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**M.Sc. in Food Engineering**

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**REPUBLIC OF TURKEY  
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**DRYING BEHAVIOR OF OLIVE POMACE WITH HOT AIR  
ASSISTED-RADIOFREQUENCY DRYING AND ITS  
ASSOCIATED EFFECT ON OLIVE POMACE OIL QUALITY**

**M.Sc. THESIS  
IN  
FOOD ENGINEERING**

**BY  
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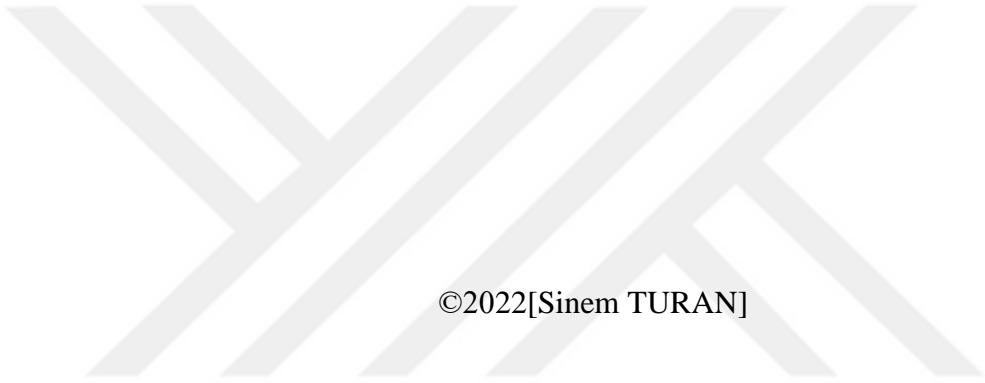
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**May 2022**



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RADIOFREQUENCY DRYING AND ITS ASSOCIATED EFFECT ON  
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**I hereby declare that all information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by these rules and conduct, I have fully cited and referenced all material and results that are not original to this work.**

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## **ABSTRACT**

### **DRYING BEHAVIOR OF OLIVE POMACE WITH HOT AIR ASSISTED- RADIOFREQUENCY DRYING AND ITS ASSOCIATED EFFECT ON OLIVE POMACE OIL QUALITY**

**TURAN, Sinem**

**M.Sc. in Food Engineering**

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In this study, it was aimed to evaluate the drying behavior of olive pomace during drying in a hot air assisted radiofrequency system and also to evaluate the final oil quality obtained from this dried pomace. The effect of sample thickness, radiofrequency electrode gap and the compression of sample (sample porosity) on drying behavior was determined. The most efficient drying took place in 60 minutes with a 1000 kg sample at 105 mm electrode gap and 10 cm sample thickness. The quality parameters of the oil obtained at these optimal conditions was determined. The free fatty acid content, peroxide value, p-anisidine value, PAH content (BaP), L\*,a\*, b\*, chlorophyll and carotenoid content of the oil were measured as 1.09 %, 12.2 meq O<sub>2</sub>/kg oil, 3.01,<1 ppb, 38.6, 7.5, 62.56, 105.25 mg pheophytin A /kg oil and 2.85 mg /kg oil, respectively.

**Key Words:** Olive Pomace, Radiofrequency Drying, Olive Pomace Oil, Drying Kinetics, Drying

## ÖZET

### SICAK HAVA DESTEKLİ RADYOFREKANS KURUTMA İLE PİRİNANIN KURUMA DAVRANIŞI VE PİRİNA YAĞI KALİTESİNE ETKİSİ

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**52 sayfa**

Bu çalışmada, pirinanın sıcak hava destekli radyofrekans sisteminde kurutma sırasındaki kuruma davranışının ve ayrıca bu kurutulmuş pirinadan elde edilen nihai yağ kalitesinin değerlendirilmesi amaçlanmıştır. Numune kalınlığının, radyo frekans elektrot mesafesinin ve numunenin sıkıştırılmasının (numune gözenekliliği) kuruma davranışı üzerindeki etkisi belirlendi. En verimli kurutma 60 dakika içerisinde 1 kg numune, 10 cm numune kalınlığı ve 105 mm elektrot mesafesi ile gerçekleştirilmiştir. Bu optimum koşullarda elde edilen yağın kalite parametreleri belirlenmiştir. Yağın peroksit değeri, serbest yağ asidi içeriği, p-anisidin değeri, PAH içeriği (BaP), L\*,a\*, b\*, klorofil ve karotenoid içeriği sırasıyla % 1.09, 12.2 meq O<sub>2</sub>/kg yağ, 3.01,<1 ppb, 38.6, 7.5, 62.56, 105.25 mg feofitin A/kg yağ ve 2.85 mg/kg yağ olarak ölçülmüştür.

**Anahtar Kelimeler:** Pirina, Radyo Frekans ile Kurutma, Pirina Yağı, Kurutma Kinetiği, Kurutma.



*“Dedicated to my family”*

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## LIST OF SYMBOLS

$^{\circ}\text{C}$	Degree of Celsius
%	Percentage
$L^*$	Lightness
$A^*$	Redness
$B^*$	Yellowness
$\epsilon''$	Dielectric Loss Factor
$\epsilon'$	Dielectric Constant
$\epsilon$	Relative Complex Permittivity
$\rho$	Specific Gravity
$E$	Electricity Field Strength
$\Delta t$	Time Change
$f$	Frequency
$\Delta T$	Temperature Change
$C_p$	Specific Heat
$Z_c$	Material Capacitance
$C_0$	Gap Capacitance
$\dot{U}$	Heat Production Rate
$\epsilon_0$	Free Space Permeability
$MR$	Moisture Ratio
$M$	Moisture Content
$DR$	Drying Rate
$Deff$	Effective Moisture Diffusion Coefficient
$L$	Half Thickness of Sample

## LIST OF ABBREVIATIONS

<b>RF</b>	Radiofrequency
<b>PAH</b>	Polycyclic Aromatic Hydrocarbons
<b>BaP</b>	Benzo[a]pyrene
<b>mg</b>	milligram
<b>ppb</b>	Parts per billion
<b>mm</b>	millimeter
<b>kg</b>	kilogram
<b>meq</b>	Fault Detection Diagnosis
<b>MW</b>	Microwave
<b>Eq.</b>	Equation
<b>h</b>	hour
<b>ppm</b>	Parts per million
<b>MHz</b>	Megahertz
<b>HA-RF</b>	Hot Air Assisted Radiofrequency
<b>TBA</b>	Thiobarbituric Acid
<b>cm</b>	centimeter
<b>nm</b>	Nanometer
<b>V</b>	Volt
<b>g</b>	gram
<b>FFA</b>	Free Fatty Acid
<b>PV</b>	Peroxide Value
<b>p-AnV</b>	p-Anisidine Value
<b>IOC</b>	International Olympic Committee
<b>AOCS</b>	American Oil Chemists' Society
<b>UV</b>	UltraViolet
<b>HPLC</b>	High Pressure Liquid Chromatography
<b>GC</b>	Gas Chromatography

## **CHAPTER I**

### **INTRODUCTION**

Olive pomace is a by product generated during the olive oil extraction process. It consists of olive pulp and kernel. Olive pomace is a commercial commodity and its price determined according to its oil containment. Traditionally it is used in animal feeding and biomass fuel industry. The crude olive pomace oil is particularly used in soap making. If it is refined, refined olive pomace oil can be consumed (Özsayın, Karaman, Karahan, 2015).

The high consumption of olive oil leads to increase in the amount of olive pomace. The fatty acid composition of the pomace oil is similar to refined olive oil but there are differences in minor components between them. Hence refined olive pomace oil gain great interest of consumer. Because the nutritional values are high, olive pomace oil has become of great interest (Rodríguez-Gutiérrez, et al., 2012).

Pomace contains moisture. Due to the high moisture content of olive pomace, it is essential to dry the pomace before oil extraction. Drying process effects quality parameters of olive pomace oil like color, composition or PAH level of the oil (Kiralan, Erdoğan, Tekin, 2016). Also it is important that the drying process is carried out in a short time without the degradation of the fatty acids contained in pomace. Therefore, the proper drying process must be carried out. Due to the increasing interest in olive pomace oil today, studies are directed to investigate new and efficient pomace drying methods which does not cause adverse affect on the quality of olive pomace oil (Aktaş, Karabeyoğlu, Akyıldız, 2015).

The radiofrequency (RF) drying is one of the new drying methods in food processing. In RF heating, which is one of the direct heating methods, heat is formed inside the material and moves out of the material. The amount of heat depends on the physical and chemical properties of the material and the applied frequency. The advantage of the RF method of drying is that the water is highly amenable to direct heating methods

and is quickly removed of from a substance. Since this method activates only water molecules, it saves energy and the heat spreads evenly to all parts of the material (Tunçer, 2006). Also, the RF method saves time and energy and it can be applied for thick materials. The RF technique provides high speed, high quality drying and convenience (Nijhuis et al. 1998). RF method provides easier control on drying (Nelson, Datta, 2001). RF helps drying uniformly and this increases quality of the product (Popovici, Popovici, 2009).

The main purpose of this thesis is to investigate the drying behavior of olive pomace in RF drying and evaluate the effect of the RF drying on the quality of olive pomace oil. In this context, the effect of sample thickness, RF electrode gap and the compression of sample (sample porosity) on drying behavior was determined. Oil quality parameters were analyzed such as; color, free fatty acid value, peroxide value, p-anisidine value, fatty acid composition, (PAHs)- Benzo (a) pyrene content, K270-K232 values, chlorophyll and carotenoid content of the oil. Obtained results were compared with values in literature.

## **CHAPTER II**

### **LITERATURE REVIEW**

#### **2.1 Olive Oil Production**

Olives are grown for consumption in olive oil production and factories. There are many factors that affect the quality of olive oil. These factors could be the climate, soil conditions and harvest time (Doymaz, Görel, Akgün, 2004). The vast majority of olive trees are found in the Mediterranean basin due to its climate conducive to olive cultivation (Lammi, et al., 2018).



**Figure 2.1 Olive Fruit**

People are turning to olive oil consumption because of the health benefits. This situation increases the consumption of olive oil and therefore its production (Nunes, et al., 2018).

The olive oil sector is important for waste studies because huge amount of waste is generating during the olive oil extraction from olive fruit. About twenty percent of the olive fruit used is obtained as olive oil, while the remaining part is released as waste (Kapellakis, et al., 2008). Moist olive pomace and/or olive mill water are released as by product in the production of olive oil (Albahari, et al., 2018). However, the amount and the composition of the olive oil processing waste changes with the extraction technique (pressing method, two-phase decantation, or three-phase decantation

system) applied. The waste part containing the seeds, skin and pulp released during olive oil production is called ‘olive pomace’ (Nunes, et al., 2018).

The main steps in industrial olive oil production line as follows;

\*Feeding and washing; In order to start the process of producing oil from the olive fruit, the olives are placed in the large feeding unit. Here, olive leaves and foreign materials should be cleaned and washed. This process is important because the factors that will adversely affect the quality of the oil must be removed at this step (Kapellakis, et al., 2008).

\*Crushing; It is aimed to facilitate the removal of the oil inside the fleshy part of the fruit by crushing in this step. The crushing process takes place with the wheels inside the unit (Kapellakis, et al., 2008).

\*Malaxation; the viscous product formed after crushing is mixed. This process takes about 20-30 minutes. It allows the oil to emerge. It is aimed to form large drops by combining the oil drops (Kapellakis, et al., 2008). The malaxation process helps to the enzymes in the olives to create aroma by mixing. It helps to separate the different densities of malax in the decanters with the centrifugal force. Studies show that malaxation is one of the most important steps for olive oil production. Because the enzyme activity of the oil affected by malaxation time, temperature and air contacting the dough (Gülal, et al., 2015).

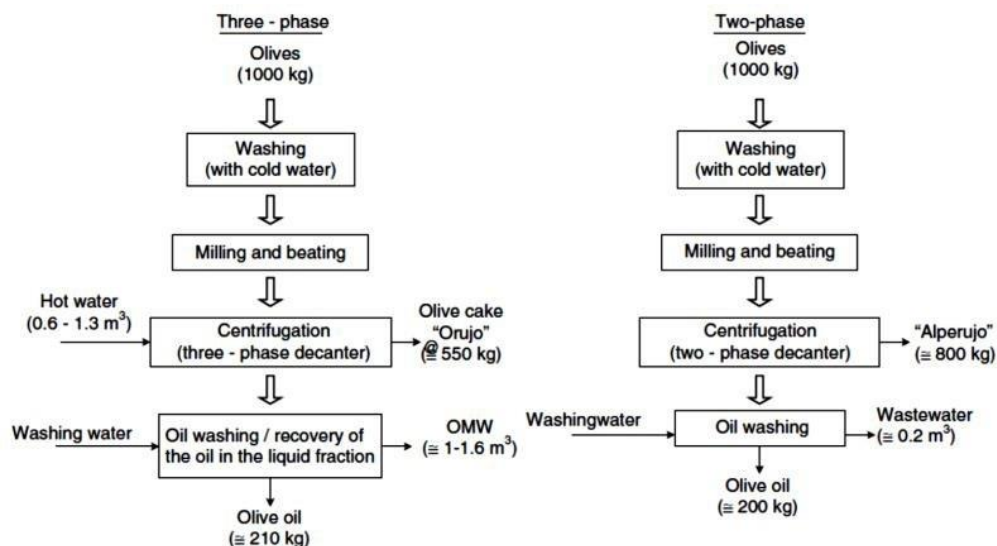
\* Separation of oil; the resulting structure contains olive residues and water. The oil is separated from the residues by applying the appropriate oil extraction method (Kapellakis, et al., 2008).

\* Centrifugation of oil; The process is carried out with a horizontal decanter for taking advantage of the density difference of olive residues (Kapellakis, et al., 2008). Centrifugation with decanters is called a continuous system (Gülal, et al., 2015). The oils that are combined in the crushing and mixing processes are separated by centrifugation and the small drops of oil are released by adding water. The speed and capacity of the decanter affect the performance and the amount of separated oil. Also, amount of water added to the dough effects the amount of oil. Centrifugation is an efficient method because does not require much work. Also, it reduces the risk of contamination because it is made with stainless steel. It does not have a diaphragm and

can collect most of the oil. Considering the negative aspects of the centrifugation process; requires water and energy. Important components are lost by centrifugation. Olive pomace comes out with high humidity (Kapellakis, et al., 2008).

Olive oil was obtained by pressing method in ancient times. However, this process is not very common today because it requires a lot of labor (Moral, Méndez, 2006). Decanters can be used as two or three phase systems (Kapellakis, et al., 2008). Pomace has a humidity of 45% and vegetable water with a high pollution rate is released in the three-stage method. The vegetable water can cause environmental problems and cause extra cost of water removal. That is why most manufacturers have switched to the two-stage method (León-Camacho, et al., 2003). The humidity of the product should be around 50% minimum in the two-phase system (Moya, et al., 2018).

The property of the oil varies depending on the time during the storage period up to the time of production because of high humidity in moist pomace in the two-phase method. Moist pomace also contains alkalis and polyphenols due to the plant water (Moral, Méndez, 2006). Two-phase decanter continuous system is more effective due to amount of product, impact on the environment, labor requirement, high amount of phenol content (Gülal, et al., 2015).



**Figure 2.2** Three-and two-phase centrifugation systems for olive oil extraction (Tsagaraki et al., 2007)

**Table 1.** Comparison of olive oil obtained by two- and three-phase decanter system on quality characteristics (Gülal, et al., 2015)

	<b>Two-Phase Centrifugation System</b>	<b>Three-Phase Centrifugation System</b>
Acidity(%)	0.35	0.34
Peroxide Value (meq/kg)	3.8	4.3
Total Polyphenols (mg/ l Gallic Acid)	333	220
Oxidation Stability (h)	15.3	11.6
Chlorophyll Pigments (ppm)	6.3	6.6
K232	1.548	1.438
K270	0.105	0.091
Sensory Evaluation	7.1	7.2

## 2.2 Olive Pomace

Olive pomace consists of pulp, stone, skin and seed pieces. Lammi, et al. (2018), has been stated that 2,881,500 tons of pomace was released in a year. It causes environmental problems where it is located. It can cause microbiological growth. Therefore, it affects the physiological and biological properties of the soil. It is evaluated in many sectors such as animal feed, fuel, energy and cosmetics in order to minimize the damage it may cause to the environment.

Pomace can exhibit different properties depending on the variety of fruit, its cultivation and soil (Lammi, et al., 2018). Olive pomace represents 80% of the raw material at the stage of olive oil production (Moya, et al., 2018).



**Figure 2.3** Olive Pomace

Olive pomace is an important surplus of olive oil production. Pomace consists of phenolic and bioactive components. Attention should be paid to the studies that can use these components in the most efficient way. Therefore, studies will help contribution to waste assessment and sustainable agriculture. Also, olive pomace poses added value economically (Nunes, et al., 2018).

Olive pomace is a valuable by-product due to its high organic matter, oil content and high calorific value (Sümer, et al., 2016). Pomace, which can contain up to eight percent oil, contains triacylglycerols fatty acids, triterpenic acids and squalene (Ketenöglü, et al., 2018). Pomace contains valuable components which are beneficial to health due to the high polyphenolic content. They also have a negative impact on the environment. Along with the correct evaluation of pomace waste, it can be evaluated by numerous applications in many sectors such as food, feed, cosmetics sectors, etc. Given all this, it should be ensured that the environmental damage of olive pomace is eliminated in the right way and evaluated as beneficial.

Useful components will be obtained with the development of non-toxic procedures by methods based on the principles of ecology and sustainability. In order to achieve this, it is necessary to new applications such as microwave, high temperature, ultrasound, taking into account parameters such as increasing efficiency and reducing the amount of solvent (Albahari, et al, 2018).

Olive pomace can cause serious environmental problems due to its high biochemical oxygen demand. Storage and stacking processes are more difficult because of high humidity in the pomace obtained by the two-phase method. Moist pomace is kept in large ponds. Drying process of the pomace is difficult because of the high humidity and the sugar content (Arjona, Garcia, Ollero, 1999). Pomace can have toxic properties and cause environmental problems due to the high content of organic components, phenolic compounds (Rodríguez-Gutiérrez, et al., 2012).

Although pomace is considered a serious source of pollution, it is also an important source. For instance, it contains bioactive components useful for health. For this reason, new methods are needed in the evaluation of pomace. Valuable bioactive components will be recovered and the use of these wastes will be an important step in terms of economic and recovery (Rodríguez-Gutiérrez, et al., 2012).

### **2.3 Drying Process**

Drying is a method used to extend the shelf life of the product and to prevent microbiological growth. It aims to reduce the amount of moisture in the product. The drying process is important in the food industry and is a factor that directly affects product quality. However, energy consumption is high during the drying process. Increasing energy efficiency in drying technologies will reduce its adverse effects on the environment.

Among the drying methods, the hot air drying method is one of the most widely used methods in agricultural food products. However, the hot air drying process takes time and has low energy efficiency. At the same time, it negatively affects the quality of the product by exposing to hot air for a long time. In addition, the drying rate slows down at low temperature which not only prolongs the drying time, but also negatively affects product quality (Shewale, et al., 2021).

On the other hand, high temperatures reduce the product quality and may cause the product to burn. For this reason, temperatures between 45°C and 60°C are applied in chamber and barrel dryers and equilibrium temperatures of 70°C in continuous flow dryers. The drying process depends on the temperature of the air, airflow rate and relative humidity (Meziane, 2011). As the air temperature rises, the air carries more

moisture and thus the relative humidity decreases. At the same time, as the airflow rate increases, the drying rate also increases (Çetiner, 2017).

Even though the drying method has been used for many years for food preservation, it is a difficult process. Keeping product quality is a critical issue for a drying process. The negative aspects of conventional air drying may be high temperatures, long drying times, deterioration in product quality and decrease in nutritional values. The heat has difficulty in transferring towards the interior of the food due to the low thermal electrical properties of food materials (Maskan, 2000).

### **2.3.1 Drying Methods**

The product is effective in determining the best drying type. The drying process can be successful with the selection of proper equipment and the optimization of the process conditions. Recycling, waste and product quality are important performance criteria of drying (Kimball, 2001).

#### ***Convection;***

In the drying process, hot air surrounds the food and heat is transferred from the surface to the center of food. At first, the moisture in the food moves towards its surface and evaporation begins. First the surface moisture increase up to the critical humidity level and after this point the food surface starts to dry. The process of removing water by transporting heated air is called ‘‘convective drying’’. Hot air passes through the food and removes water. Cabin dryers, tunnel dryers, fluid bed dryers and spray dryers are commonly used convective drying equipments. Air temperature and flow rate are important in convection drying (Kılıç, Çınar, 2019).

#### ***Conduction;***

The heat required to evaporate the moisture in the food product is carried by conduction in the contact drying method. The product is stationary or in motion and the heat is transferred from the surface to the material by contacting the hot surface during drying.

### ***Solar Drying;***

In solar drying system principle is converting solar energy to thermal energy by using solar collector unit (Desa, et al., 2020). The biggest advantage of drying with the sun is being environmentally friendly. It continues to be widely used because it is an inexpensive method. However, undesirable conditions in the quality of the final product are its disadvantages (Borah, et al., 2015). Other disadvantages of the sun drying process are drying times, difficult process control, changes in weather conditions, workload, area, insects, foreign matter and pollution (Nasıroğlu, Kocabıyık, 2009).

In infrared drying, the energy of radiation is turn into heat by vibration of water molecules and penetrates the material (İsmail, Kocabay, 2018). Infrared drying technology is a good drying method as it reduces the drying time and provides energy efficiency. In addition, the availability, simplicity and cheapness of the equipment in the infrared drying method increase its preferability (Nasıroğlu, Kocabıyık, 2009).

### ***Dielectric;***

RF and microwave (MW) drying are dielectric drying techniques. In RF heating, a frequency of 10-300 MHz is applied while a frequency of 300-30,000 MHz is applied in MW. Dielectric heating is related to the dissipation of electromagnetic energy.

Since the 1950s, studies on the MW technique have been carried out. Studies on the method of heating with RF began in 1940. High quality food products are dried without deteriorating the quality of the food while drying times are short by dielectric drying methods. While the use of MW has spread to homes, the RF method has not been widespread. Some parameters such as the interaction of the product from electromagnetic waves, electromagnetic field distribution and dielectric properties of food affect the use of dielectric methods in drying of foods (Wang, et al., 2011).

### ***Combined;***

The objectives of combined drying methods are to obtain more effective and efficient drying by reducing the deterioration or negative effects of drying process on product. For example; when a method with a long drying time is combined with another method, the drying time can be shortened. The negative effects of drying on the

product can be minimized. In the combined drying method, it is necessary to know the characteristics of the food product to be dried. Thus, the right methods can be combined. Hot air-MW finish drying, hot air-radiofrequency (HA-RF) drying are some of the combined drying methods which are studied in laboratory or pilot scale.

### **2.3.2 Olive Pomace Drying**

In the olive pomace oil extraction process the first and indispensable stage is the drying of olive pomace. One of the most common methods for olive pomace drying is countercurrent rotary dryers. These dryers are known for their versatility and providing great input-output. The biggest problems in conventional drying are excessive use of energy, low efficiency and deterioration of the quality of the final product. Also, high temperature causes decreasing of the quality of pomace oil. For this reason, studies of novel methods have started for olive pomace drying. Efforts are being made to solve these problems (Jumah, et al., 2007). Humid pomace is dried until the moisture content is between 5-10%. A lot of energy is consumed in this drying process (Rodríguez-Gutiérrez, et al., 2012).

Common application for rotary drying in the olive pomace industry is the use of high temperatures up to 400 to 800 °C. Moreover, as a general application olive pomace is used as a fuel in this drying process and the pomace to be dried directly contact with the exhaust gas from combustion of olive pomace (Moral, Méndez, 2006).

Olive pomace oil extracted after this traditional drying method, is dark in color and have unpleasant odour due to deteriorative reactions caused by high temperature and exhaust gas exposure. Moreover, there is an increase in the K232 value and oxidized compounds as well as formation of PAHs (Moral, Méndez, 2006).

### **2.4 RF Drying System**

Studies on the use of the RF method in the food industry were initiated in the 1940s. It started with processes such as drying vegetables, cooking meat and heating. Afterwards, researches on thawing frozen products were carried out and thus the studies continued for years (Wang, et al., 2011).

RF heating is an alternative heating technique that can replace traditional heating methods. It is a new method with positive aspects such as deeper heat penetration and homogeneous heat distribution.

Waves are produced between two electrodes fed by high voltage alternating current in the RF system. The electrical energy is trapped by the food products as internal energy, thus it is converted into heat (Yazar, İçier, 2013).

A dielectric is formed between the metal capacitors charged with opposite polarity charges and the product in the RF system. An alternating electric field is formed between the electrodes. The frequency value charges the electrodes with current from positive to negative. In this event, the polar molecules in the food material show a continuous arrangement and heat is released in the food (Yazar, İçier, 2013).

Debye Resonance is the name given to the frequency after the frequency increase reaches a point. The delay given by the molecules and ions in the food to this new electric field and the delay time is called the relaxation time (Yazar, İçier, 2013).

Dielectric properties of foods are important in the RF system. Dielectric properties are affected by parameters such as frequency, density, temperature, moisture content or structure of the food and storage time. In addition, the rotation of the dipole and the electrical resistance resulting from the movement of insoluble ions provide the combination of heating because foods are dielectric products (Yazar, İçier, 2013).

RF heating is an effective application for various purposes. For example, it can be recommended for pasteurization/sterilization, pest control and enzyme inactivation. RF heating is seen as a novel method for drying of fresh fruit/vegetables but homogeneity is still difficult to achieve in products with high humidity.

Based on the studies, it is predicted that when the RF heating method and hot air drying methods are combined, improvements in the drying process and product quality could be seen. Combinations of RF drying together with the correct process sequence will provide more effective results (Zhou, et al., 2019).

Non-uniform temperature distribution may cause different parts of the food product to remain moist or cause quality loss due to uneven heating. This may cause insect or microbial contamination of food products. Understanding of RF technology is

important to prove the applicability. So, some studies and simulations have been made in order to better understand RF technology. These studies, together with RF, provided an understanding of the dielectric properties of the food product, its uniform heating property (Tiwari, et al., 2011). RF devices are good at drying moist solids with poor thermal conductivity and better in control of product humidity (Dziak, 2008).

According to many studies, it has been determined that drying with RF or MW is applied in a shorter time compared to traditional drying methods. Also, the energy efficiency is higher. At the same time, process control is more successful and the desired operation can be performed in less space.

The most important property for RF or MW heating is the dielectric permittivity of the materials. The humidity, temperature and electric field frequencies are important factors for dielectric drying methods (Guo, Zhu, 2014).

#### **2.4.1. Dielectric Properties**

Frequency values are important in heating with RF. Heat conduction in foods is a serious loss factor. The dielectric loss factor is called the dipole rotation and ionic motion in the product and represents the symbol  $\varepsilon''$ .

In the equation given below (1.1.);  $d$  denotes dipole rotation and  $\sigma$  denotes ion polarization. In RF frequency values, as the temperature increases, the ionic loss increases.

$$\varepsilon'' = \varepsilon''_d + \varepsilon''_{\sigma} \quad (1.1)$$

Since food products show dielectric properties, they are intended to absorb energy. They can also dissipate electrical energy.

These properties represent dielectric properties and are expressed by this equation (1.2). It is represented by the relative complex permittivity ( $\varepsilon$ ). The permeability value expresses the dielectric properties that affect the distribution of wave energy in the food with the reflection of electromagnetic waves.

$$\varepsilon = \varepsilon' - j\varepsilon'' \quad (1.2)$$

$$j: \sqrt{-1}$$

The dielectric constant ( $\epsilon'$ ) is the electrical energy storage property of a product. The loss factor ( $\epsilon''$ ) indicates the ability of the food to convert electrical energy into heat. These two properties represent the product's temperature profile, penetration depth, power absorption and similar dielectric properties in the RF system.

Temperature increase of food products is calculated from 1.3.

$$\rho C_p \frac{\Delta T}{\Delta t} = 5.563 \times 10^{-11} f E^2 \epsilon'' \quad (1.3)$$

$\rho$ : specific gravity of the product (kg/m<sup>3</sup>),  $C_p$ : specific heat of the product (Jkg<sup>-1</sup>°C<sup>-1</sup>),  $\Delta t$ : time (s),  $\Delta T$ : temperature rise in the product (°C),  $E$ : electricity field strength (Vm<sup>-1</sup>) and  $f$ : frequency (Hz) symbols are expressed in the equation.

In order to calculate the capacitance value for the food products placed between the electrodes, 1.4 was used.

$$Z_c = \frac{1}{2\pi f C_0} \frac{\epsilon'' - j\epsilon'}{\epsilon''^2 + \epsilon'^2} \quad (1.4)$$

$Z_c$ : Material capacitance ( $\Omega$ ),  $f$ : frequency (Hz),  $\epsilon'$ : dielectric coefficient (absorption level) (F/m),  $\epsilon''$ : dielectric loss factor (F/m),  $C_0$ : gap capacitance (8, 85x10<sup>-12</sup> F/m) and  $j$ :  $\sqrt{-1}$  symbols are expressed in the equation.

The heat production rate ( $\dot{U}$ ) in a food product is expressed by 1.5.

$$\dot{U} = 2\pi f \epsilon_0 \epsilon'' |\Delta V|^2 \quad (1.5)$$

$|\Delta V|^2$ : electric field strength,  $f$ : frequency (Hz),  $\epsilon_0$ : free space permeability (8.854 x 10<sup>-12</sup> Farad/m) and  $\epsilon''$ : dielectric loss constant (F/m) symbols are expressed in the equation.

The RF method is used in the food industry in the fields of drying, cooking, pasteurization, disinfection, freezing and thawing.

The disadvantages of the RF method for now;

- \* Lack of experience and knowledge about the method due to its new use
- \* Installation with high cost

\* Not knowing the details of dielectric properties for all food products (Yazar, İçier, 2013).

Some advantages for the RF system are;

\*Applicability to large sized foods,

\*The product directly heats the product by ignoring the surrounding factors due to being a superior heating method.

\*Short drying time

\*Providing higher quality in the final product

\*Fast and homogeneous heat distribution (Çakmak, Tavman, 2011).

## **2.5 Olive Pomace Oil**

Olive pomace oil is one of the important commercial commodities. Olive oil can be obtained by three different methods namely pressing, two-phase decantation or three-phase decantation. Hence the properties of olive pomace generating from these systems are different in content and composition.

Oil, residual water and pomace are obtained by the three-phase method, while oil and solid waste (i.e. pomace with high humidity) are obtained by the two-phase method. Hence, high-humidity pomace is released in two-phase production while less-humidity pomace and waste water is obtained in the three-phase method. Therefore it requires differences in storage and drying stages of these olive pomaces.

After drying process of pomace, extraction step will take place. There are two different extraction methods to obtain crude olive pomace oil. The first is to obtain oil from dried pomace with the help of a solvent by the traditional method (Moral, Méndez, 2006).

Generally, n-hexane is used as a solvent. It is a strong degreaser and has no effect on oil quality during extraction. With its low boiling temperature, it is evaporated without damaging oil (60°C) (Moral, Méndez, 2006).

Another method is called the second centrifuge, with the help of horizontal centrifugal machines or decanters. Oil is obtained by centrifuging pomace obtained by the two-

phase method for the second time. The oil is obtained by both of these methods are called “crude olive pomace oil”. The properties of the oil will change in proportion to the storage time and distortions will occur in such important parameters as free acidity, the formation of methyl and ethyl esters (Moral, Méndez, 2006). In order to obtain edible olive pomace oil, after it is refined, it is mixed with virgin olive oil and ready-to-eat olive pomace oil. Except for polar phenols, bioactive components are more abundant in olive pomace oil than virgin olive oil (Rodríguez-Gutiérrez, et al., 2012).

The popularity of olive pomace oil is increasing because it is cheaper and has bioactive components. Olive pomace oil contains functional compounds. Minor components such as phytosterols, tocopherols, aliphatic alcohols, squalene and triterpenic acid in its content are some of the known disease prevention agents. These components are known to be good for Alzheimer's disease, cancer, cholesterol and some tumoral diseases (Rodríguez-Gutiérrez, et al., 2012).

Pomace oil has important bioactive components and lipophilic compounds with high amounts of triterpennic acids. Phenolic compounds can be used as food antioxidants. There are high levels of phenolic compounds in olive pomace. After the olive pomace oil is taken, pomace can be used as fuel material, feed or fertilizer. Pomace is stored in large ponds and oil can be produced every season of the year, but if it is kept too long, it will cause undesirable spoilage (Moya, et al., 2018).

Pomace oil can be used in the cosmetics and soap industry as well as used in kitchen tables after refining (Sümer, et al., 2016). Refining is essential because drying process applied prior to extraction can cause high amounts of unwanted products such as polycyclic aromatic hydrocarbons (PAHs). These harmful components are eliminated in refining process (León-Camacho, et al., 2003).

## **2.6 Oil Quality Parameters**

Quality is the achievement of the desired parameters in the desired conditions, in terms of time and money. Throughout the world, oil quality is given more importance than the amount of production.

All factors affecting the quality of the oil must be improved from the soil to the moment it is presented to the consumer. Along with new technologies, alternative methods should be found to improve the quality of olive pomace oil.

### **2.6.1 Oxidative Stability of Oil**

The oxidative stability of oils defines the shelf life and performance criteria of the oil. We can call oxidation resistance. Oxidation stability depends on the composition of the product and environmental conditions. When examining the oxidation of the oil, it is necessary to examine the conditions such as whether it is exposed to air or oxygen flow, heated at a constant temperature, exposure to light. These conditions are called oxidative conditions (Guillén, Cabo, 2002).

Unsaturated vegetable oils are more reactive to react with chemicals or oxygen. Environmental factors such as heat, light, humidity, metals increase the tendency to chemical reaction. When the oil interacts with oxygen, it reacts and spoils such as bad taste, decrease in nutritional values (Naz, et al., 2004). Oxidative stability of oils is determined by parameters such as peroxide number, thiobarbituric acid (TBA), conjugated diene and trienes, anisidine and hexanal values. While the peroxide number of these determines the primary oxidation products of oils; TBA, conjugated diene and trienes, anisidine and hexanal values indicate secondary products of oxidation (Can, 2019).

### **2.6.2 Free Fatty Acid**

Free fatty acid (FFA) content of the oils represents the amount of FFA (unbounded to a triacylglycerol structure). FFA is formed by hydrolysis of oil from triglyceride structure. It is an important quality parameter for crude oils as well as for refined oils. Because it shows the direction about how much caustic should be used in the neutralization process in crude oil (Yağlarda Serbest Asitlik Tayini, n.d). It is important to remove the FFA from the oil (Özdoğan, Tunalıoğlu, 2017).

### **2.6.3 Peroxide Value**

The peroxide value (PV) represents the amount of active oxygen in the oils. Oils tend to deteriorate with factors such as oxygen, metal ions, heat and temperature. PV of oils give the oxidative rancidity degree of its. The PV also used as an indicator in

determination of effectiveness of the deodorization process during the refining process (Yağlarda Serbest Asitlik Tayini, n.d). PV is the measurement of hydroperoxides released as a result of oxidation in oil (Özdoğan, Tunalıoğlu, 2017). PV shows the quality and safety of oil and initial stage of the deterioration (Gotoh, Wada, 2006).

#### **2.6.4 Fatty Acid Composition**

Vegetable oils contain saturated and unsaturated FFA. The more unsaturated fatty acids in the oil, the more prone the oil is to deterioration. The fatty acid composition of the oil gives a comment on the stability of the oil. It is important in determining the negative effect of oil on human health (Zambiasi, et al., 2007). Fatty acids are divided into saturated and unsaturated fatty acids.

Unsaturated fatty acids are found in vegetable oils and in sea creatures living in cold waters such as fish and salmon. While the negative effects of saturated fatty acids on human health are known, unsaturated fatty acids reduce the risk of cardiovascular diseases and have positive effects on human metabolism. Unsaturated fatty acids have a protective effect against diseases such as cardiovascular diseases, depression, migraine, joint pain and cancer (Çakmakçı, Kahyaoğlu, 2012).

#### **2.6.5 Color**

Color is an important sensory quality parameter that enables the product to be preferred for the end customer. During the processing of the product, there may be color changes due to some reactions (Özbek, et al., 2021). In addition, the color parameter allows us to comment on the refining process. Oils have their own unique colors (Fengxia, et al., 2001).

#### **2.6.6 Polycyclic Aromatic Hydrocarbons (PAHs)- Benzo (a) pyrene**

Food products subjected to high heat treatments may contain high concentrations of benzo[a]pyrene (BaP) and polycyclic aromatic hydrocarbons (PAHs) harmful to human health (Purcaro, et al., 2009). PAHs are compounds that can be found in food products. They are likely to occur in processed foods, especially when exposed to high temperatures. They contain carbon and hydrogen atoms and contain aromatic rings. The number of rings in their chemical structure determines their properties. In studies, PAHs in food products are formed due to environmental contamination and processing

conditions. PAHs can be found in large amounts in edible oils. Drying of oil-containing raw material at high temperatures can cause the formation of PAHs. In oil extraction processes, the drying process before the extraction process affects the PAH content of the product.

Drying the pomace is a step that affects the quality parameters of the oil such as color, oxidative stability, etc. In addition, high temperature treatment in drying stage will cause undesired compounds such as PAHs or oxidized compounds to occur in the olive pomace oil. For this reason, studies are carried out for alternative techniques that will reduce these negative effects (Rodríguez-Gutiérrez, et al., 2012).

### **2.6.7 *p*-Anisidine value**

*p*-Anisidine value (*p*-AnV) indicates progressive oxidative bitterness in oils. Aldehydes cause bad flavors in oils. Anisidine analysis provides a qualitative evaluation of low peroxide and highly unsaturated fats. Anisidine analysis and peroxide analysis are used to determine the total oxidation value. Also, anisidine value can be used when evaluating the quality of the oil (Labrinea, et al., 2001).

### **2.6.8 K270- K232**

Ultraviolet spectrophotometric analysis is performed to determine the oxidation degree of the oil. Analysis is done at 232 and 270 nm wavelengths. It is an easy and effective way to determine the degree of oxidation. The absorbance value at 232 nm, K232, indicates diene conjugates formed during olive pomace correction. The absorbance value at 270 nm represents K270, diene conjugate, primary and secondary oxidation (Balaky, et al., 2020).

### **2.6.9 Chlorophyll and Carotenoid Content**

Carotenoids and chlorophyll give the oil its distinctive colour. Color is a property that influences consumer choice and can represent deterioration in oil (Konuşkan, 2008).

The amount of chlorophyll and carotenoids varies depending on environmental factors, ripeness of the fruit and the fruit. Carotenoids can absorb light. It readily isomerizes, oxidizes and can bind to hydrophobic surfaces. It is also a singlet oxygen sedative and singlet oxygen sedative is important on the oxidative stability of the oil (Konuşkan, 2008).

Chlorophyll is the pigment that gives plants their green color. As the olive matures, it loses its green color and thus the amount of chlorophyll decreases. Chlorophyll is sensitive to heat. Factors such as oxygen, temperature and light accelerate the breakdown of chlorophyll. Chlorophyll is broken down faster than carotenoid. Chlorophyll is important on the oxidation of oil as it shows chlorophyll prooxidant in the dark (Konuskan, 2008).

## **2.7 Literature Survey on Olive Pomace Drying**

Göğüş, Maskan (2006) examined the drying of olive pomace at different temperatures, various sample thicknesses and particle sizes by air drying method. From this study, it was concluded that temperature, thickness and particle size affect the drying rate of olive pomace. The increase in temperature and particle size caused the drying time to decrease and the increase in sample thickness caused the drying time to increase. In a similar study, it was observed that high temperature and small particle size increase the speed of conventional drying (Freire, Figueiredo, Ferrao, 2001)

In other study, Göğüş, Maskan (2001) studied on the drying of olive pomace by microwave drying. It was obtained that there was no effect of sample thickness on drying rate in studied range but it was also concluded that the moisture content of the olive pomace was at close levels in thicker or thinner samples. Therefore, the same results are obtained and energy is saved by spending less energy on bulk samples. Microwave oven and fan-assisted microwave drying increased the drying speed of olive pomace compared to air drying. The increased temperature shortened the drying time.

Tea drying with high frequency (RF) has been tried. As a result of the studies conducted, it was concluded that less energy is spent and the quality of the dried tea obtained is better (Tunçer, 2006).

**Table 2.** Summary on literature survey on olive pomace drying

<b>Sample</b>	<b>Drying Method</b>	<b>Referance</b>
Olive Pomace	Air Drying	Göğüş, Maskan (2006)
Olive Pomace	Microwave oven and fan-assisted microwave drying	Göğüş, Maskan (2001)
Olive Pomace	-	Yanık (2017)
Olive Pomace	Air Drying (by a forced air laboratory drier)	Qasem, et al. (2013)
Olive Pomace	Open-Air Drying	Missaoui, et al. (2017)
Olive Pomace	Fluid-Bed Dryer	Terramoccia, et al. (2013)
Olive Pomace	Microwave preheating+Conventional Drying	Kiralan, Erdogdu, Tekin (2017)
Olive Pomace	Drum Dryer-Tray Dryer	Baysan, Koç, Güngör et al. (2020)

## CHAPTER III

### MATERIALS AND METHODS

#### 3.1 Materials

In this study, olive pomace was collected directly from a three-phase decanter outlet of an olive oil extraction company located in Nizip, Gaziantep. The moisture content and the oil content of the pomace were %56 and %6.32, respectively. Olive pomace was stored at 5 °C in polyethylene bags for further studies. Standard chromatographic grade BaP and fatty acid methyl ester (FAME Supelco® 37 mixture) were purchased from SigmaAldrich. In Gas Chromotography analysis, *n*-hexane and *n*-heptane solvents were used. Other reagents and solvents were of analytical grade. Ultra-pure water for HPLC analysis was purified by a Millipore system (Milli-Q system, Millipore, Bedford, MA, USA).

#### 3.2 Hot Air Assisted Radiofrequency Drying System

During the pomace drying experiments, a RF system with a pilot plant scale 10 kW, 27.12 MHz free oscillating through-field electrode hot air heating unit was used (Figure 3.1). This system was obtained from Sonar, Izmir, Turkey in Figure 3.1. In this system, the upper electrode (82.0×40.0 cm<sup>2</sup>) potentially was charged while the lower electrode was ground. The electrode gap of the system could be set in the range of 5 to 20 cm. The blowing air temperature inside the RF system could be changed up to 80°C.



**Figure 3.1** Hot Air Assisted RF Drying System

### **3.3 Hot Air Assisted Radiofrequency Drying**

Studies were carried out by operating the drying system at 2 power level. (leading to the potential of 3120 V along the charged top electrode). This setting ensured that the pomace was dried in the optimum time. It would cause the pomace to burn before the desired drying criteria could be achieved at higher power levels. However, the RF effect could not be seen at lower power settings. During the study, the drying process was completed by keeping the parameters of 50 °C and 1.5 m/s constant. One hour before the start of the experiment, the system was turned on and made ready for drying. Thus, when the temperature and the system itself were stabilized, the work was started. Drying studies were carried out by placing the moist pomace polypropylene tray (28.5 cm x 19.5 cm x 10 cm) designed to allow hot air to pass through the sides and bottom.

During the drying process, the tray was removed from the system every 10 minutes and the weight was measured and noted. The electronic balance (SW, CAS, Korea) was used for weight measurement. Drying process was carried out until moisture content of olive pomace reduced to  $0.04 \pm 0.01$  g water/g dry solids (ds).

First of all the effect of electrode gap on the HA-RF drying behavior of olive pomace was studied between the 90 mm and 120 mm with a 1000 g of sample and 7.5 cm of sample thickness. Although drying was rapid at 90 mm electrode gap, burns were observed on sample surface during the drying. Hence 105mm of electrode gap was accepted as the most suitable electrode distance. One of the dried olive pomace visual is given in Figure 3.2.

Secondly the effect of the sample thickness on the drying behavior of bulk olive pomace in HA-RF drying was determined at four different thicknesses: 2.5 cm, 5 cm, 7.5 cm and 10 cm. The sample weight was kept constant at 1000 g and electrode gap at 105 mm.

Then, in order to observe the effect of pressing (sample porosity) on the air permeability 3 different experiments were performed in HA-RF drying. The pressing experiment was carried out using 2.5 kg of sample. First, we dried the sample at 10 cm thickness in loose without pressing. Then we applied moderate pressure to the 10 cm sample and pressed it to 7.5 cm. Then, by increasing the pressure, we compressed it well and pressed it to 5.5 cm (it decreased to a maximum of 5.5 cm). In this way, we also observed the effect of pressing on drying.

Finally, in order to show the effect of RF on drying behavior of olive pomace, first air-drying experiments were performed with 1000 g at 2.5 cm of sample thickness, 50 °C of air temperature and 1.5 m/s of air flow rate without RF application. Then in another experiment air blowing was turned off and only RF (at 105 mm of electrode gap) was turned. And lastly HA and RF were combined (HA-RF) to observe how the synergetic effect of them at 2.5 cm of sample thickness, 50 °C of air temperature and 1.5 m/s of air flow rate and 105 mm of electrode gap.



**Figure 3.2** Dried Olive Pomace by HA-RF

### 3.4 Drying Kinetics

Drying curves were modeled with the drying data of moist olive pomace dried by HA-RF drying method. In the modeling, the kinetics of drying was specified together with the preliminary during drying of moist olive pomace. All time, weight data recorded during drying studies were used in modeling drying curves.

The following equation was used in the calculation of the moisture ratio (MR) of the samples:

$$MR = \frac{M_t - M_e}{M_0 - M_e} \quad (1.6)$$

where  $M_t$ ,  $M_e$  and  $M_0$  are the moisture content (db) at time  $t$ , equilibrium moisture content (db) and initial moisture content (db), respectively.

MR data and time from drying studies were fitted to the models using the Sigma Plot 12.0 (Systat Software Inc., Erkrath, Germany) curve fitting tool. The fitting of the selected models was obtained using the coefficient of determination ( $R^2$ ), root mean square error (RMSE), and standard error of mean (SEM). RMSE and SEM were previously reported by Gong et al. (2020) and the drying rate (DR) was calculated by the equation given below:

$$DR = \frac{M_{t1} - M_{t2}}{t2 - t1} \quad (1.7)$$

Where  $M_{t1}$  and  $M_{t2}$  are the moisture content at time  $t_1$  and  $t_2$  in g water/g dry solid,  $t$  is the drying time (min).

### **3.5 Moisture Content and Oil Content Determination**

In order to determine the amount of moisture in olive pomace, 10 g of dried olive pomace samples were taken and kept at 130 °C until there was no change in weight and the moisture content was calculated. In order to determine the amount of oil in pomace, the total oil amount was determined with the help of n-hexane by using the Soxhlet method.

### **3.6 Extraction of Olive Pomace Oil**

As a result of the studies carried out in the drying process under the different conditions, the most effective diffusivity of the pomace was achieved with 2500 g sample, 100 mm thickness and 105 mm electrode distance. Therefore, the oil quality evaluation was made on dried olive pomace obtained at these conditions. Extraction of oil process was completed by cold extraction method and with the help of n-hexane. Hexane was removed by using a rotary vacuum evaporator (Heidolph, Germany) in order to evaporate the hexane. The moisture content of the dried pomace to be used in oil extraction was below 2%.

### **3.7 Oil Quality Evaluation**

The FFA content, PV and p-AnV values in olive pomace oil were determined by AOCS method Ca 5a-40, AOCS standard method Cd 8–53 and AOCS Official Method Cd 18– 90 respectively. The total oxidation value (Totox) value of the oil was calculated by the formula given below 1.10 (Moigradean et al., 2012).

$$Totox \text{ value} = 2PV + pAnV \quad (1.10)$$

All analyzes were repeated three times in order to obtain accurate results and the results were given with the averages with their standard deviations.

### 3.7.1 Spectrophotometric Examination in The Ultraviolet (K232 and K270 values)

The UV absorbance value of olive pomace oil at 232 and 270 nm was determined with reference to the IOC (2019) standard. 0.25 g, approximately 1 mg oil sample was put into a 25 ml graduated cylinder and the volume of the cylinder was completed with cyclohexane to ensure a homogeneous state. Absorbance values were determined by spectrophotometer (Optima, SP-3000 nano, Japan) at 232 and 270 nm.

### 3.7.2 Color Measurement

The color of the oil was analyzed using the Hunter-Lab ColorFlex calorimeter. Color specified based on CIELAB definition.

L\* (bright; 100- white from 0 to black), a\* (+ a = redness, -a = greenery) and b\* (+ b = yellowness, -b = blue) values were measured by the parameters on the daylight D65 and observer angle of 10°. Hue angle (H\*) and chroma (C\*) values were calculated by substituting the measured L\*, a\* and b\* values in the 1.11 and 1.12 given below.

$$H^* = \tan^{-1}(b^*/a^*) \quad (1.11)$$

$$C^* = \sqrt{((a^*)^2 + (b^*)^2)} \quad (1.12)$$

### 3.7.3 Determination of Fatty Acid Composition

For the fatty acid composition analysis of olive pomace oil, the fatty acids were first converted to methyl esters as specified in the IOC (2017). After this step, analysis was started by injecting 1 µL of FAME into gas chromatography (GC). The Agilent 7890A GC (Agilent Technology, USA) was used for GC analysis. GC was equipped with a flame ionization detector, a split / splitless injector and an HP-88 capillary column (88% Cyanopropylaryl 100 m x 0.250 mm ID x 0.20 µm). The initial column temperature was kept constant at 120 °C for one minute and then increased by 10 °C per minute to 175 °C. And again after 10 minutes, the temperature was increased to 210 °C by increasing 5 °C per minute and this temperature was kept stable for five minutes. After five minutes, the temperature was increased by 5 °C per minute to 230

°C and this temperature was kept stable for 15 minutes. FAMES were determined by comparing the retention time of the standard mixture 37 FAMES.

### 3.7.4 Determination of Chlorophyll and Carotenoid Content

Chlorophyll of olive pomace oil was determined by spectrophotometric method based on the method in AOCS Cc 13k-13. The absorbance value of olive pomace oil was measured at 630, 670 and 710 nm against air. The content of chlorophyll pigments was calculated as pheophytin a in mg/kg of oil with the help of 1.13

$$C = kx \frac{A_{670} - 0.5(A_{630} + A_{710})}{L} \quad (1.13)$$

Where k is a constant factor, L is light path of spectrophotometer cell (cm).

The total carotenoid content of olive pomace oil was determined as described by Gao et al (2007). 0.5 g oil sample was taken and dissolved in 4 mL of hexane. The absorbance of the solution was measured at 445 nm with the help of n-hexane as blank. The total carotenoid content was calculated by using 1.14

$$\text{Total Carotenoid (mg/kg)} = \frac{A_{445} \times V \times 10^6}{2500 \times m \times 100 \times d} \quad (1.14)$$

Where, d: cell diameter (cm); V: volume of solution (mL); m: mass of oil sample; 2500 dL g<sup>-1</sup>cm<sup>-1</sup> is specific extinction coefficient of carotenoids at 445 nm in n-hexane.

### 3.7.5 Determination of BaP

Extraction PAHs from olive pomace oil was performed by SPE method based on the method used by Moret, Conte (2002). For (BaP) content, extraction was performed with the Shimadzu Prominence / LC-20 AB HPLC method specified by Yanık (2017).

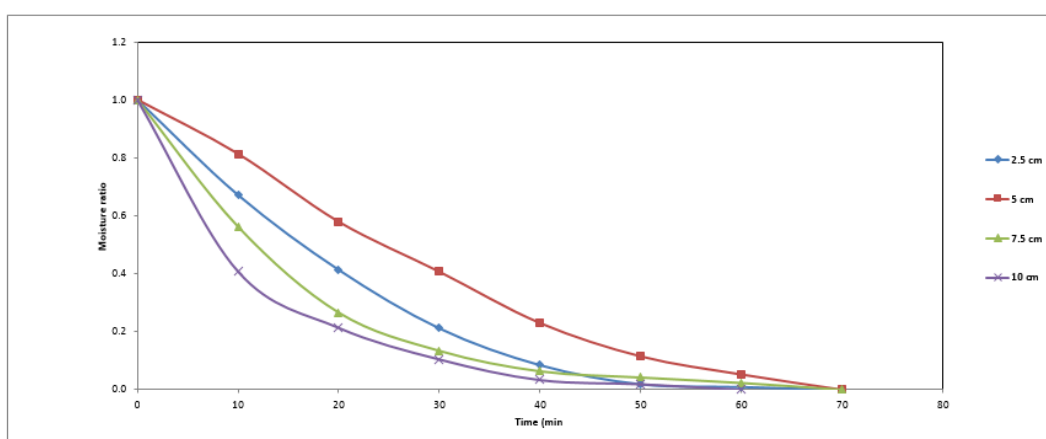
## CHAPTER IV

### RESULTS AND DISCUSSION

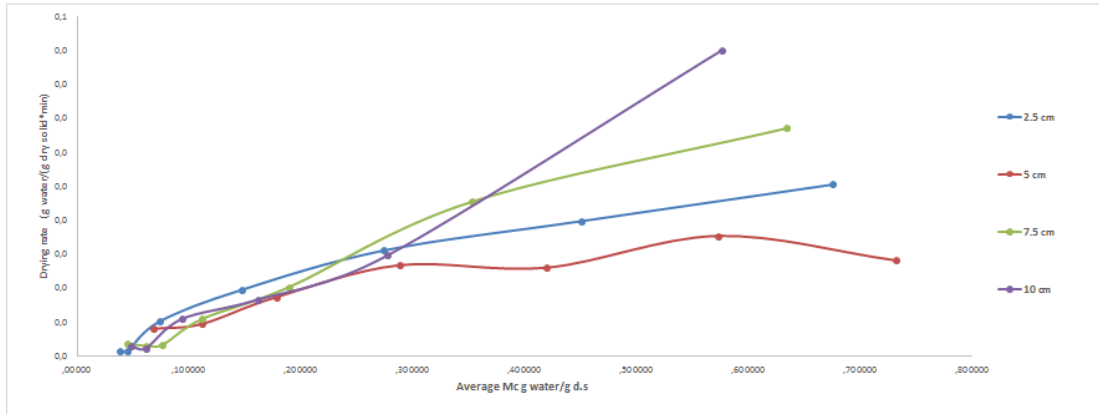
#### 4.1 Drying Kinetics

##### 4.1.1 Effects of Sample Thickness on Drying Behavior of Olive Pomace in HA-RF drying

The effect of sample thickness (2.5-10 cm) on moisture curve and drying rate of olive pomace was given in Figure 4.1 and Figure 4.2, respectively. It was seen that increasing the sample thickness caused to increase in drying rate after a critical thickness. This trend is related to an increase in height on the side surface and acquiring of drying also from the sides. Drying was completed within the 60 min for sample with 10 cm thickness. In contrast to our findings, Elik (2021) reported that the drying rate is higher at smaller thickness in HA-RF drying of balck carrot pomace. Also, Özbek (2021) reported that drying time increaes with the increasing sample thickness in HA-RF drying of carrot powder. The differences in findings are related to the nature of the product to be dried. Olive pomace is a bulk sample with high porosity in loose form. Hence increasing height of sides contributes to the drying.



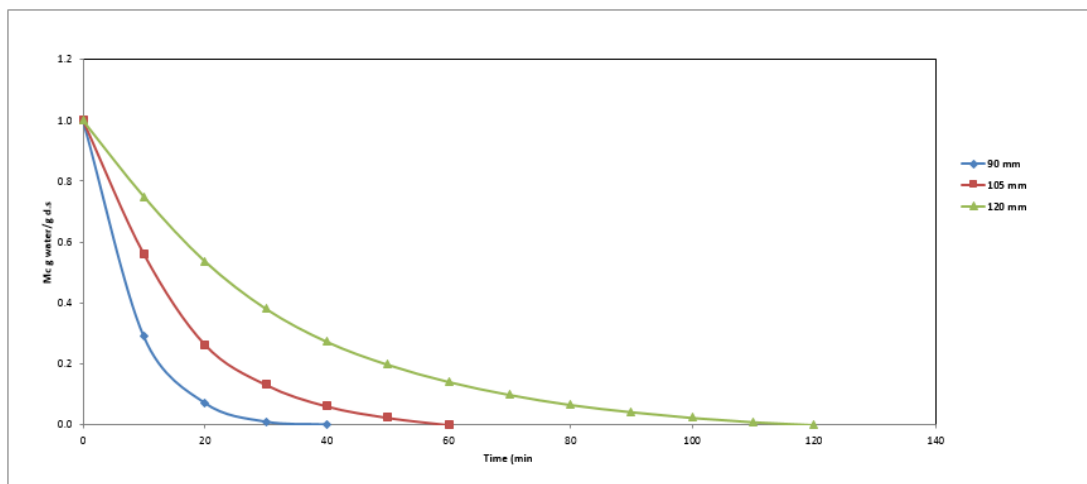
**Figure 4.1** Drying curve of olive pomace by HA-RF with different sample thickness



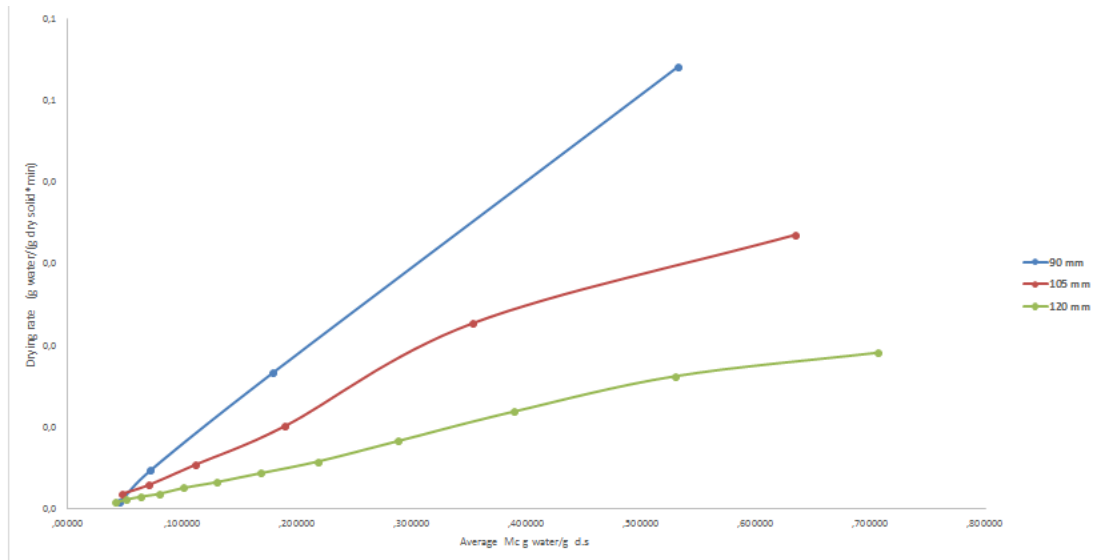
**Figure 4.2** Drying rate of olive pomace by HA-RF with different sample thickness

#### 4.1.2 Effects of Electrodes Gap on Drying Behavior of Olive Pomace in HA-RF drying

The effect of electrode gap (90, 105 and 120 mm) on moisture curve and drying rate of olive pomace was given in Figure 4.3 and Figure 4.4, respectively. The sample weight and sample thickness were kept constant (1 kg and 7.5 cm samples) in these studies just electrode gap was changed. It was seen that the shorter the electrode gap, the higher the drying rate. According to Elik (2021), shorter electrode gap increases drying rate because of the more intense electrical field. Also, Jiao, et al. (2016) reported that drying rate increases with shorter electrode gap by HA-RF drying of peanuts.



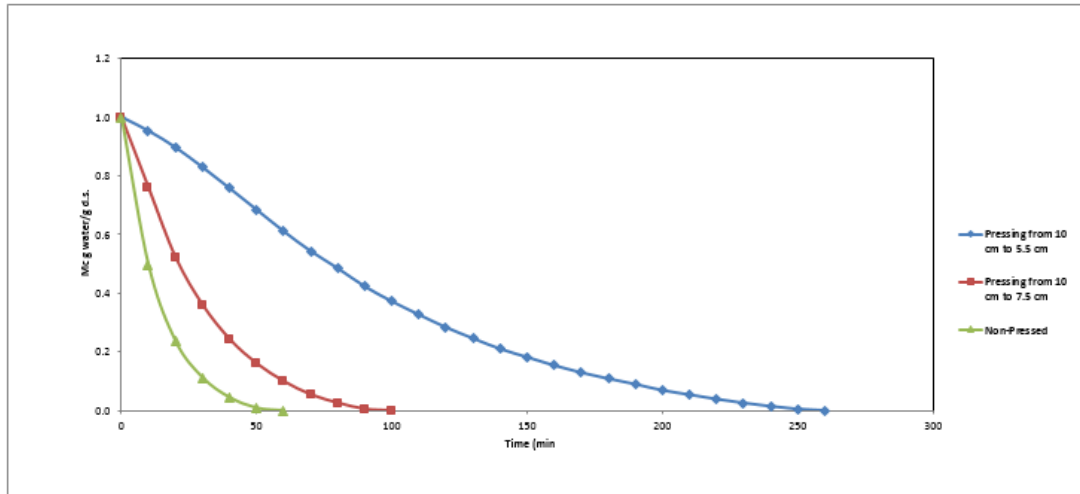
**Figure 4.3** Drying curve of olive pomace by Hot air assisted RF with different electrode gap



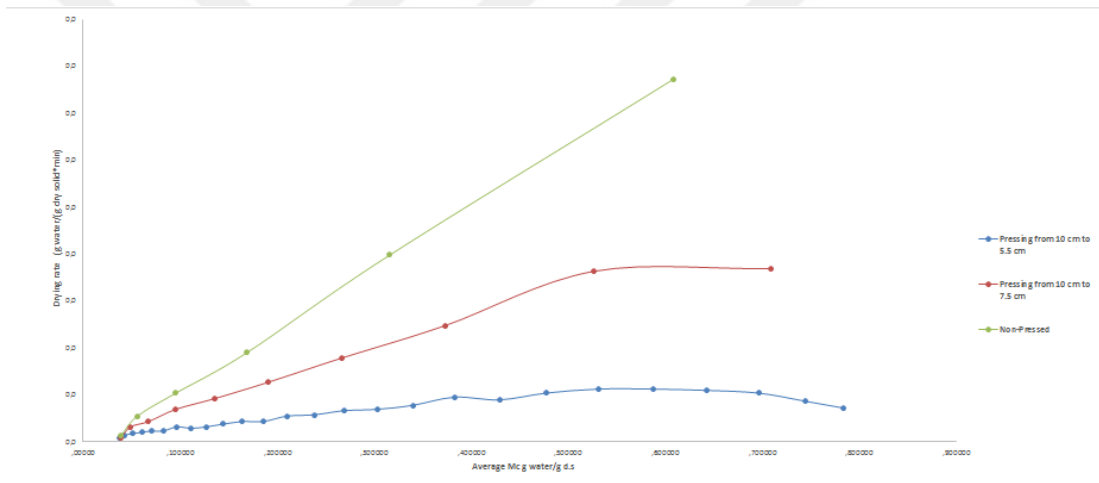
**Figure 4.4** Drying rate of olive pomace by Hot air assisted RF with different electrode gap

#### 4.1.3 Effects of Sample Porosity on Drying Behavior of Olive Pomace in HA-RF drying

The effect of sample porosity on moisture curve and drying rate of olive pomace was given in Figure 4.5 and Figure 4.6, respectively. The sample weight was kept constant (2.5 kg) in these studies and pressing force was applied to the sample. At the beginning, sample thickness was 10 cm in loose form. Then it was pressed to 7.5 cm and 5.5 cm, to reduce the porosity in bulk pomace to observe the effect of porosity on drying behavior. The highest drying rate was observed for the 10 cm thick sample (the unpressed sample) since air easily passes through the pores in the unpressed sample. However, as the amount of pressing increases, the air passage through the gaps in the bulk sample decreases and thus the drying rate slows down. Elik (2021) reported that sample porosity was the major parameter affecting drying rate of the sample. As the porosity of the sample increases, the drying rate also increases.



**Figure 4.5** Drying curve of olive pomace by Hot air assisted RF with pressing and without pressing

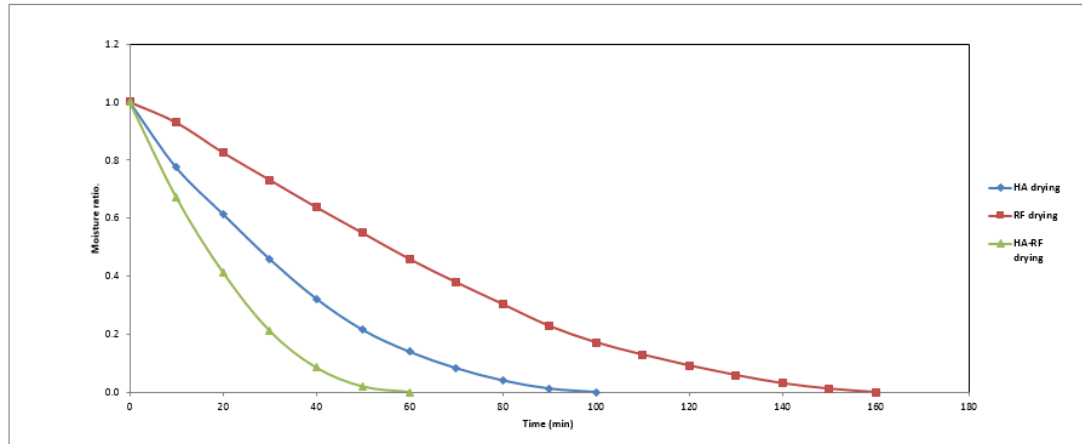


**Figure 4.6** Drying rate of olive pomace by Hot air assisted RF with pressing and without pressing

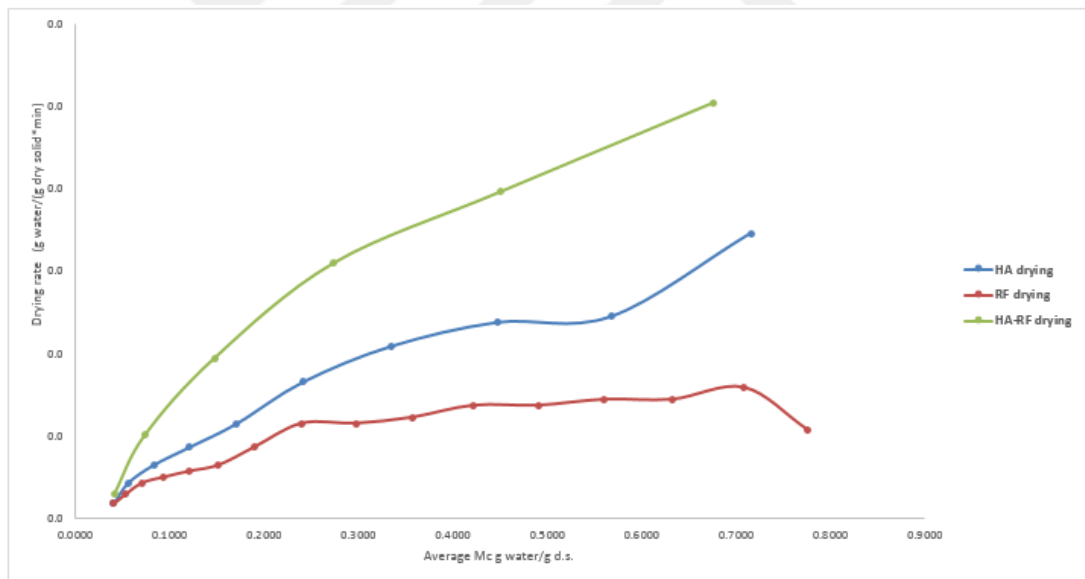
#### 4.1.4 Comparison of Drying Behavior of Olive Pomace by Hot air, Hot air assisted RF and RF Drying

Figure 4.7 and 4.8 show the effect of the different drying methods on the drying curve and the drying rate of olive pomace, respectively. It was seen that the drying rate significantly increase by the RF application. Moreover, RF application together with hot air help to remove more moisture from the solid matrix compared to the alone hot air application. Hence, HA-RF application result in lower equilibrium moisture content than that of reached in alone hot air application. Similarly, Elik (2021) reported that the drying rate increased by HA-RF application in drying of black carrot pomace.

In another study, Zhang, et al. (2016) studied on HA-RF drying of in-shell walnuts and obtained that drying was more effective in HA-RF compared to alone hot air drying. Also, Wang, et al. (2014) studied on HA-RF drying of Macadamia nuts and obtained HA-RF reduced drying time by half compared to alone hot air drying.



**Figure 4.7** Drying curve of HA, HA-RF and RF drying applications.



**Figure 4.8** Drying rate of HA, HA-RF and RF drying applications.

## 4.2 Physical and Chemical Properties of Crude Olive Pomace Oil

It was important to determine the optimum conditions during the drying phase of moist olive pomace using the HA-RF method. Because the most important stage affecting the quality parameters of pomace oil is the pomace drying stage.

**Table 3.** Physical and chemical properties of crude olive pomace oil

	Crude olive pomace oil
Free fatty acid content (% oleic acid)	1.09±0.02
Peroxide value (meq O <sub>2</sub> /kg oil)	12.20 ± 0.07
<i>p</i> -AnV	3.01 ± 0.29
Totox	27.40 ± 0.14
PAH content (BaP, µg/kg oil)	< 1*
Chlorophyll content (mg pheophytin a/kg)	105.25 ± 0.84
Carotenoid content (mg /kg)	2.85 ± 0.02
UV absorption	
K <sub>232</sub>	1.43 ± 0.01
K <sub>270</sub>	0.23 ± 0.01
Color (CIE)	
L*	38.60 ± 0.03
a*	7.50 ± 0.01
b*	62.56 ± 0.21
Hue Angle	83.11 ± 0.01
Chroma	62.98 ± 0.12
Fatty acid composition (%) <sup>a</sup>	
C16:0	13.36 ± 0.19
C16:1	0.68 ± 0.02
C18:0	4.52 ± 0.03
C18:1	65.14 ± 0.54
C18:2	14.23 ± 0.07
C20:0	0.66 ± 0.03
C18:3	0.65 ± 0.01
C20:1	0.31 ± 0.01

\*Below the detection limit of the instrument

L\*represents lightness.

a\*represents redness.

b\*represents yellowness.

<sup>a</sup> Results expressed as percent over the total content (relative content).

It is important that the drying method is not carried out in a way that adversely affects the quality of the olive pomace oil. For this reason, with the preliminary studies we made in our drying experiments, optimum conditions were determined for olive pomace oil and drying was carried out under optimum conditions for olive pomace whose oil will be extracted.

The quality parameters of the crude oil of the pomace dried by the HA-RF method are given in the Table 3. No limit for crude olive pomace oil has been set by the European Commission regulation 2016/2095. However, these quality parameters are important to determine the commercial value of olive pomace oil. Moreover, these parameters indicator of how easily refining will be achieved.

The degree of hydrolytic rancidity was measured by FFA content, while degree of oxidative rancidity of RF-dried olive pomace was measured by PV, p-AnV and totox value.

The PV indicates the first stage of degradation in oils (Gotoh, Wada, 2006). The p-AnV indicates the amount of products formed as a result of secondary oxidation (Gordon, 2004). Totox value is calculated with PV and p-AnV. The higher these values represent the higher the oxidation degree of the oil.

The results showed that the FFA content and the PV, p-AnV and totox values were sufficiently low because olive pomace was directly obtained from the process line and immediately transferred to the laboratory for drying experiments. However, in general practices in olive pomace industry the olive pomace released is kept in the garden of the factory for months, and the acidity and PV getting worse over the time.

The FFA value in crude oil was found to be  $1.09 \pm 0.02$  % oleic acid. Looking at the different previous studies, the amount of FFA varied between 6.52-14.09%, based on the samples analyzed from fourteen different crude olive pomace oils taken from pomace plants in Italy by Gomes, Caponio, (1997). The FFA content of the crude olive pomace oil purchased by Kiralan et al., (2019) from a company in Turkey was

measured as 12.58%. Considering these values, it can be said that the FFA content of the crude oil obtained from pomace dried by HA-RF method is low. As a result, when we take the FFA ( $\leq 1\%$ ) value specified for olive-pomace oil (category 8) as a basis in the European commission regulation 2016/2095, it can be said that the olive pomace oil dried with the HA-RF system is of high quality because it is close to the limit value.

The PV in crude oil was found to be 12.2 meq O<sub>2</sub>/kg oil. This value is lower than the PV for crude olive pomace oil in the literature.

It also complies with the PV specified by the European Commission regulation 2016/2095 for crude olive pomace oil, <15 meq O<sub>2</sub>/kg oil. Looking at the other pomace oil studies in the literature, Burn (2017) reported this value as 17.80 meq O<sub>2</sub>/kg oil in crude pomace oil obtained from moist pomace by microwave method. In addition, Yanık (2017) stated the PV of industrial oil as 47 meq O<sub>2</sub>/kg oil. Gomes, Caponio, (1997) measured the PV in the range of 10.6-38.8 meq O<sub>2</sub>/kg oil in the analysis of fourteen samples of olive pomace oil. Gomes, Caponio, (1997) stated that the PV in the crude olive pomace oil by vacuum drying at 30°C under laboratory conditions was in the range of 13.5-24.1 meq O<sub>2</sub>/kg oil.

The p-anisidine value, which represents the secondary oxidation products, was also calculated for the oil due to the conversion of primary oxidation products into secondary oxidation products. The p-AnV of this crude olive pomace oil was found to be 3.01, thus the lowest p-AnV reported in the literature for crude olive pomace oil. Gomes, Caponio, (1997) stated the p-anisidine value of 8.74 to 14.12 for crude olive pomace oil in their studies. In another study, Gomes, Caponino, (2001) measured p-AnV in the range of 5.48-11.22 from samples taken from refined olive pomace oils obtained from different plants.

As mentioned earlier, the totox value was also calculated to indicate the oxidation state of the oil. The totox value of the oil was 27.4, shows that there is no serious oxidative deterioration in the oil based on the information in the literature.

The totox value was calculated through the PV and the p-AnV according to method of Gomes, Caponio (1997) and stated that the totox value in industrial crude olive pomace oil is in the range of 30.37-80.38. Cerretani et al., (2009), on the other hand, calculated

the tototox value of a mixture of extra virgin olive oil and refined pomace oil to be over 25.

In addition, the specific UV absorption values at 232 and 270 nm are other parameters that provide information about the oxidation level of the oil. The absorbance value at 232 nm wavelength represents the diene conjugates, while the absorbance value at 270 nm represents the conjugates trienes in oil.

Gomes, Caponio, (1997) stated the K<sub>232</sub> value of crude olive pomace oil as 3.88, while the value was measured as 1.43 in crude oil of pomace dried by HA-RF method. Rodríguez-Gutiérrez et al., (2012) measured the K<sub>232</sub> value between 3.23 and 5.27 in processed and unprocessed crude pomace oils and it is still high.

The K<sub>232</sub> and K<sub>270</sub> values in the crude oil of the pomace dried by the HA-RF method are low, as are the PV and the p-AnV, and there is consistency between these parameters.

The PAH are components that occur in products that undergo high heat treatment and are dangerous to human health. BaP is a PAH that poses a great risk to human health. In general, the reason for the increase in PAH concentration in crude olive pomace oil can be shown as excessive exposure to smoke or incomplete heat treatment in traditional drying processes (Yanik, 2017; Liebanes et al., 2006; León-Camacho et al., 2003). The European Commission regulation No. 208/2005 limited the amount of benzo(a)pyrene required in oils to a maximum of 2 µg/kg oil. It was measured to be very significantly low, < 1 µg/kg oil, in the oil of the pomace dried by the HA-RF method.

When we look at the literature, Yanik (2017) reported the amount of benzo(a)pyrene in crude olive pomace oil as 1.67 µg/kg oil in previous studies. Ergönül, Sánchez (2013) stated it as 13.55 µg/kg oil and León-Camacho et al. (2003) as 185-364 µg/kg oil. Since the amount of PAH in oils is an important parameter, the low BaP content in olive pomace oil dried by HA-RF method indicates that RF drying will play an important role in reducing the amount of PAH.

Chrophopyll and caratenoids are natural coloring pigments (Gallardo-Guerrero et al., 2002). The higher amount of chlorophyll pigment, the more intense green color of olive pomace. The chlorophyll and carotenoid content of crude olive pomace oil were

obtained as 105.25 mg pheophytin A /kg oil and 2.85 mg/kg oil, respectively. Serafini, Tonetto (2019) were reported the chlorophyll content (77 mg pheophytin/kg crude olive pomace oil) and the chlorophyll content of crude olive pomaceoil was higher than reported value. However, according to average values of extra virgin olive oil (11.75 mg chlorophyll /kg oil, 8.21 mg carotenoid/ kg oil) reported by Cho, Lee, (2014) ,carotenoid content of crude olive pomace oil was lower.

The concentration of the coloring pigments in olives depends on the region where the olive is grown, agricultural factors, and the processing of the olive (Criado et al., 2007; Giuliani et al., 2011). For this reason, the chlorophyll and carotenoid contents specified in the literature may differ from each other.

The drying process of pomace is another important factor that affects the color of raw olive pomace oil. The color acceptance of olive oil and olive pomace oil derivatives is determined by the consumer himself. Customer could choose desired color oil in shelves. Because of that reason color of final product is important. When color of product is not proper for customer, processes take places to obtain desired color and they cost to extra money. Exposure of the product to high temperatures during the drying process affects the color of the product. As a result, a darker color than desired is obtained. This non-compliance will increase the cost with the increase in the interventions to the color of the oil during refining.

As a result of the color analysis, the L\* (brightness), a\*(redness) and b\*(yellowness) values in the raw oil of the pomace dried by HA-RF method were determined as 38.6, 7.5, and 62.56, respectively. While the brightness values reported by Yanık (2017) were in the oil obtained by the microwave method (L\*: 36), they were in the oil obtained by the conventional method (L\*: 15.41), and the brightness value was better in HA-RF oil. In this case, it is thought that the color values of the raw oil of the pomace dried by HA-RF method are better and it will reduce the cost of bleaching in the refining process.

According to the fatty acid composition analysis results; the highest rate of oleic acid (65.14%) was detected in the composition, followed by linoleic (14.2%) and palmitic acid (13.4%). It is consistent with the fatty acid composition previously stated for crude olive pomace oil in the literature (Yanık 2017; Yorulmaz 2018). It can be

concluded that there was no any adverse effect of RF heating on fatty acid composition of olive pomace oil.



## **CHAPTER V**

### **CONCLUSION**

- The highest drying rate was HA-RF application in drying of olive pomace with compared to hot air and RF drying.
- The best drying was performed within the 60 min, for 1000 gr of sample at 105 mm electrode gap and 10 cm of sample thickness.
- The increase in the sample thickness from 2.5 to 10 cm (at constant weight) increased the drying rate in HA-RF drying of olive pomace after a critical thickness (5 cm).
- The shorter the electrodes gap the higher the drying rate.
- Pressing of sample (reducing the porosity in bulk sample) reduced the drying rate.
- No adverse effects of HA-RF on final oil quality were observed.
- The drying method with HA-RF shows that it can be a reliable method in the process of obtaining olive pomace oil, with its rapid and homogeneous drying.
- HA-RF could be declared as promising drying method for olive pomace with its low oxidation level, good color quality and no PAH formation.
- Further studies are needed to examine the energy efficiency and cost of HA-RF method as well as continuous option for industrial scale.

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### **EDUCATION STATUS**

**GAZİANTEP UNIVERSITY (2014-2015)**

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**GAZİANTEP UNIVERSITY (2015-2019)**

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Graduation grade: 3.29/4.00

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**GAZİANTEP UNIVERSITY (2016-2021)**

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**KAUNAS UNIVERSITY OF TECHNOLOGY (LITHUANIA) (09.2018-02.2019)**

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**GAZİANTEP UNIVERSITY (2019-)**

- Graduate School of Natural and Applied Sciences - Food Engineering Master's  
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## **FOREIGN LANGUAGE AND LEVEL**

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## **COURSES AND CERTIFICATES**

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Upper Intermediate Level English Course At the end of the English Preparatory class.

- ISO 22000: 2005 (Food Safety Management System)
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