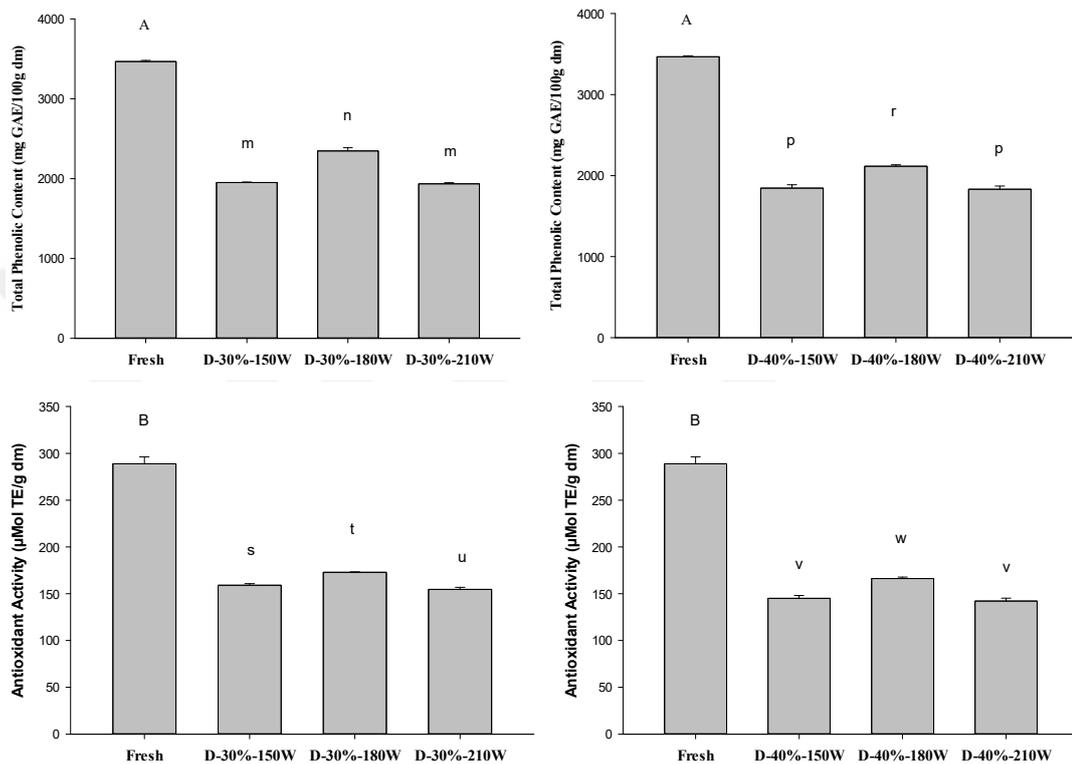
Antioxidant activity of fresh blueberry was found as  $288.76 \pm 6.08$   $\mu\text{mol TE/g dm}$ . Lohachoompol et al. (2008) reported the antioxidant activity of different blueberry cultivars in the range of 67.3 and 155.7  $\mu\text{mol TE/g dm}$  by DPPH assay. AA obtained in the present study is higher than these values. As observed in the case of TPC, all drying conditions resulted in significant losses in AA ( $p < 0.05$ ) as shown in the Figures 4.4 and 4.5. Antioxidant activity losses were between 40.13% and 57.56%. AA values of dried blueberries exhibited high correlation with TPC ( $r = 0.942$ ,  $p < 0.01$ ).



**Figure 4.4** TPC and AA levels for hot air drying of blueberries

There were significant differences between the three levels of temperature applied in hot air drying ( $p < 0.05$ ) as shown in the Figures 4.4. TPC of samples D-30%-60°C and D-40%-60°C were  $1805.86 \pm 3.82$  mg GAE/100g dm and  $1791.62 \pm 23.45$  mg GAE/100g dm, respectively. At 70 °C, these values were decreased to  $1635.45 \pm 27.66$  mg GAE/100g dm for 30% ultrasound power level and  $1537.24 \pm 10.37$  mg GAE/100g dm for 40% ultrasound power level, likely due to the increase in temperature. The loss of phenolics was attributed particularly to the temperature and processing time conditions (Youssef and Moktar, 2014). At 80 °C, despite the increasing temperature, phenolics contents increased ( $p < 0.05$ ). A substantial reduction in drying time may be the reason for this increase. Also, as it was reported in some studies (Udomkun et al., 2015; Jéssica et al., 2013; Somsong et al., 2010; Zielinska and Michalska, 2016), at high temperatures TPC may show a tendency to increase. Udomkun et al. (2015) explained this increase with the formation of phenolic compounds at high temperatures due to the presence of phenolic precursor molecules resulting from the nonenzymic interconversion between the phenolic molecules. López-Vidaña et al. (2017) suggested that inactivation of oxidative and hydrolytic enzymes at high temperatures could prevent phenolics decomposition.

They also associated the increase in antioxidant activity at high temperatures to the generation of melanoidins and Maillard reaction products since they also behave as antioxidant substances. As in the case of TPC, AA values increased at 80 °C air temperature for both power levels of ultrasound (30% and 40%). López-Vidaña et al. (2017) observed a similar phenolics content trend with this study for the temperatures of 60, 70, and 80°C in hot air drying of blueberry.



**Figure 4.5** TPC and AA levels for hybrid drying of blueberries

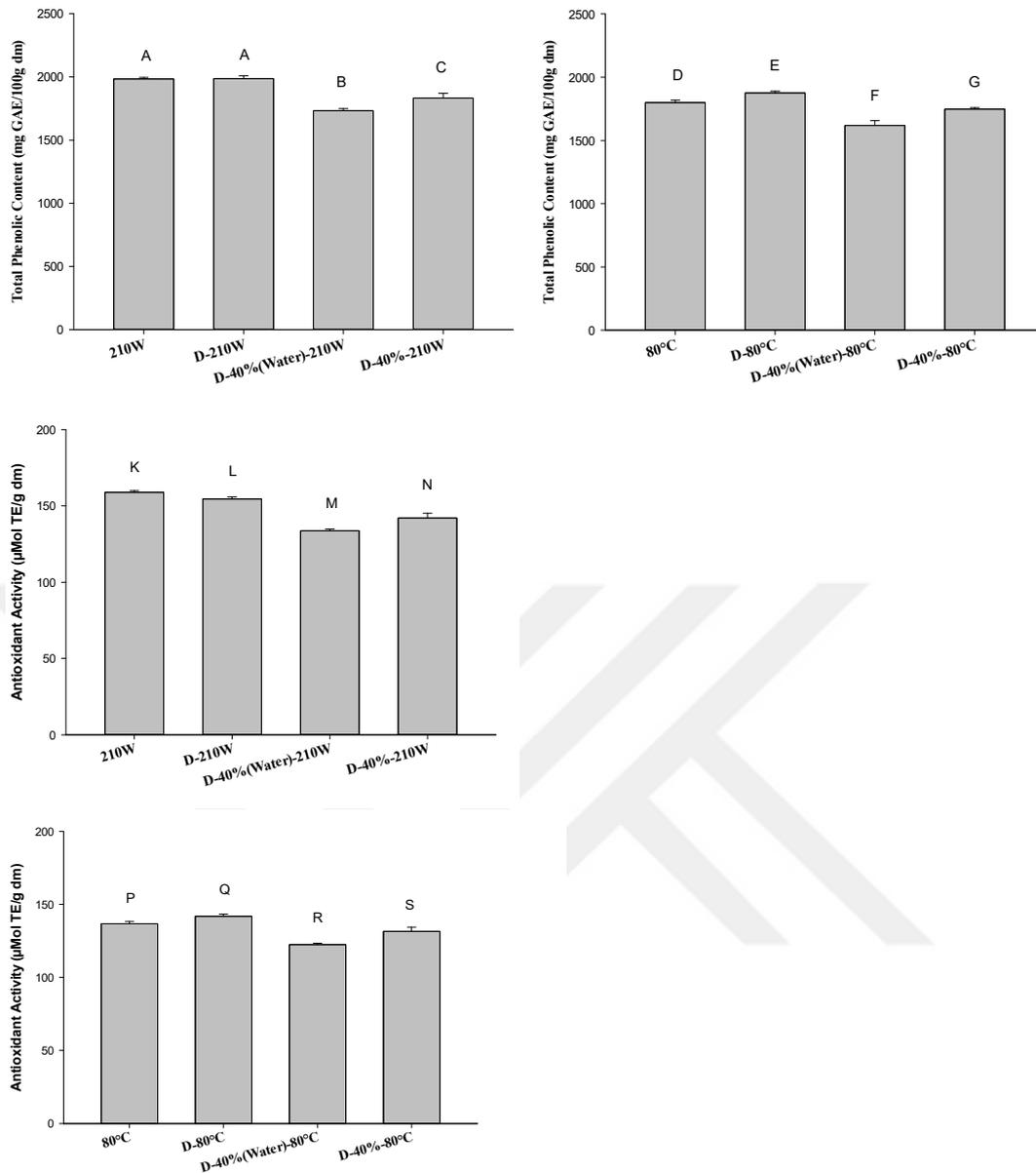
According to the Figure 4.5, berries dried in hybrid oven showed a different trend in TPC and AA values than hot air drying. The increase of microwave power to 180 W significantly increased TPC values ( $p < 0.05$ ) for both ultrasound power levels, and then, they were significantly decreased at 210 W power level. By increasing the power to 180 W, the drying time was considerably decreased, which can be the reason of an increase in TPC. However, at 210 W power level, the application of high microwave power led to a significant decrease in TPC. Antioxidant activity analysis resulted in a similar trend with TPC, increasing at 180 W and decreasing at 210 W. The negative effect of high power overtook the effect of decreasing drying time. Rapid moisture transfer and structural changes such as cell wall damage, shrinkage and surface microcracking can be the reason of such decrease (Zielinska et

al., 2018). Also, formation of hot spots due to the nonhomogeneity of microwave heating and increase in product temperature can be responsible for low TPC and AA at higher power level. Similar effects of high microwave power on bioactive components were observed for the drying of cranberries (Zielinska et al., 2018) and sour cherries (Wojdylo et al., 2014).

When the two drying methods are compared, all hybrid drying conditions resulted in significantly higher TPC and AA values than their hot air drying equivalents ( $p < 0.05$ ). For example, the TPC was  $1951.46 \pm 5.10$  mg GAE/100g dm and  $1805.86 \pm 3.82$  mg GAE/100g dm for D-30%-150W and D-30%-60°C samples, respectively. Similar results were obtained by Carranza-Concha et al. (2012), Izli et al. (2014), and Horuz et al. (2017) for raisins, goldenberry, and sour cherry samples, respectively. Reduced drying times, provided by the microwave energy may result in higher retention of phenolic and antioxidant compounds compared to the convectonal drying, where the materials are exposed to oxygen for longer times.

#### **4.3.2 Effect of Pretreatments on TPC and AA**

To evaluate the effect of ultrasound and osmosis on total phenolics content and antioxidant activity of dried blueberries, a comparison can be made between dried berries subjected to only dipping and dried berries subjected to both dipping and ultrasound and osmosis. This effect is shown in Figure 4.6.



**Figure 4.6** TPC and AA levels of blueberries for varying pretreatments

For both drying methods, air and hybrid drying, application of ultrasonic osmosis treatment led to a significant decrease ( $p < 0.05$ ) in TPC and AA values. Oxidation of bioactive compounds can occur during osmotic dehydration and/or ultrasound treatments, which leads to loss of phenolics and reduction in antioxidant activity. Ultrasound application may contribute to the free radical formation and phenolics content may decrease due to the increased level of polymerization (Hamedi et al., 2018).

Two different ultrasound power levels (30% and 40%) were used in this study during the ultrasound and osmosis treatment of blueberries. Significant differences ( $p < 0.05$ )

between these power levels were obtained for all the TPC and AA values of dried blueberries except the TPC value of air drying at 60 °C. Lower levels of total phenols and antioxidant activity were observed at 40% ultrasound power. TPC values for D-30%-180W and D-40%-180W samples were  $2347.20 \pm 34.33$  mg GAE/100g dm and  $2115.77 \pm 15.40$  mg GAE/100g dm, respectively. AA values for D-30%-180W and D-40%-180W were  $172.89 \pm 0.46$   $\mu\text{mol TE/g dm}$  and  $166.24 \pm 1.26$   $\mu\text{mol TE/g dm}$ , respectively. Ruptures occurring on the skin layer due to the cavitation phenomenon can lead to the leakage of bioactive substances to the ultrasound medium (Stojanovic and Silva, 2007). Increasing the power level may induce more changes in surface structure of berries and cause more leakage of these materials. Also, increasing the ultrasound power may increase free radical formation.

Significantly lower levels of TPC and AA were obtained ( $p < 0.05$ ) when the ultrasound pretreatment was performed in distilled water compared to the application of ultrasound in apple juice concentrate (Figure 4.6). Ultrasound is known to alter the skin surface structure of the materials to allow better mass transfer. Due to the changes on blueberry skin caused by ultrasonic waves, the leakage of phenolics and antioxidant compounds to the water medium may take place. The leakage of polyphenolic compounds to the water medium was reported by Mieszczakowska-Frac et al. (2016). They stated that the ultrasound treatment caused an open structure that caused a considerable polyphenols leakage when distilled water was used as ultrasonic medium; whereas sucrose solution prevented the leakage of highly polymerized molecules. In addition, some phenolic compounds may be transferred to the berries by the immersion in apple juice concentrate during ultrasound pretreatment, which may explain higher phenolic levels. This behaviour was observed by Kowalska et al. (2017) and Lech et al. (2018) in the osmotic dehydration of *Honeoye* strawberries and pumpkin, respectively.

#### **4.4 Color Parameters**

Color values for varying drying conditions were given in Table 4.4 and Table 4.5. Generally, all color properties were similar to each other and there were no distinguishable differences.

**Table 4.4** Color parameters for varying drying conditions

Drying condition	$L^*$	$a^*$	$b^*$
D-30%-150W	13.45 ± 0.03 <sup>a,A,O</sup>	4.95 ± 0.07 <sup>a,A,O</sup>	-0.19 ± 0.03 <sup>a,A,O</sup>
D-30%-180W	13.60 ± 0.19 <sup>a,C,Q</sup>	4.45 ± 0.22 <sup>b,C,Q</sup>	-0.46 ± 0.05 <sup>b,C,Q</sup>
D-30%-210W	12.68 ± 0.10 <sup>b,E,S</sup>	4.18 ± 0.20 <sup>b,E,S</sup>	0.13 ± 0.04 <sup>c,E,S</sup>
D-40%-150W	13.75 ± 0.05 <sup>d,B,U</sup>	4.58 ± 0.13 <sup>d,B,U</sup>	-0.13 ± 0.06 <sup>d,A,U</sup>
D-40%-180W	13.60 ± 0.37 <sup>d,C,W</sup>	4.56 ± 0.20 <sup>d,C,W</sup>	-0.30 ± 0.05 <sup>e,D,W</sup>
D-40%-210W	12.76 ± 0.22 <sup>e,E,Y</sup>	4.37 ± 0.34 <sup>d,E,Y</sup>	0.17 ± 0.08 <sup>f,E,Y</sup>
D-30%-60°C	12.48 ± 0.31 <sup>g,G,P</sup>	3.78 ± 0.10 <sup>g,K,P</sup>	-0.11 ± 0.06 <sup>g,G,O</sup>
D-30%-70°C	12.86 ± 0.19 <sup>g,K,R</sup>	4.65 ± 0.14 <sup>h,K,Q</sup>	-0.09 ± 0.03 <sup>g,K,R</sup>
D-30%-80°C	12.69 ± 0.35 <sup>g,M,S</sup>	4.33 ± 0.19 <sup>h,M,S</sup>	-0.15 ± 0.07 <sup>g,M,T</sup>
D-40%-60°C	12.49 ± 0.33 <sup>k,G,V</sup>	4.45 ± 0.31 <sup>k,H,U</sup>	-0.21 ± 0.06 <sup>k,G,U</sup>
D-40%-70°C	12.96 ± 0.17 <sup>k,K,W</sup>	5.08 ± 0.24 <sup>k,K,W</sup>	-0.12 ± 0.05 <sup>k,K,X</sup>
D-40%-80°C	12.84 ± 0.20 <sup>k,M,Y</sup>	4.83 ± 0.17 <sup>k,N,Y</sup>	-0.14 ± 0.10 <sup>k,M,Z</sup>

Data are shown as the average and standard deviations for three replicates. Values in the same column having different letters for each parameter represent significant difference at a confidence level of 95% (a-b-c for hybrid drying at 30% US power; d-e-f for hybrid drying at 40% US power; g-h-i for air drying at 30% US power; k-l-m for air drying at 40% US power; A-B, C-D, E-F, G-H, K-L, and M-N for dryings at 150W, 180W, 210W, 60°C, 70°C, and 80°C, respectively, at 30%-40% US powers; O-P for dryings at 150W-60°C at 30% US power; Q-R for dryings at 180W-70°C at 30% US power; S-T for dryings at 210W-80°C at 30% US power; U-V for dryings at 150W-60°C at 40% US power; W-X for dryings at 180W-70°C at 40% US power; Y-Z for dryings at 210W-80°C at 40% US power).

**Table 4.5** Color parameters for varying pretreatments

Drying condition	$L^*$	$a^*$	$b^*$
210W	16.96 ± 0.15 <sup>a</sup>	4.85 ± 0.07 <sup>a</sup>	0.15 ± 0.09 <sup>a</sup>
D-210W	14.68 ± 0.23 <sup>b</sup>	4.53 ± 0.11 <sup>a</sup>	0.26 ± 0.02 <sup>a</sup>
D-40% (Water) -210W	14.77 ± 0.14 <sup>b</sup>	5.67 ± 0.26 <sup>b</sup>	0.46 ± 0.10 <sup>b</sup>
D-40%-210W	12.76 ± 0.22 <sup>c</sup>	4.37 ± 0.34 <sup>a</sup>	0.17 ± 0.08 <sup>a</sup>
80°C	15.11 ± 0.13 <sup>e</sup>	4.28 ± 0.28 <sup>e</sup>	-0.13 ± 0.05 <sup>e</sup>
D-80°C	13.50 ± 0.24 <sup>f</sup>	4.65 ± 0.20 <sup>e,g</sup>	-0.17 ± 0.10 <sup>e</sup>
D-40% (Water) -80°C	13.48 ± 0.22 <sup>f</sup>	6.49 ± 0.27 <sup>f</sup>	0.27 ± 0.04 <sup>f</sup>
D-40%-80°C	12.84 ± 0.20 <sup>g</sup>	4.83 ± 0.17 <sup>e</sup>	-0.14 ± 0.10 <sup>e</sup>

Data are shown as the average and standard deviations for three replicates. Values in the same column having different letters for each parameter represent significant difference at a confidence level of 95% (a-b-c-d for dryings at 210W and e-f-g-h for dryings at 80°C).

Berries dried without pretreatment applications (210W and 80°C) gave slightly higher  $L^*$  values. That means these berries were brighter than the others. This may be due to the removal of surface bloom by the dipping pretreatment. This layer appears as a bluish white coating (Chu et al., 2017). Giovanelli et al. (2012) studied the blanching pretreatment of blueberries and observed that unblanched blueberries had higher luminance values, which is an index of lightness of the colour, compared to the blanched berries. They indicated that unblanched berries were brighter than the blanched berries because the waxy skin layer was removed by the blanching pretreatment. Among hybrid drying conditions, drying at 210 W power level resulted in slightly lower lightness values compared to 150 W and 180 W levels. Overheating and formation of hot spots at higher microwave power levels may be the reason for the darker berries. Beaudry et al. (2003) observed more brown or black colored cranberries due to the presence of burned berries dried at higher power levels. Lightness values of all air drying conditions were very similar and not statistically significant ( $P > 0.05$ ) among drying temperatures. There were also no significant differences between the ultrasound power levels (30% and 40%). Therefore, one can say that ultrasound power level does not affect the lightness of dried blueberries. When the ultrasonic medium is considered, berries pretreated by ultrasonic osmosis (D-40-210W and D-40-80°C) resulted in lower  $L^*$  values than the berries treated by ultrasound in distilled water (D-40Water-210W and D-40Water-80°C). That is, osmotic dehydration with apple juice concentrate caused darker berries.

Similar  $a^*$  values were obtained for both hybrid drying and air drying conditions. According to these results, it can be said that, neither the microwave power levels in hybrid drying nor the temperature levels in air drying affects the redness of blueberries in the conditions tested in this study. This behaviour was also observed by Horuz et al. (2017). Application of dipping and ultrasound-assisted osmotic dehydration pretreatments did not make a significant change in redness of berries as well. On the other hand, pretreating berries with ultrasound in water medium led to slightly higher  $a^*$  values for both hybrid drying and hot air drying.

Significant differences of  $b^*$  values for the microwave power levels were observed in hybrid drying for both ultrasound power levels. Berries dried at 180 W gave more negative  $b^*$  values, that is, they were bluer than the others. Hence, drying of blueberry by hybrid drying at 180 W power level can be more acceptable due to the the relatively higher retention of original blue color. This condition also had the highest phenolics and antioxidant levels, which can be related to the color characteristics. On the other hand, drying at 210 W resulted in positive  $b^*$  values indicating the occurrence of yellowness. Again, this can be the result of overheating problems associated with high microwave power levels. There were no significant differences ( $P>0.05$ ) between air drying conditions. Also, the differences between ultrasound power levels (30% and 40%) were not significant. When the pretreatments are compared, only ultrasound in water medium led to significantly higher  $b^*$  values ( $P<0.05$ ). At this condition,  $a^*$  values were higher, too. When considered together, it can be said that the berries got more orange. The structural changes caused by ultrasound waves, which may be more effective in distilled water rather than a dense osmotic medium may be responsible for this situation. Also, the loss of blue, purple colored pigments to the water medium might have taken place.

According to the visual observations redness of blueberries increased during drying processes and the blueness decreased. When visually observed, there were more brown colors in berries dried at 210 W and berries pretreated by ultrasound in distilled water. Also, the use of apple juice concentrate as the ultrasound medium gave a glossy or polished appearance to the berries, whereas other conditions which do not involve osmosis resulted in dull berries. Therefore, use of juice concentrate as an osmotic medium may be more acceptable to the consumer (Figure 4.7). Other than that, there were no distinguishable differences between dried berries in all conditions.



**Figure 4.7** Photographs of dried blueberries: a) ultrasonically pretreated with distilled water; b) ultrasonically pretreated with apple juice concentrate

## CHAPTER 5

### CONCLUSION

Study of blueberries by different pretreatments and drying processes revealed the following conclusions:

Chemical dipping treatment reduced drying times of blueberry; however this reduction was not significant in terms of drying rates. Ultrasound pretreatments performed with apple juice concentrate or with distilled water prolonged the total processing times, therefore application of ultrasound was not considered a practical and effective pretreatment method for blueberry within the conditions tested in this work. Also, two different ultrasound power levels were tested and drying times were similar for these levels. Hybrid drying resulted in shorter drying times compared to air drying. In hybrid drying, as the microwave power level increased, drying times decreased and drying rates increased significantly. In air drying, the increase in air temperature reduced the drying times and increased drying rates significantly. Five different mathematical models were applied to the drying data and Midilli et al. was selected as the model best describing the drying behaviour of blueberry.

All drying experiments led to significant losses of phenolics and antioxidant molecules. A high correlation was observed between TPC and AA levels. Ultrasound pretreatment caused significant reduction in both phenolics content and antioxidant activity, but use of apple juice concentrate during this treatment showed better results compared to the use of distilled water.

Color values were generally similar among all drying conditions, although slight differences were recorded. Removal of the bluish white colored skin layer by dipping lowered the lightness of berries.

In summary, application of ultrasounds as a pretreatment for drying of blueberry fruit did not favor the drying characteristics and product quality. Different application times and power levels can be tested. On the other hand, hybrid drying method looks promising and open to improvements. The conditions experimented in this study may

be guiding for other studies in order to improve current techniques for blueberry drying. Further studies are needed to determine drying conditions to preserve the optimal physical and nutritive properties of dried blueberries. Also, the results of this study may be helpful to the manufacturers of industrial dryers.



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**APPENDIX**

**Table A.1** Graph data for the proposed model for D-30%-60°C

<b>Time</b>	<b>Moiture Ratio</b>	<b>Parameters</b>	<b>Predicted</b>	<b>Residuals</b>
0,0000	1,0000	0,9879	0,9879	0,0121
60,0000	0,8963	0,0006	0,9079	-0,0117
120,0000	0,8056	1,1962	0,8145	-0,0089
180,0000	0,7173	-0,0001	0,7214	-0,0041
240,0000	0,6300		0,6325	-0,0025
300,0000	0,5555		0,5494	0,0061
360,0000	0,4848		0,4728	0,0121
420,0000	0,4155		0,4029	0,0126
480,0000	0,3464		0,3396	0,0068
540,0000	0,2840		0,2826	0,0014
600,0000	0,2276		0,2314	-0,0038
660,0000	0,1754		0,1857	-0,0103
720,0000	0,1303		0,1448	-0,0145
780,0000	0,0951		0,1084	-0,0133
840,0000	0,0681		0,0760	-0,0079
900,0000	0,0514		0,0471	0,0043
963,0000	0,0411		0,0201	0,0211

**Table A.2** Graph data for the proposed model for D-30%-70°C

<b>Time</b>	<b>Moiture Ratio</b>	<b>Parameters</b>	<b>Predicted</b>	<b>Residuals</b>
0,0000	1,0000	0,9879	0,9879	0,0121
30,0000	0,8977	0,0014	0,9066	-0,0089
60,0000	0,7983	1,1984	0,8115	-0,0133
90,0000	0,7097	-0,0002	0,7170	-0,0073
120,0000	0,6297		0,6270	0,0027
150,0000	0,5491		0,5433	0,0058
180,0000	0,4798		0,4666	0,0132
210,0000	0,4090		0,3970	0,0120
240,0000	0,3426		0,3343	0,0083
270,0000	0,2795		0,2782	0,0014
300,0000	0,2226		0,2281	-0,0055
330,0000	0,1725		0,1837	-0,0112
360,0000	0,1289		0,1444	-0,0155
390,0000	0,0973		0,1095	-0,0123
420,0000	0,0717		0,0788	-0,0070
480,0000	0,0383		0,0275	0,0108
487,0000	0,0362		0,0223	0,0139

**Table A.3** Graph data for the proposed model for D-30%-80°C

Time	Moiture Ratio	Parameters	Predicted	Residuals
0,0000	1,0000	0,9939	0,9939	0,0061
30,0000	0,8505	0,0021	0,8622	-0,0117
60,0000	0,7098	1,2279	0,7120	-0,0022
90,0000	0,5784	-0,0002	0,5722	0,0063
120,0000	0,4550		0,4492	0,0058
150,0000	0,3494		0,3445	0,0049
180,0000	0,2594		0,2571	0,0023
210,0000	0,1784		0,1852	-0,0068
240,0000	0,1162		0,1266	-0,0104
270,0000	0,0703		0,0791	-0,0089
300,0000	0,0446		0,0408	0,0038
313,0000	0,0369		0,0265	0,0104

**Table A.4** Graph data for the proposed model for D-40%-60°C

Time	Moiture Ratio	Parameters	Predicted	Residuals
0,0000	1,0000	0,9848	0,9848	0,0152
60,0000	0,8863	0,0007	0,9007	-0,0144
120,0000	0,7914	1,1732	0,8053	-0,0139
180,0000	0,7055	-0,0001	0,7115	-0,0060
240,0000	0,6258		0,6228	0,0030
300,0000	0,5485		0,5404	0,0081
360,0000	0,4781		0,4649	0,0133
420,0000	0,4089		0,3962	0,0127
480,0000	0,3428		0,3341	0,0086
540,0000	0,2800		0,2783	0,0016
600,0000	0,2273		0,2283	-0,0010
660,0000	0,1721		0,1836	-0,0115
720,0000	0,1249		0,1437	-0,0188
780,0000	0,0896		0,1081	-0,0184
840,0000	0,0646		0,0763	-0,0118
900,0000	0,0471		0,0480	-0,0009
960,0000	0,0382		0,0227	0,0156

**Table A.5** Graph data for the proposed model for D-40%-70°C

<b>Time</b>	<b>Moiture Ratio</b>	<b>Parameters</b>	<b>Predicted</b>	<b>Residuals</b>
0,0000	1,0000	0,9945	0,9945	0,0055
30,0000	0,9052	0,0017	0,9090	-0,0038
60,0000	0,8098	1,1249	0,8171	-0,0073
90,0000	0,7238	-0,0004	0,7276	-0,0038
120,0000	0,6429		0,6426	0,0003
150,0000	0,5679		0,5628	0,0051
180,0000	0,4955		0,4883	0,0071
210,0000	0,4264		0,4193	0,0071
240,0000	0,3571		0,3555	0,0015
270,0000	0,2994		0,2966	0,0028
300,0000	0,2392		0,2424	-0,0032
330,0000	0,1865		0,1925	-0,0059
360,0000	0,1370		0,1465	-0,0095
390,0000	0,0949		0,1042	-0,0092
420,0000	0,0612		0,0651	-0,0039
456,0000	0,0393		0,0222	0,0171

**Table A.6** Graph data for the proposed model for D-40%-80°C

<b>Time</b>	<b>Moiture Ratio</b>	<b>Parameters</b>	<b>Predicted</b>	<b>Residuals</b>
0,0000	1,0000	0,9964	0,9964	0,0036
30,0000	0,8523	0,0024	0,8590	-0,0067
60,0000	0,7053	1,1970	0,7083	-0,0030
70,0000	0,6607	-0,0003	0,6602	0,0005
100,0000	0,5327		0,5265	0,0062
130,0000	0,4150		0,4101	0,0049
160,0000	0,3139		0,3109	0,0030
190,0000	0,2246		0,2276	-0,0030
220,0000	0,1506		0,1582	-0,0076
250,0000	0,0949		0,1007	-0,0059
280,0000	0,0525		0,0532	-0,0006

**Table A.7** Graph data for the proposed model for D-30%-150W

<b>Time</b>	<b>Moiture Ratio</b>	<b>Parameters</b>	<b>Predicted</b>	<b>Residuals</b>
0,0000	1,0000	0,9862	0,9862	0,0138
30,0000	0,9516	0,0004	0,9542	-0,0026
60,0000	0,9055	1,2675	0,9122	-0,0068
90,0000	0,8577	-0,0002	0,8666	-0,0089
120,0000	0,8146		0,8191	-0,0045
150,0000	0,7671		0,7709	-0,0038
180,0000	0,7221		0,7228	-0,0006
210,0000	0,6736		0,6752	-0,0016
240,0000	0,6320		0,6286	0,0035
270,0000	0,5866		0,5831	0,0035
300,0000	0,5432		0,5391	0,0041
330,0000	0,4999		0,4966	0,0033
360,0000	0,4596		0,4557	0,0040
390,0000	0,4217		0,4164	0,0053
420,0000	0,3856		0,3789	0,0067
450,0000	0,3477		0,3431	0,0046
480,0000	0,3134		0,3090	0,0044
510,0000	0,2783		0,2765	0,0017
540,0000	0,2441		0,2457	-0,0016
570,0000	0,2107		0,2165	-0,0058
600,0000	0,1814		0,1889	-0,0075
630,0000	0,1527		0,1627	-0,0100
660,0000	0,1279		0,1379	-0,0100
690,0000	0,1046		0,1145	-0,0099
720,0000	0,0858		0,0924	-0,0066
750,0000	0,0680		0,0715	-0,0035
780,0000	0,0542		0,0518	0,0024
810,0000	0,0439		0,0332	0,0108
825,0000	0,0395		0,0242	0,0152

**Table A.8** Graph data for the proposed model for D-30%-180W

<b>Time</b>	<b>Moiture Ratio</b>	<b>Parameters</b>	<b>Predicted</b>	<b>Residuals</b>
0,0000	1,0000	0,9918	0,9918	0,0082
30,0000	0,9065	0,0009	0,9159	-0,0094
60,0000	0,8112	1,2997	0,8163	-0,0052
90,0000	0,7128	-0,0002	0,7130	-0,0003
120,0000	0,6128		0,6126	0,0002
150,0000	0,5253		0,5184	0,0068
180,0000	0,4381		0,4322	0,0059
210,0000	0,3582		0,3545	0,0037
240,0000	0,2869		0,2854	0,0015
270,0000	0,2209		0,2246	-0,0038
300,0000	0,1670		0,1715	-0,0045
330,0000	0,1170		0,1254	-0,0084
360,0000	0,0776		0,0855	-0,0079
390,0000	0,0510		0,0511	-0,0002
419,0000	0,0353		0,0224	0,0129

**Table A.9** Graph data for the proposed model for D-30%-210W

<b>Time</b>	<b>Moiture Ratio</b>	<b>Parameters</b>	<b>Predicted</b>	<b>Residuals</b>
0,0000	1,0000	0,9945	0,9945	0,0055
40,0000	0,7936	0,0015	0,8076	-0,0141
60,0000	0,6934	1,3062	0,6980	-0,0046
90,0000	0,5472	-0,0006	0,5398	0,0074
105,0000	0,4826		0,4664	0,0162
120,0000	0,4054		0,3976	0,0078
135,0000	0,3308		0,3337	-0,0028
150,0000	0,2739		0,2747	-0,0007
165,0000	0,2142		0,2205	-0,0062
180,0000	0,1597		0,1709	-0,0112
195,0000	0,1172		0,1258	-0,0086
210,0000	0,0786		0,0847	-0,0060
225,0000	0,0505		0,0474	0,0031
235,0000	0,0386		0,0244	0,0141

**Table A.10** Graph data for the proposed model for D-40%-150W

Time	Moiture Ratio	Parameters	Predicted	Residuals
0,0000	1,0000	0,9857	0,9857	0,0143
30,0000	0,9407	0,0007	0,9457	-0,0050
60,0000	0,8912	1,1624	0,8993	-0,0081
90,0000	0,8411	-0,0002	0,8512	-0,0101
120,0000	0,7971		0,8029	-0,0058
150,0000	0,7521		0,7549	-0,0028
180,0000	0,7078		0,7078	0,0001
210,0000	0,6646		0,6617	0,0029
240,0000	0,6231		0,6168	0,0063
270,0000	0,5804		0,5733	0,0071
300,0000	0,5384		0,5312	0,0071
330,0000	0,4951		0,4906	0,0045
360,0000	0,4559		0,4515	0,0044
390,0000	0,4172		0,4138	0,0033
420,0000	0,3799		0,3777	0,0022
450,0000	0,3426		0,3429	-0,0004
480,0000	0,3085		0,3096	-0,0011
510,0000	0,2741		0,2777	-0,0036
540,0000	0,2417		0,2471	-0,0053
570,0000	0,2119		0,2178	-0,0058
600,0000	0,1843		0,1897	-0,0054
630,0000	0,1569		0,1628	-0,0059
660,0000	0,1327		0,1371	-0,0044
690,0000	0,1089		0,1125	-0,0036
720,0000	0,0873		0,0889	-0,0016
750,0000	0,0639		0,0663	-0,0024
780,0000	0,0480		0,0447	0,0033
813,0000	0,0377		0,0220	0,0157

**Table A.11** Graph data for the proposed model for D-40%-180W

Time	Moiture Ratio	Parameters	Predicted	Residuals
0,0000	1,0000	1,0004	1,0004	-0,0004
30,0000	0,8868	0,0020	0,8874	-0,0006
60,0000	0,7646	1,1918	0,7605	0,0040
90,0000	0,6392	-0,0002	0,6400	-0,0007
120,0000	0,5259		0,5304	-0,0045
150,0000	0,4308		0,4332	-0,0024
180,0000	0,3504		0,3484	0,0020
210,0000	0,2789		0,2751	0,0038
240,0000	0,2149		0,2123	0,0026
270,0000	0,1591		0,1587	0,0004
300,0000	0,1094		0,1131	-0,0038
330,0000	0,0707		0,0745	-0,0037
367,0000	0,0381		0,0347	0,0033

**Table A.12** Graph data for the proposed model for D-40%-210W

<b>Time</b>	<b>Moiture Ratio</b>	<b>Parameters</b>	<b>Predicted</b>	<b>Residuals</b>
0,0000	1,0000	0,9847	0,9847	0,0153
20,0000	0,9096	0,0006	0,9208	-0,0111
40,0000	0,8016	1,5035	0,8200	-0,0184
60,0000	0,6997	-0,0006	0,7053	-0,0057
90,0000	0,5510		0,5303	0,0208
105,0000	0,4794		0,4472	0,0322
120,0000	0,3665		0,3693	-0,0028
135,0000	0,2872		0,2973	-0,0101
150,0000	0,2086		0,2318	-0,0233
165,0000	0,1634		0,1729	-0,0095
180,0000	0,1152		0,1202	-0,0051
195,0000	0,0729		0,0736	-0,0008
205,0000	0,0510		0,0457	0,0053
212,0000	0,0406		0,0275	0,0131

**Table A.13** Data of rate curves for air drying at 30% ultrasound power

<b>D-30%-60°C</b>		<b>D-30%-70°C</b>		<b>D-30%-80°C</b>	
<b>Avarage Moisture Content</b>	<b>Rate</b>	<b>Avarage Moisture Content</b>	<b>Rate</b>	<b>Avarage Moisture Content</b>	<b>Rate</b>
6,3117	0,0115	6,9517	0,0250	6,7404	0,0363
5,6646	0,0101	6,2126	0,0243	5,6833	0,0342
5,0687	0,0098	5,5239	0,0216	4,6924	0,0319
4,4842	0,0097	4,9066	0,0195	3,7641	0,0300
3,9457	0,0083	4,3182	0,0197	2,9298	0,0256
3,4626	0,0078	3,7691	0,0169	2,2175	0,0218
2,9967	0,0077	3,2559	0,0173	1,5948	0,0197
2,5359	0,0077	2,7531	0,0162	1,0732	0,0151
2,0981	0,0069	2,2787	0,0154	0,6792	0,0112
1,7029	0,0062	1,8394	0,0139	0,4184	0,0062
1,3415	0,0058	1,4476	0,0122	0,2969	0,0043
1,0175	0,0050	1,1040	0,0107		
0,7503	0,0039	0,8284	0,0077		
0,5433	0,0030	0,6191	0,0062		
0,3978	0,0019	0,4029	0,0041		
0,3080	0,0011	0,2727	0,0022		

**Table A.14** Data of rate curves for air drying at 40% ultrasound power

<b>D-40%-60°C</b>		<b>D-40%-70°C</b>		<b>D-40%-80°C</b>	
<b>Avarage Moisture Content</b>	<b>Rate</b>	<b>Avarage Moisture Content</b>	<b>Rate</b>	<b>Avarage Moisture Content</b>	<b>Rate</b>
6,3623	0,0128	6,7297	0,0223	6,5430	0,0348
5,6586	0,0107	6,0580	0,0225	5,5020	0,0346
5,0489	0,0097	5,4173	0,0203	4,8252	0,0315
4,4903	0,0090	4,8276	0,0191	4,2156	0,0301
3,9609	0,0087	4,2767	0,0177	3,3476	0,0277
3,4629	0,0079	3,7559	0,0170	2,5747	0,0238
2,9920	0,0078	3,2563	0,0163	1,9021	0,0210
2,5355	0,0074	2,7674	0,0163	1,3253	0,0174
2,1005	0,0071	2,3188	0,0136	0,8670	0,0131
1,7109	0,0059	1,9025	0,0142	0,5207	0,0100
1,3470	0,0062	1,5037	0,0124	0,3070	0,0064
1,0015	0,0053	1,1428	0,0117		
0,7235	0,0040	0,8193	0,0099		
0,5201	0,0028	0,5516	0,0079		
0,3767	0,0020	0,3551	0,0043		
0,2878	0,0010				
0,2560	0,0005				

**Table A.15** Data of rate curves for hybrid drying at 30% ultrasound power

<b>D-30%-150W</b>		<b>D-30%-180W</b>		<b>D-30%-210W</b>	
<b>Avarage Moisture Content</b>	<b>Rate</b>	<b>Avarage Moisture Content</b>	<b>Rate</b>	<b>Avarage Moisture Content</b>	<b>Rate</b>
7,1086	0,0118	6,9838	0,0228	6,5702	0,0378
6,7642	0,0112	6,2920	0,0233	5,4470	0,0367
6,4223	0,0116	5,5824	0,0240	4,5446	0,0357
6,0912	0,0105	4,8558	0,0244	3,7726	0,0315
5,7612	0,0115	4,1689	0,0214	3,2529	0,0377
5,4245	0,0109	3,5290	0,0213	2,6969	0,0364
5,0842	0,0118	2,9170	0,0195	2,2153	0,0278
4,7560	0,0101	2,3633	0,0174	1,7881	0,0291
4,4391	0,0110	1,8602	0,0161	1,3697	0,0266
4,1154	0,0106	1,4210	0,0132	1,0143	0,0207
3,7994	0,0105	1,0403	0,0122	0,7174	0,0188
3,4950	0,0098	0,7128	0,0096	0,4730	0,0138
3,2104	0,0092	0,4711	0,0065	0,3263	0,0087
2,9407	0,0088	0,3161	0,0039		
2,6709	0,0092				
2,4079	0,0083				
2,1551	0,0085				
1,9028	0,0083				
1,6566	0,0081				
1,4283	0,0071				
1,2168	0,0070				
1,0221	0,0060				
0,8469	0,0057				
0,6935	0,0045				
0,5603	0,0043				
0,4452	0,0033				
0,3575	0,0025				

**Table A.16** Data of rate curves for hybrid drying at 40% ultrasound power

<b>D-40%-150W</b>		<b>D-40%-180W</b>		<b>D-40%-210W</b>	
<b>Avarage Moisture Content</b>		<b>Avarage Moisture Content</b>		<b>Avarage Moisture Content</b>	
<b>Rate</b>	<b>Content</b>	<b>Rate</b>	<b>Content</b>	<b>Rate</b>	<b>Content</b>
0,0144	7,0689	0,0267	6,6647	0,0319	6,7453
0,0120	6,6726	0,0288	5,8331	0,0382	6,0444
0,0122	6,3101	0,0295	4,9586	0,0360	5,3028
0,0107	5,9672	0,0267	4,1156	0,0350	4,4178
0,0109	5,6430	0,0224	3,3793	0,0337	3,6396
0,0108	5,3180	0,0189	2,7595	0,0532	2,9878
0,0105	4,9989	0,0169	2,2229	0,0373	2,3090
0,0101	4,6905	0,0151	1,7440	0,0370	1,7512
0,0104	4,3838	0,0131	1,3211	0,0213	1,3139
0,0102	4,0750	0,0117	0,9484	0,0227	0,9839
0,0105	3,7645	0,0091	0,6362	0,0199	0,6642
0,0095	3,4641	0,0062	0,3843	0,0154	0,4376
0,0094	3,1800			0,0105	0,3237
0,0090	2,9033				
0,0091	2,6316				
0,0083	2,3714				
0,0084	2,1218				
0,0078	1,8788				
0,0072	1,6525				
0,0067	1,4433				
0,0067	1,2429				
0,0059	1,0549				
0,0058	0,8799				
0,0052	0,7145				
0,0057	0,5507				
0,0039	0,4077				
0,0023	0,3122				

**Table A.17** TPC values for air drying at 30% ultrasound power

<b>Fresh</b>	<b>D-30%-60°C</b>	<b>D-30%-70°C</b>	<b>D-30%-80°C</b>
3482,39	1809,89	1605,57	1906,49
3456,95	1800,73	1628,55	1898,20
3459,94	1806,95	1672,24	1891,79

**Table A.18** TPC values for air drying at 40% ultrasound power

<b>Fresh</b>	<b>D-40%-60°C</b>	<b>D-40%-70°C</b>	<b>D-40%-80°C</b>
3482,39	1801,61	1551,55	1756,14
3456,95	1814,02	1532,81	1753,74
3459,94	1759,24	1527,35	1734,60

**Table A.19** TPC values for hybrid drying at 30% ultrasound power

<b>Fresh</b>	<b>D-30%-150W</b>	<b>D-30%-180W</b>	<b>D-30%-210W</b>
3482,39	1957,77	2395,33	1951,86
3456,95	1945,29	2328,66	1926,77
3459,94	1951,34	2317,62	1930,30

**Table A.20** TPC values for hybrid drying at 40% ultrasound power

<b>Fresh</b>	<b>D-40%-150W</b>	<b>D-40%-180W</b>	<b>D-40%-210W</b>
3482,39	1864,64	2103,19	1844,86
3456,95	1876,27	2106,65	1864,11
3459,94	1797,29	2137,46	1789,08

**Table A.21** AA values for air drying at 30% ultrasound power

<b>Fresh</b>	<b>D-30%-60°C</b>	<b>D-30%-70°C</b>	<b>D-30%-80°C</b>
286,58	143,94	130,96	137,10
297,06	143,24	130,60	138,87
282,65	147,12	128,79	138,16

**Table A.22** AA values for air drying at 40% ultrasound power

<b>Fresh</b>	<b>D-40%-60°C</b>	<b>D-40%-70°C</b>	<b>D-40%-80°C</b>
286,58	130,48	118,32	129,60
297,06	134,70	122,03	129,98
282,65	131,18	127,23	134,91

**Table A.23** AA values for hybrid drying at 30% ultrasound power

<b>Fresh</b>	<b>D-30%-150W</b>	<b>D-30%-180W</b>	<b>D-30%-210W</b>
286,58	161,02	172,77	156,31
297,06	158,83	173,50	155,56
282,65	157,37	172,40	152,16

**Table A.24** AA values for hybrid drying at 40% ultrasound power

<b>Fresh</b>	<b>D-40%-150W</b>	<b>D-40%-180W</b>	<b>D-40%-210W</b>
286,58	148,25	165,74	145,65
297,06	142,07	165,00	139,60
282,65	144,78	167,97	141,11

**Table A.25** TPC values for hybrid drying for varying pretreatments

<b>210W</b>	<b>D-210W</b>	<b>D-40%(Water)-210W</b>	<b>D-40%-210W</b>
1991,89	1989,44	1725,94	1844,86
1970,06	1963,54	1717,85	1864,11
1990,72	2009,55	1753,66	1789,08

**Table A.26** TPC values for air drying for varying pretreatments

<b>80°C</b>	<b>D-80°C</b>	<b>D-40%(Water)-80°C</b>	<b>D-40%-80°C</b>
1820,73	1859,11	1610,18	1756,14
1784,66	1887,86	1584,76	1753,74
1793,13	1879,04	1660,62	1734,60

**Table A.27** AA values for hybrid drying for varying pretreatments

<b>210W</b>	<b>D-210W</b>	<b>D-40%(Water)-210W</b>	<b>D-40%-210W</b>
158,66	155,90	133,49	145,65
157,91	153,31	132,76	139,60
160,15	154,42	134,97	141,11

**Table A.28** AA values for air drying for varying pretreatments

<b>80°C</b>	<b>D-80°C</b>	<b>D-40%(Water)-80°C</b>	<b>D-40%-80°C</b>
134,98	143,21	122,09	129,60
137,10	142,11	123,54	129,98
138,15	140,28	121,72	134,91