Drying of Fruits:Improvement of Quality and Process Modelling

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DRYING OF FRUITS:
IMPROVEMENT OF QUALITY AND
PROCESS MODELLING

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Conference Proceedings

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Önal, B., Adiletta, G., Russo, P., Crescitelli, A., Di Matteo, M.,. Influence of process parameter and pre-drying treatment on nutritional quality of traditional Southern Italian Cultivar "Annurca" apple, 5th International ISEKI Food Conference, July 3-5, 2018, Stuttgart, Germany, Book of Abstracts published by: ISEKI-Food Association, ISBN: 978-900932-52-2. Editors: Pittia, P., Silva, C.L.M., Schleining, G., Braun, S., Reinhardt, N., Hebrard, F.J.C., Chrysanthopolou, F., Habershuber, A.

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Abstract

In the recent years, dehydrated food industry has gained prominence in the world. In concern with increasing demand of high quality and healthy products and changing customer behaviour, the food market needs to maintain at a high level nutritional and sensory properties of the initial fresh products. Drying of fruits and vegetables enables the availability of these products on the global markets during all seasons.

The aim of this research was to investigate the effects of different pre-treatments (i.e natural - innovative dipping solutions, microwave and ultrasound applications) and hot air drying process conditions on the drying characteristics and quality propertiesof selected fruits in terms of colour, shrinkage, total phenolics, antioxidant activity, volatile aroma compounds, microstructure, texture, preliminary sensorial evaluation, rehydration behaviour. New mathematical models were derived using drying parameters and new models may be very useful for the design and optimization of industrial dryers. The optimal pre-treatment and drying process conditions were determined according to the type of fruits to dry. From this viewpoint, the combined drying pre-treatment and optimal drying conditions have been proposed with the aim of reducingadverse changes and obtaining high quality/healthy dried snacks. Furthermore, 'Annurca' apple and 'Terzarola Gialla'peach (traditional Southern Italian fruits), as well as, 'Rocha'pear (traditional Portuguese fruit) were used in this research to valorize the traditional fruits.

'Annurca' apple, a Southern Italian cultivar, is known for its reddening, taste and flavour among the other types of apples, and also for health promoting effects. The aim of this part was to evaluate the effect of a novel carbohydrate/salt dipping pre-treatment, and of drying process conditions (temperature and time) on drying kinetics and quality attributes of dried apple slabs. Drying experiments were carried out by convective drying at temperatures of 50, 55, 60 and 65°C at a constant air velocity of 2.3 m/s. Pre-treatment solution provided an increment of moisture loss, and a reduction of drying time and shrinkage at all temperatures. The combination of pre-treatment and drying at 65°C assured the lowest colour changes, the best preservation of structure, less shrinkage, higher rehydration capacity and the highest score for sensorial overall acceptability. On the contrary, the

used pre-treatment combined with lower drying temperatures (50 and 55°C)better preserved the antioxidant activity of apple slabs. In conclusion, the proposed solution enabled to reduce the processing time and better retain the quality attributes (i.e. physical, chemical, nutritional, sensorial) of dried apples slabs for their commercialization as snacks. Also, the influence of this pre-treatment and drying/rehydration temperatures on quality attributes of rehydrated 'Annurca' apple slabs were investigated. The rehydration experiments were carried out at both rehydration temperatures of 30 and 70°C. The combination of pre-treatment, higher drying temperature of 65 °C and low rehydration temperature of 30°C enabled to the best preservation of rehydrated apples' quality in terms of rehydration indices (i.e, water absorption capacity, rehydration ability, water holding capacity), structure and colour properties.

'Terzarola Gialla' is known traditional cultivar of peach in Southern Italy. The impacts of novel dipping pre-treatment and air-drying temperatures (45, 50, 55, and 60°C) on drying characteristics and quality attributes of 'Terzarola Gialla' peach slabs were investigated using a convective dryer and fixed air velocity of 2.3 m/s. Physical, chemical and sensory properties of peach samples were assessed. The obtained results revealed that novel pre-treatment solution not only affected the drying kinetics of peach slabs, it also improved the quality attributes. The combination of pre-treatment and higher drying temperature of (60°C) exhibited shorter drying time, better colour retention, less shrinkage, and higher rehydration capacity. Treated samples dried at 55 and 60°C had higher scores of overall quality attributes by untrained panelists. The antioxidant activity was better retained at lower drying temperatures and the highest antioxidant activity was found in treated dried ones at 50°C. In conclusion, this proposed novel solution was effective to shorten drying time of peach slabs and preserve the overall quality attributes.

'Rocha' pear is the main traditional cultivar produced in Portugal, which is characterized by flavour, crispness and also its sweetness. This part of the thesis was carried out on in collaboration with the Centre for the Biotechnology and Fine Chemistry (CBQF) of the Catholic University, Porto, Portugal. In this framework, microwave (MW) and ultrasound (US) applicationswere used as a pre-treatment prior to the drying process. Vacuum-packed pear slabs weretreated with ultrasound in an ultrasonic bath using 35 kHz for 10 min. The microwave pre-treatment was applied to pear slabs at afrequency of 2450 MHz and microwave power 539 Watt for 4min. Drying experiments were carried out at three different drying temperatures (55, 60, 65°C) using the pilot convective tray dryer at a constant air velocity of 0.75 m/s. The final dried pears'quality were evaluated by means of colour, shrinkage, total phenolic, antioxidant activity, texture and rehydration capacity. The ultrasound pre-treatment did not accelerate the drying process

of pear slabs, while microwave pre-treated samples had shorter drying time in comparison with control and ultrasound pre-treated ones at each investigated temperature. The combination of ultrasound pre-treatment and higher drying temperature of 60°C resulted in less colour changes and shrinkage, better retained total phenolics and antioxidant activity, as well, higher rehydration capacity. On the other hand, microwave pre-treated dried samples indicated the lower overall quality attributes.Therefore, the combined application of ultrasound pre-treatment and higher drying temperature of 60°C may be promising technique forthe efficiency of 'Rocha' pear drying and the better quality pear snacks.

Chapter I
Introduction

I.1 Background of drying process

Nowadays, global foodconsumption is increasingly affected by the three major trends of sustainability, health and convenience. Sustainability is driven by the growing awareness of environmental effects caused by conventional agricultural practices which resulted, for example in an increasing expansion of organic agriculture and markets. Health attitudes are driven by an increasing consumer's interests toward functional food which, beyond the basic function of supplying nutrients, claims to have health-promoting or disease-preventing properties (i.e. obesity, type II diabetes, coronary heart disease ans some cancers). In concern with convenience, the easiness of food consumption (i.e. ready-to eat meals, extended shelf life, home-delivered food, etc.) and food producers during the last decades have responded to this consumer demand by launching on the market an increasing number of convenient food (Asioli et al., 2019; Deng et al., 2019; Soria and Villamiel, 2010; Stehfest et al., 2014; Westhock et al., 2014)

According to the guidelines stated on the EU regulations 834/2007 (European Commission, 2007) and 848/2018 (European Commission, 2018), organic processing methods should guarantee genuineness, authenticity and preservation of the natural properties of the raw materials and also follow the three principles: freshness, minimal processing, and careful treatment (Asioli et al., 2019). From a Western nutritional diet and healthiness point view, fresh fruit and vegetables play an important role in the prevention of chronic disease and premature death. There are many varieties of fresh fruits and vegetables which are known to be rich in vitamins, fibre, antioxidants, polyphenols and minerals. In this respect, processing methods have great importance in terms of preserving not only the nutritional and sensory quality, but also the bioactivity of certain of their constituents. The increasing consumer demand for potentially healthier and more environmentally friendly food products indicate to seek the development of novel processing methodologies to meet the requirements of internal

standards, regarding food safety and simultaneously providing a product appealing to the consumer.

The moisture content of fresh fruits and vegetables is more than 80%, they are classified perishable commodities. Due to their high moisture content and tender texture, it is required the use of some post-harvest treatments in order to be effectively preserved.

Drying is well-known one of the most important preservation techniques in the food industry and it is commonly applied for prolonging the shelf life of fruits and vegetables and improving food stability by reducing moisture content-microbial growth and minimizing chemical deterioration (Brasiello et al., 2017). Moreover, drying can reduce the weight and volume of foodstuffs with also some benefits, such as minimizing packing, storage, and transportation costs (Zhang et al., 2019).

Over the last decades, dried food industry has received growing interest in the world. Drying of fruits and vegetables have made it possible their consumption on the global market during all seasons. In addition to this, in concern with increasing demand of high quality shelf-stable/healthy products and changing customer behaviour, the food markets needs to maintain at a high level nutritional and sensory properties of the initial fresh product. From this viewpoint, valorization of dried foods has become an important issue and its areas of usage has enhanced in the different kind of meals.

Fruits can dried whole (i.e. grapes, berries, apricots and plums), in halves, or as slices (i.e. mangoes, papayas and kiwis). Apples, apricots, dates, figs, peaches, pears and raisins are referred as 'conventional' or 'traditional' dried fruits (Chang et al., 2016). Dried food products can be used in a dry state or will need rehydration before consumption or further processing. Dried fruit products are widely used as valuable ingredients in numerous processed foods (i.e. snack preparations, integral breakfast cereals, preparation of baby foods etc.) by confectionery, baking and sweets industries. Soup manufacturing plants use dried fruits in the various sauces, garnishments, puddings, and ice powders, and food for infants and children. Some fruits such as blueberries, cherries, strawberries, mangoes infused with sugar solutions, fruit juices or pulps which facilitates the dissolution quickly before drying. The development of the fruit powders was possible through processing, which preserves the colour and flavour (vacuum drying, lyophilization, and swelling). Artificial drying made it economically possible to use raw materials at competitive prices and of high quality; examples are apples,prunes,and rose hips. Various milling procedures make it possible to dry highly valuable berries with soft flesh (strawberries, raspberries) and mature stone-fruits (apricots, peaches). In addition to these, rehydration food products are used in milk products (i.e. yogurt, ice-cream, smoothie), instant products, various fruit tea preparations and liqueurs. (Chang et al., 2016; Doymaz, 2010; Lewicki, 2006; Önal, 2019).

The above presented requirements resulting from the ways the dry products will be used shows that dehydration is a versatile method to design and create properties of the material needed in further processing and expected by consumers. However, to use drying efficiently a thorough knowledge of the process and processing variables is necessary (Lewicki, 2006).

I.2 State of water in food products

Fruit drying involves removing water in different forms as free water and bound water. Free water is located in the pores and the capillaries and which can easily be removed during the drying process while bound water is physically trapped within a restricted environment and therefore is hard to transport during food drying (Khan and Karim, 2017). The amount and manner of water removal effect on the change the structure of fruit and vegetables depending on type of bonding and, also determine the character of the reconstituted dried foods. Removal of free water does not the change the character of food products in both dried and rehydrated states. Higher energy and special procedures are required to remove bound water compare to free water (Barta, 2006).

The presence of water in foods is a key factor for product quality, consumability and acceptability. In dried fruit and vegetables, water content determines the degree of undesirable changes to occur as a result of the activity of microorganisms and enzymes or through non enzymatic chemical changes, therefore, the maximum achievable shelf life (Brennan, 2003; Sturm and Hensel, 2017). The influence of moisture on the stability of foods is known as important issue including two main factors: a) total moisture content of food products b) how much of that moisture is available to support the activity of microorganism and enzymes, and enable chemical changes to occur.

Water activity (a_w) concept is used to represent the water availability of foods, constituting a critical approach for assessing food stability (Oliveira et al.,2015; Kou et al., 1999; Rahman, 2010). This concept has an important role in the behaviour of food products during processing and storage. The generally accepted assumption was that a low water content is desirable, however more recent investigations have demonstrated that the optimum level of water activity is different for every food. The water activity level that is too high (> 0.6) may increase the risk of microbial spoilage of the product, whereas overdrying may cause a reduction of product quality through lipid oxidation, enzymatic and non-enzymatic activities and loss of valuable components (Brennan, 2003). A graph of moisture content as a function of water activity, with which it is in equilibrium, is known as a sorption isotherm. Such sorption isotherms may be constructed by

adsorption and desorption, which, in the case of many food results in two different curves, exhibiting hysteresis (Figure I.1).
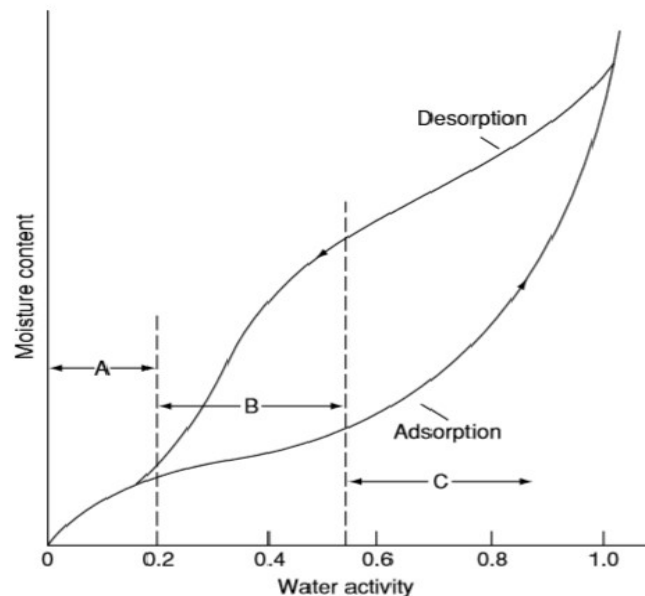
Figure I.1 *Adsorption and desorption isotherms, hysteresis (Brennan, 2003)*

A knowledge of the sorption isotherm characteristics of foods is of great importance from practical of view (Wolf et al., 1985). It may be useful for selection the drying procedure, prediction of drying times and dryability, the bindings of strength moisture and the shelf life of the fruit (Barta, 2006; Brennan, 2007).

I.3 Drying mechanism

The drying behaviour of food materials can be described by measuring the function of moisture content loss and versus drying time (Mujumdar,2006). In the air drying process, three major periods generally can be observed:

1. Transient early stage, during which the food product is heating up (transient period)
2. Constant or first period, in which moisture is comparatively easy to remove (constant rate period)
3. Falling or second period, in which moisture is bound or held within the solid matrix (falling rate period)

Typically, two falling rate periods are observed for both hygroscopic and nonhygroscopic solids. At the initial period of drying process, the temperature of food material reaches the temperature of wet thermometer. The temperature does not change in the constant rate period until reaching the critical moisture content. The moisture content at which the change from the first to second periods occurs is known as the critical moisture content.The temperature of food material increases in the falling rate period and becomes equal to the temperature of the drying air when drying stop. The first falling rate period is postulated to depend on both internal and external mass transfer rates, while the second falling rate period, during which drying is much slower, is to the solid-water interaction or glass-rubber transition. In the second falling rate period, the vapor pressure of the solid becomes equal to partial vapor pressure of the drying air and no longer further drying takes place. The limiting moisture content at this stage to which material can be dried under a given drying condition is referred to as the equilibrium moisture content (Rahman and Perera, 2007).

The drying curves of food material demonstrate a typical case where the moisture from the solid material evaporates first from the moisture layer on the surface and decreases continuously until water evaporates from the inside of the solid material. It can be seen in the figure that variations in the drying rated depend on time and moisture content of the fruit product. Drying rate can be presented as a function either of the drying period or moisture content of the material. Curves for the drying rate and drying flow rate can be divided into several parts. These parts are the result of the inner mechanism of drying and of changes occurring during drying. In the first step of drying is known as temperature equalization and moisture transport. In the next step, which is the constant rate period, there is a constant moisture flow to the surface, therefore, the surface is always wet. The average moisture measured at drying of surface is so called critical moisture content. Drying stop and the drying rate becomes equal to zero when then average moisture content reaches the equilibrium.
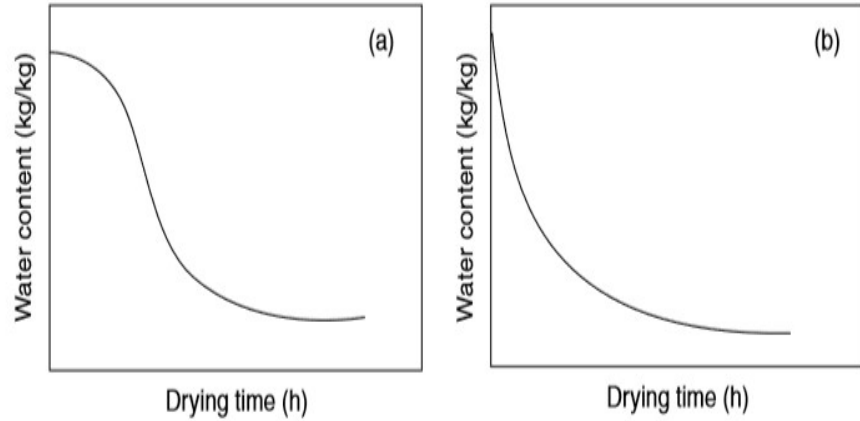
Figure I.2 *Typrical drying curves (water content versus drying time) (a) with a lag period, (b) without lag period (Rahman and Perera, 2007)*
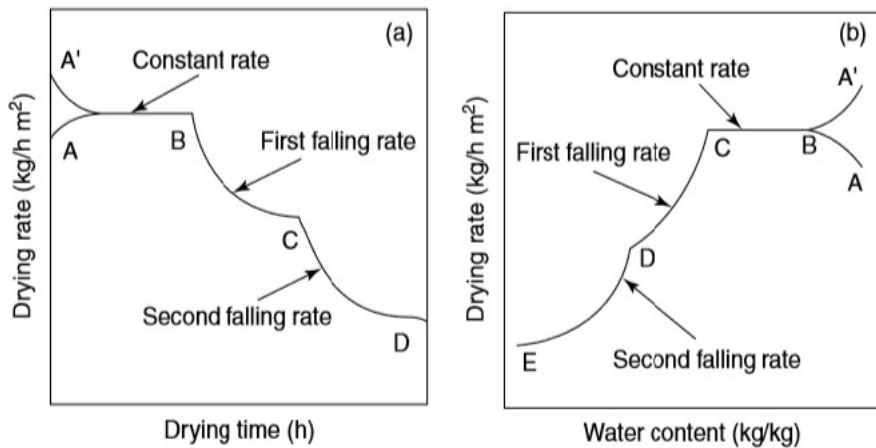
Figure I.3 *Typical drying rate curves: (a) drying rate versus drying time, (b) drying rate versus water content (Rahman and Perera, 2007)*

The drying behaviours depend on porosity, homogeneity, and hygroscopic properties of food materials. Also, dimensions and shape of the material being dried are the most important parameters to evaluate drying curves in the details.

It is necessary to know factors determining the quality of the finished product, which can help to establish the parameters of the drying procedure. The concept of optimum drying must include the concept of economy, or the optimal application of heat used for drying. The external factors influencing drying are the following: temperature, moisture content, flow rate, direction of drying air, and drying period. These factors must fit the

properties of the food material being dried (variety, water content, dimensions) and the methods of preparation. The most important factor is the temperature of the applied air.

I.4 Convective hot air drying

The process of drying food products is complex, involving coupled transient mechanisms of heat, mass and momentum transfer processes accompanied by physical, chemical and phase change transformations (Saberez, 2012). In convective drying of food products, two distinct transport mechanism occur simultaneously in opposing directions: a) heat transfer; from the drying medium (air) through to the food material, and b) water transfer; from the interior of the solid product to its surface and eventually transported away by a carrier gas. In hot air drying, the heat and mass transfer phenomena are generally affected by the physical properties of food products (moisture content, particle size, geometry), the physical arrangement food with air (crossflow, through flow, tray load), the physical properties of air (temperature, humidity, velocity), water concentration gradients and the design characteristics of drying equipment (Jayaraman and Da Gupta, 1995). A conceptual representation of the transport phenomena occurring during the convective air drying of a solid food product is demonstrated in Figure I.4.
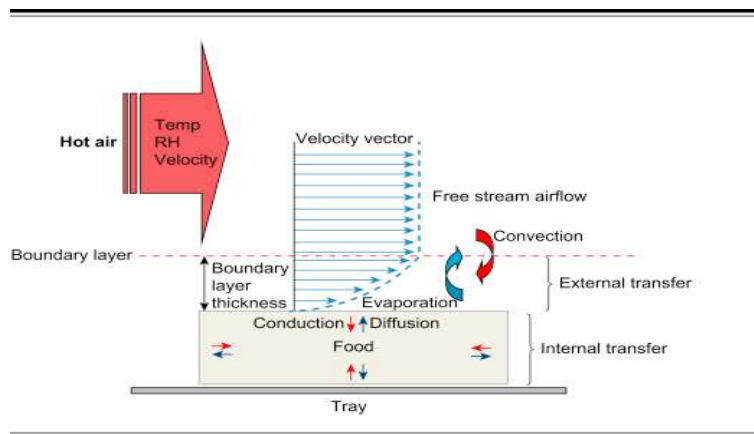
Figure I.4*A conceptual representation of convective air drying process of solid food product (Sabarez, 2016)*

Energy is required to generate a phase change of water from liquid to vapor (solid to vapor) in order to activate molecular movement. Hot air (drying medium) is employed both to supply heat by convection and, as the carrier gas, to take away to moisture using the motion of molecules. Heat is

supplied at the boundaries of the drying food product so that the heat must diffuse into the solid primarily by conduction.

The mass transfer phenomenon during drying may be controlled by either the rate of moisture diffusion (liquid or vapor) throughout the food matrix (internal transfer) or by the rate of moisture evaporation from the food product surface to the drying medium (external transfer). The internal mass transfer controlled drying process is affected by temperature and dominates once the rate of replenishment of moisture from the interior to the surface of the food products is slower than the external mass transfer rate. The moisture throughout the food product can migrate in several ways via a number of different mechanisms as liquid or vapor phase (Heldman and Hartel, 1997). The liquid transport mechanism involves capillary flow, surface diffusion and liquid diffusion. Vaporization in some cases can occur within the food product and hence water diffuses in the form of vapor through the food matrix, with the difference in vapor pressure as the driving force for moisture transfer. The differences in pressure among the drying medium and internal food structure (pressure, flow) and the differences in temperature between the surface and the interior flow (thermal flow) may also affect on the internal mobility of moisture. The moisture travels to the boundary of food product before it is then transported away by the carrier gas.

To summarize, in hot air drying process, heat is usually transferred by convection to the food surface and by conduction inside the food. Mechanisms involved in mass transport which are known as complex process, particularly in the case of those foods with an organized cellular structure. Moisture that evaporates inside the solid diffuses out as a vapor due a pressure gradient. Also, liquid water is usually transferred by diffusion due to water activity gradients. Unbound moisture in porous granular solid moves through the capillaries and intercies by a mechanism including surface tension. Across two sides of permselective membrane, the transport of water takes place by an osmotic pressure gradient promoted mechanism. In addition, since cell water loss involves the considerable volume reduction, pressure gradients also appear to chemical potential ones in mass transfer phenomena (Barrera et al., 2016).

Conventional hot air drying is a cost-effective preservation method and provides uniform distribution, hygiene and better dried product with optimal drying conditions (Adiletta et al., 2015a; Doymaz, 2010; Onwude et al., 2016; Zielinska and Markowski, 2016). On the other hand, the air drying process usually leads to physico-chemical (i.e. colour, chemical composition), structural (i.e. shrinkage, texture) and nutritional changes (i.e. loss of high added value components) which influence the overall quality of dried product and therefore the consumer acceptability (Önal et al., 2019).

I.5 Pre-treatments applied before drying of fruits and vegetables

In this context, drying combined with pre-treatments have been proposed with the aim of reducing various adverse changes. Foodstuff is usually subjected to pre-treatments prior to drying in order to increase moisture diffusion (Junqueira et al., 2017), prevent loss of colour and valuable bioactive compounds (Adiletta et al., 2016a; Da Costa Ribeiro et al., 2016; Olieveira et al., 2015), inactive enzymes (Deng et al., 2017), accelerate the drying process by reducing drying time (Kaymak-Ertekin, 2002), improve rehydration characteristics (Barrera et al., 2016; Vega-Gálvez et al., 2008), stabilization of food structure thereby protection functionality on cell walls (Lewicki, 1998).

A wide variety of pre-treatments have been utilized depending upon the type of food to be dried, food composition and its availability. Examples of pre-treatments are immersion in chemical solutions (Adiletta et al., 2018a; Junqueira et al., 2017; Vásquez- Parra et al., 2013), hot water blanching (Doymaz, 2010; Mdziniso et al., 2006), physical pre-treatments (Fratianni et al., 2018; Adiletta et al., 2015b,2016a; Di Matteo et al., 2000). Chemical pre-treatments, usually comprise sulphate and its derivatives, immersion in sodium chloride, potassium carbonate, organic acids or sugar, use of surface active agents and impregnation with biopolymers. Moreover, physical pre-treatments are known as thermal and non-thermal treatments: 1) Thermal treatments (i.e. hot water, steam, ohmic heat, microwave), 2) Non-thermal treatments (i.e. ultrasonic field, pulsed electric field,high hydrostatic pressure).

Carbohydrate/salt solutions may be used as an alternative pre-treatment for biological systems during the drying process. Among the carbohydrates, trehalose (non-reducing dissacharides) has recently received much attention owing to its many potential advantages in food industry such as high glass transition (Tg) of its aqueous solutions, protection of flavour and colour, improvement of reconstitutional properties of dried fruits and vegetables, inhibition of protein denaturation and higher nutritional content (Adiletta et al., 2016a, 201b; Aktas et al., 2007; Atarés et al., 2008; Betoret et al;2015., Xin et al., 2013). Trehalose confers to certain plant and animal cells the ability to survive dehydration and to restore activity soon after rehydration. For this reason, it has been proposed as an excipient during freeze drying of a variety of products in the pharmaceutical industry, and as an ingredient for dried and processed food (Patist and Zoerb, 2005). However, the mechanisms resulting in this unique behaviour of preservation (i.e. interaction between trehalose and cell membranes or proteins) are still under debate. With regards to salt solutions, sodium chloride applied to foodstuffs prior to drying has shown to contribute to the texture reinforcement and to obtain faster and better rehydration (Dermesonlouoglou et al., 2007; Lewicki, 1998).
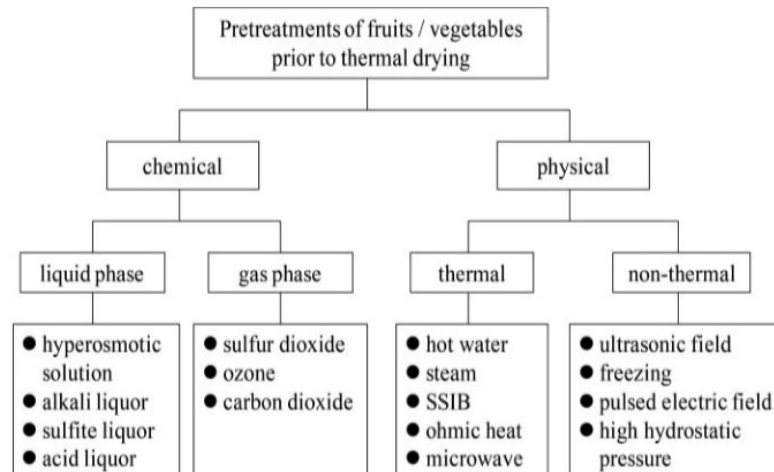
Figure I.5 *Pre-treatment methods of fruits and vegetables prior to the drying process (Deng et al., 2019)*

I.5.1 Some chemical pre-treatments

Sulfitation or sulfuring has been commonly used in the food industry to minimize darkening during drying and prevent quality loss during process and storage of foods. It is usually performed using sulfur dioxide gas or water-soluble sulfide salts for example, potassium metabisulfite ($K_2S_2O_5$) and sodium metabisulfite ($Na_2S_2O_5$) and sodium hydrogen sulfite ($NaHSO_3$). When SO_2 is absorbed into the fruit and vegetables, it is converted mainly to the bisulphate ion (Deng et al., 2019). The advantages of using the sulfitation treatment prior to drying have been reported by some authors: An acceleration of drying process through changing the cell membrane permeability (Lewicki, 1998), inactivation of enzymes (Negi and Roy, 2000), preservation colour and nutritive attributes (Krokida et al., 2000; Mir et al., 2009; Mujumdar, 2006), and improvement rehydration behaviour (Latapi and Barrett, 2006). Besides, among the sulfites, metabisulfite pre-treatment caused the total loss of ascorbic acid in apricots by leaching from the fruit into the sulfite solution during immersion treatment, which creates an undesirable flavour and soft texture (Garcia-Martinez et al., 2013).

Sulfites are being discarded and often forbidden by legislation. The most prominent problem of sulfite pre-treatment is the chemical residue in the food products, which can cause some allergenic (asthma) reactions like nettle rash, angio-neurotic edema and anaphylactic shock in ordinary individuals in sensitive people (Aydın and Gocmen, 2015; Kamiloglu et al., 2016). New standards for food additives by the Ministry of Health of the

People's Republic of China has increased the standard for use of sulphur treatments in food processing; the residue of sulfur dioxide, potassium metabisulphite, sodium metabisulphite, sodium sulphite in food products has been strictly limited to 0.2g/kg of dried vegetables, and 0.1 g/kg of dried fruits (Ministry of Health of the People's Republic of China, 2011). In addition, the Food and Drug Administration requires a label declaration on any food containing more than 10 ppm of sulfating agents since 1986, because of their alleged hazard to asthmatics (FDA 1986) (Deng et al., 2019). For their allergenic effect, consumers tend to avoid sulfites and thus as organic foods have become increasing popular.

Acid pre-treatments such as citric acid (Pao and Petracek, 1997, Zhu et al., 2007), lactic acid (Uyttendaele et al., 2010), or ascorbic acid (Santerre et al. 1988) may be an alternative pre-treatment method that can reduce the number of normal flora and pathogenic organisms (Chiewchan et al., 2010), accelerate the drying process (Hiranvarachat et al., 2011; Doymaz, 2010), inhibit the enzymatic browning (Langdon, 1987; Zhu et al., 2007), maintain the colour (Pan et al., 2008) and modify the texture (Deng et al., 2019).

The dipping alkaline emulsions (i.e. ethyl or methyl esters, sodium hydroxide, ethyl oleate solutions, potassium carbonate) can widely used as a dipping chemical pre-treatment to dissolve the wax layer of some fruits, berries, grapes, etc. enhance the permeability of the skin to moisture, facilitate moisture diffusion, increase the drying rate and improve the colour parameter of dried food samples (Corona et al., 2016; Doymaz and Pala, 2002; Doymaz and Altıner, 2012; Esmaiili et al., 2007). The dipping alkaline solutions such as KOH and NaOH solutions significantly enhanced weight loss rate of grape and plum (Tarhan, 2007). In addition, the residue of alkaline liquor in the dried products may cause food safety issues, how to deal with larger quantities of corrosive chemicals is a serious problem and do harm to human health. So alkaline dipping pre-treatment technology should be applied only if a major drying time reduction is needed (Adiletta et al., 2016b, Carranza-Concha et al., 2012).

Dipping of foodstuffs in sodium chloride solution (NaCl) prior to drying inhibits the browning reaction and controls for microbial growth but causes some dehydration of food tissue. The treatment with NaCl allowed to enhance the drying process and preserve better the colour during drying. The crystals of NaCl dissociate and form concentrated spots of Na and Cl ions during the rehydration. Solvation of the ions resulted in faster and better rehydration (Lewicki, 1998, 2006; Kaymak-Ertekin, 2002). Also, NaCl can be used before the drying with the aim of texture reinforcement of dried samples (Dermesonlouglou et al., 2007).

Osmotic dehydration widely used as a pre-treatment before the drying process in order to reduce energy consumption and improve food quality. It includes the immersion the food material in hypertonic solution (mainly sugar or salt) for several hours (Deng et al., 2019). Francisa et al. (2010)

used ultrasound for the dehydration of Malay apple by immersion in an osmotic solution at low temperature. The percentage of solid gain and water loss was significantly higher in ultrasound treated samples than untreated ones. However, osmotic dehydration pre-treatment resulted in a decrease of the hardness and increase of the darkness in dried mandarin samples (Pattanapa et al., 2010).

Immersion in $CaCl_2$ can increase the concentration of Ca^{+2} in the cell wall and modify the structural and mechanical properties of plant cellular matrix (Alonso et al., 1997; Dermesonlouglou et al., 2007).

Edible coating such as starch coating pre-treatment resulted in dehydrated pumpkin with better colour and significantly higher retention of reans-α-carotene and β-carotene than those without pre-treatment (Lago-Vanzela et al., 2013). Alginate-based edible coatings are one of a wide range of polysaccharides and proteins that have been used to improve antimicrobial and physical properties, as well as increase safety, quality and shelf life of diverse foodstuffs. On the other hand, edible coatings are fine layers of digestible material added to the food product. During a drying process, the application of these coatings may reduce the loss of aroma, colour and nutrients by reducing oxygen diffusion into the food, minimizing solute incorporation and maintaning the products' physical integrity.

I.5.2 Some physical pre-treatments

I.5.2.1 Blanching

Blanching is a common pre-treatment applied to fruits and vegetables prior to drying because of its simple equipment and easy operation. It usually employs water or steam at a constant temperature ranging from 70 to 100°C for a short period of time. Generally, hot water blanching has been used to enhance the drying rate, prevent quality deterioration by inactivating the enzymes, destroying microorganisms, improving the colour, flavour, texture, nutritional quality and overall acceptability of dried fruit and vegetables (Ando et al., 2016; Doymaz, 2010; Deng et al., 2016; Mukherjee and Chattopadhyay, 2007; Neves et al., 2012; Olieveira et al., 2015). On the other hand, water blanching using boiling water or close to boiling temperatures was described as unfavorable regarding some nutritional parameters of vegetables. The marked deterioration in food quality is also implicated in oxidase inactivation, especially with the development off-flavours, colour alteration and significant reductions in the levels of nutrients. The loss of nutrients during hot water blanching is caused mainly by leaching and diffusion. All water-soluble nutrients, such as vitamins, flavours, minerals, carbohydrates, sugars and proteins can leach out from plant tissues to the blanching water. Hot water blanching can also cause to degradation of some thermal sensitive substances such as ascorbic acid,

aroma and flavour compounds, as well sensory properties of dried products. Furthermore, hot water blanching had a negative influence on the texture properties of the cell structure of fruits and vegetables. The softening of the final textural properties of the product is due to both turgor loss caused by a cell membrane disruption and changes in cell wall polymers such loosening of the hemi-cellulose, cellulose and pectin networks (Greve et al., 1994; Xiao et al., 2017). In addition, hot water blanching produces large quantity of waste water and increases the pollutant charge. The discharged wastewater from hot water blanching include high concentrations of biochemical, soluble solids, and chemical oxygen demand due to leaching and dissolution of sugars, proteins, carbohydrates and water-soluble minerals (Xiao et al., 2017). According to environmentally friendly activities, the emerging and innovative blanching technologies have become important in food industry such as steam blanching, high humidity hot air impingement blanching, ohmic blanching and microwave blanching. The differences can be attributed to hot water blanching conditions such as a ration of material to water, blanching time, temperature, and product properties. Due to these reasons, blanching should be applied to foodstuffs under optimal conditions to minimize the quality alterations.

To minimize nutritional components and reduce the waste water, steam blanching systems were developed to replace hot water blanching. The steam blanching contributes to retention of most minerals and water-soluble components when compared with water blanching due to the negligible leaching effects. As compared to hot water blanching and steam blanching for equal treated time resulted in significantly higher ascorbic acid retention (Lin and Brewer, 2005). Also, Gamboa-Santos et al. (2012) found that the steam blanching provided the higher retention of vitamin C (81.2%) in carrot samples than those blanched in water at 60°C (1.3%). However, during the steam blanching process, softening of the tissue and undesirable quality changes often resulted a long heating time due to the lower heat transfer in steam blanching than hot water blanching, especially when the velocity of the steam is very slow. Steam blanching can significantly inactivate the biological enzyme due to high entalphy contents. Besides of oxidative inactivation, steam blanching enhanced to the increment of the contentof phytochemicals content in blueberry samples as a result of increased permeability of cellular membrane (Del et al., 2012).

In accordance with these findings, blanching may not appropriate method protection of the nutritional quality of fruits and vegetables submitted to drying process, also in some particular cases, it might even cause increased degradation of valuable nutrients. Quality deterioration is more seen when foodstuffs were pre-treated using water blanching close to boiling temperatures and the nutrients sensitive to heat are the most affected, such as vitamin C, polyphenols and antioxidant activity. However, steam and water blanching combined with chemical pre-treatments and low blanching

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temperatures enable to improve colour, nutritional attributes and sensory properties, after dehydration and during long-term storage (Oliveira et al., 2015).

I.5.2.2 Ultrasound pre-treatment

During the past few years, among the emerging and promising alternative technologies, ultrasound (US) technology has gained importance for food processing applications such as food drying of fruits and vegetables. The ultrasound technology can be applied using two different approaches in drying processes: ultrasound assisted drying (continuous way, during dehydration) or as a pre-treatment. Related to ultrasound pre-treatment can be implemented in two ways: 1) using an ultrasonic water bath or 2) a probe that transmits ultrasound waves into the liquid medium. All systems using power ultrasound in foods consist of three basic parts, namely, a generator, a transducer and a coupler. The samples should be immersed in liquid medium and submitted to ultrasonic waves for a period of time (Mothibe at al., 2011).

Ultrasound technology is based on mechanical waves at a frequency above the human hearing range (>18 kHz). Based on the range of frequency, ultrasound application can be divided into two groups as low intensity with low energy and high frequency (more than 100 kHz) and high intensity with high energy and low frequency 20 and 100 kHz. Low intensity ultrasound (more than 100 kHz) is widely used to control the quality of food material as a non-destructive method, provide information on pysico-chemical properties of food (composition, structure, physical state) and to monitor the changes during the food production process However, high intensity ultrasound application is applied to break down cellular structures in order to activate and prevent the physical and chemical alterationsin food material, which consequently leads to the intensification of heat and mass transfer during the different process. There are many high intensity applications in food processing such as cell disruption, microorganism inactivation drying, freezing, thawing, extraction, filtration (Dehghannya et al., 2019; Mothibe et al., 2011, Musielak et al., 2016,Nowacka and Wedzik, 2016).

In concern with ultrasound mechanisms, in general, two main physical phenomena occur during the ultrasound treatments in food applications: 1) 'sponge effect' and 2) 'cavitation'. In a solid medium, ultrasonic waves cause rapid alternating compressions and expansions of the food material cells, what leads to the bubble formation in both surrounding and sonicated samples which is called 'sponge effect'due to the similarity to squeezing and releasing a sponge. In the liquid medium,ultrasound application is based on the effect of mechanical waves which are mainly related to cavitation phenomenon. In the liquid medium, the sonication leads to cavitation, thus thousands of fast-moving microbubles generated during the ultrasound applications and which can explosively collapse. These effects lead to in fast

and very short pressure and temperature changes in the point, what result in changes of viscosity, surface tension and destroying cell walls. As a result of these changes, which may create the formation of micro channels. The phenomenon of cavitation is responsible for the creation of microscopic channels that reduce the diffusion of boundary layer and increase the mass transfer in food materials. From this viewpoint, these changes in food properties are affected by many factors such as liquid medium, ultrasound frequency and sonication time during the ultrasound applications (Dehghannya et al., 2019; Nowacka and Wedzik, 2016).
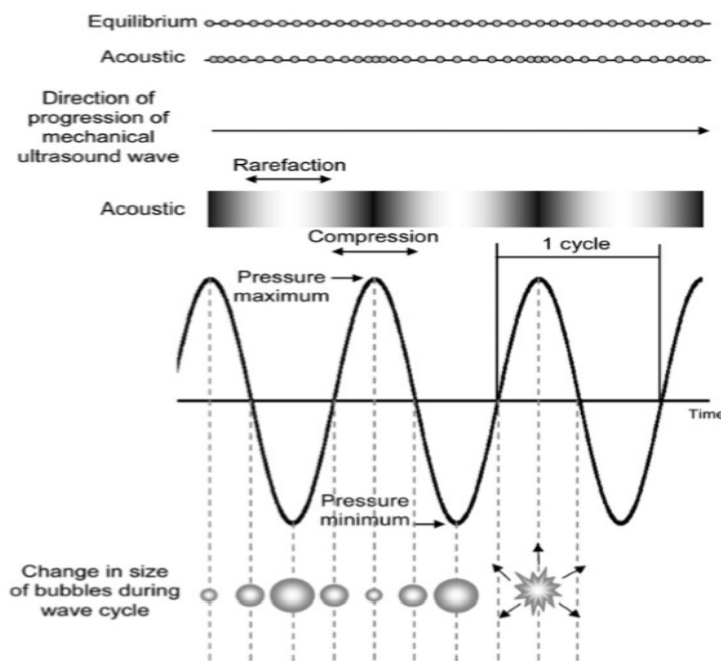
Figure I.6 *Ultrasonic Cavitation (Soria and Villamiel, 2010)*

The ultrasound technology is an innovative technique to improve the conventional drying, being also used to enhance the mass transfer process in drying. When ultrasound travels across a medium, it affects both internal and external resistances and could intensify the mass transfer process in the system (Yao, 2016). The effects of ultrasound pre-treatment on the drying process and the quality attributes of dried fruits and vegetables have been reported many authors (Dias da Silva et al., 2016; Fernandes et al., 2008; Goula et al., 2017; Magalhaes et al., 2017; Ricce et al., 2016). Ricce et al. (2016) investigated the effect of ultrasound technology as pre-treatment and the consequent enhancement of the drying and rehydration process of carrot slices. These authors stated that short times of pre-treatment are not enough to create the micro-channels. It caused the cell become bloated hindering the

drying process. However, when long ultrasound pre-treatments were applied, micro-channels were formed increasing the porosity of the samples and enhancing the drying process. Also, longer ultrasound pre-treatments increased the rehydration rate. It was corroborated that when the drying temperature was increased, the ultrasound effect was more effective. Ultrasound pre-treatment resulted in the lower rehydration rate for the rabbiteye blueberries. This behaviour may be explained by the higher degree of cell deformation and the most severe tissue collapse of resulted in the highest degree of cell deformation and the most severe tissue collapse of the surface layer (Stojanovic and Silva, 2006). Opalićet al. (2009) reported that prolonged ultrasound pre-treatment cause a decrease in total phenolics and flavonoids as well as in the antioxidant capacity of dried apples. Similar results found by Stojanovic and Silva (2006), high-frequency ultrasound had a negative influence on the anthocyanins and phenolic content of rabbiteye blueberries. Ultrasound pre-treatment had higher retention of carotenoids than blanching at 80°C for 3 min in hot air-dried carrots (Rawson et al., 2011). Dried melon pre-treated using ultrasound and vacuum combined found that lower total carotenoids loss, softer texture and better colour preservation. Sensory evaluation showed that this pre-treated samples had good acceptance (Dias da Silva et al., 2016). Deng and Zhao (2008), showed that the ultrasound pre-treatment increased hardness in air-dried and freeze-dried apples. This increased hardness is a positive attribute that leads to improved crispiness of dried fruit slices .

Therefore, the ultrasound technology revealed to be an advantageous alternative pre-treatment compared to conventional pre-treatments such as blanching. This novel technology can reduce drying time by the enhancement of drying processing rate or efficiency, which also lowers the cost of the process as a whole. In addition to these, it can improve all quality properties of dried quality products in terms of colour, nutritional and sensorial attributes, texture and rehydration. Nevertheless, the ultrasonic power, ultrasound intermittency, and exposure time need to be chosen giving due consideration to the thermal effects of ultrasound.

I.5.2.3 Microwave pre-treatment

Microwave processing is considered to be one the most novel thermal technologies for food processing. Typically, applications of microwaves for food processing include drying, extraction, pasteurization, blanching, tempering, sterilization, baking, etc. (Ekezie et al., 2017; Fathi Achachluei et al., 2019). The microwave processing can be used two ways in drying processes: Microwave-assisted food drying or as a pre-treatmentsubmitted prior to drying. Microwaves are electromagnetic waves with the frequency varies from 300 MHz and 300 GHz.The frequency of the microwave oven is defined to avoid interference with communications. Home microwave frequency is 2.45 GHz, while industrial microwave is 915 MHz or 2.45 GHz.

Microwave heating depends on the transformation of alternating electromagnetic field energy into thermal energy by affecting the polar molecules of food material. The most important characteristic of microwave heating is known as 'volumetric heating'.Due to the volumetric heating, food materials can absorb microwave directly and internally and convert it into heat (Vadivambal and Jayas, 2007). Thus, heat is generated within the food material by including two mechanisms as dipolar rotation and ionic conduction.The high heating rate of microwaves raises the product temperature rapidly, leading to high water vapour pressure to develop inside the food material, resulting in a very rapid transfer of water to the surface of the food material (Paengkanya et al., 2015).

In general, microwave related drying process have some advantages: speed of operation, shorter drying time, energy efficiency, cost of operation and improvement of food quality (Vadivambal and Jayas, 2007; Zhang et al., 2006).Microwave assisted drying technologies provided the higher drying rate, more porous structure with lower shrinkage, lower bulk density, higher rehydration capacity and lower energy consumption in comparison to traditional drying method. (Aydogdu et al., 2015, Duan et al., 2010, Guo et al., 2017, Horuz and Maskan, 2015).However, a high mass transport rate in microwave drying may result in low product quality or undesirable changes in food texture, leading to some product damage (Zhang et al., 2006).

On the other hand, there are a few studies related to the effect of microwave pre-treatment on the quality properties of dried fruits in the literature. Sabry et al. (2016) investigated the effect of microwave pre-treatment on the quality of carrot samples. The obtained results indicated that the using microwave pre-treatment before drying reduced the drying time in comparison to other methods. Non enzymatic browning was lower in microwave treated dried carrot samples, while the ascorbic acid and carotenoid content was higher than the others. Mothibe et al. (2014) stated that microwave pre-treatment had significant influence on the drying rate compared to drying without pre-treatment. Microwave pre-treated apple

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cubes had very low water activities. Also, microwave pre-treatment played an important role in the improvement of quality attributes of dried apples in terms of the highest values of rehydration capacity and hardness. However, in some regions of microwave treated apple samples (at 200 and 300 W), the cell wall breakdown is seen, causing an increase in interacellular spaces. Similar results were reported by Funebo et al. (2000) who showed that microwave heat treatment of apple disrupted the cell walls, and the conjunctions between cells became shorter. This confirmed that the cell structures were affected during different pre-treatments.

Non-uniformity temperature distribution is known one of the main disadvantages in microwave applications. The microwave pattern is responsible for creating a hot spot and cold spot, and hot spot is concentrated in a region where the electromagnetic field intensity is higher. This non-uniformity temperature distribution directly influences the overall quality of dried food material, as well as the consumer acceptability. Therefore, it is important point is to improve the uniformity during the microwave related drying applications.Uneven heating is an intrinsic characteristic of the microwave heating and achieving heating uniformity is a challenge in microwaving, which could be overcome by employing a feedback control loop to control the heating process. Therefore, it is important to know the temperature distribution of foods in the process of microwave heating.

I.6 Influence of drying pre-treatments on quality of dried products

Optimum freshness plays a crucial role in determining the quality and stability of dried food products; fresher the raw material, more stable and better is the quality of the product. Drying pre-treatments may have significant influence on the quality attributes of dried products to prevent or minimize the various adverse changes during the drying process. Generally, the quality attributes of dried foods can be grouped as physico-chemical and nutritional.

I.6.1 Physico-chemical quality attributes

Colour is one of the most important quality attributes of dried food products because it is a critical point in the acceptance or rejectance of those dried products by consumers. It underwent a serious deterioration during the air drying process, usually may cause by enzymatic and non-enzymatic browning reactions. The fresh fruit and vegetables contain high amount of polyphenols, polyphenol oxidase (PPO) and peroxidase (POD) which are prone to enzymatic browning. Non-enzymatic browning involves a wide number reactions such as the Maillard reaction, caramelisation, chemical

oxidation of phenols, and maderisation (Deng et al., 2019). Adiletta et al. (2016a) reported that the air drying temperature and used pre-treatment had an important role on colour changes of dried eggplants. The combined pre-treatment of trehalose and sodium chloride solution was capable to preserve the colour of eggplant samples during drying process up to 60°C. On the contrary, the change in colour of untreated samples could be mainly attributed to the enzymatic browning reaction that took place during the drying process. Dried melon pre-treated using ultrasound and vacuum obtained better colour preservation (Dias da Silva et al., 2016). A positive effect of ultrasound on colour changes was found in the case of convective and microwave-convective drying of carrot (Kroehnke et al., 2015) and green pepper (Szadzinska et al., 2015). Fratianni et al. (2018) evaluated the effect of the abrasive pre-treatment on the colour changes of goji berries at drying temperature of 50, 60 and 70°C. The findings indicated that the untreated samples were darker than treated ones and that pre-treatment seemed to reduce the browning reactions. Also, a significant colour change was found in treated goji samples dried at 70°C, demonstrating that pre-treatment was able in preserving the colour of goji berries up to 60°C. Krokida et al. (2000) observed that, sulfuring pre-treatment (sodium metabisulfite) had a protective effect on the enzymatic browning reactions and colour of convective dried apples, bananas and carrots. Osmotic dehydration can improve the colour of mango chips and banana slices, as the monosaccharide present in the plant tissue, which is a reactive substance for browning reaction, is leached out with simultaneous sucrose uptake during dehydration (Tabtiang et al., 2012; Zou et al., 2013). The use of ultrasound as a pre-treatment to onions contributed to significant colour changes. The longer sonication time was resulted in the highest colur differences. The greatest colour change for the onion samples can be explained by the formation of free radicals and sonochemicals as a result of cavitation, also the presence of enzymatic browning during the air drying process. In the case of blanching, the colour was better preserved as the contact between the samples and air was limited. The colour of fruit and vegetables is determined by natural colour compounds that can be oxidized during the pre-treatment. Also, the high temperature and the presence of oxygen have an impact on the accelerating the colur degradation.Enzymatic browning plays an important in colour change due to brown pigments formed from colourless polyphenols (Ren et al., 2018).

One of the most important physical changes is known as shrinkage: a volume reduction, coupled with the shape and porosity changes and hardness increase. Such phenomena could also be followed by surface cracking. Another negative effect of shrinkage, for instance, the reduction of rehydration capability of dried fruit and vegetables. Therefore, shrinkage has to be avoided because such physical changes contribute in general to decrease the quality perceived by the end consumer of dried products,

traditionally consumed fresh (Brasiello et al., 2013). Obvious exceptions represented by foods like dried plums and dates, usually eaten shrunken. Shrinkage occurs first on the surface of food products and then gradually moves to bottom as the drying time increases (Rahman and Perera, 2007). Junqueira et al. (2017) found that an increase in shrinkage with the removal moisture. When moisture removed from food, a pressure difference is created between the inside and outside, generating contracting stresses that lead to material shrinkage. At the same moisture content, the cape gooseberries pre-treated with combination of freezing and chemical pre-treatments with ethyl oleate/Na_2CO_3 promoted lower shrinkage than untreated samples. These results indicated that a positive effect on the maintenance of the structure of the cape gooseberry dried fruits. Pan et al. (2008) showed that the dipping pre-treatment with ascorbic and acid solutions improved shrinkage in freeze-dried banana samples during the drying process. On the other hand, osmotic dehydration also can has adverse effects on the drying rate of some food products, attributes to increase increased resistance to water flux caused by shrinkage and solute uptake (Deng et al., 2019; Nieto et al., 2001). Dehghannya et al. (2019) reported that at the beginning of the processing the shrinkage rate of potato samples was higher because of the higher moisture content of the samples. As the moisture content decreased, shrinkage rate also reduced. In general, shrinkage increased significantly compared to the control samples when then ultrasound treatment time increased. More moisture is removed during the drying process and as a result, shrinkage is increased. Because volume change and thus shrinkage were larger in ultrasound pre-treated potato samples than the control ones, the higher shrinkage of pre-treated potatoes continued throughout the process. The ultrasound pre-treatment with higher ultrasound time, destroy cell walls, change tissue structure, and thus create surface stress and crack in the tissue structure (Mothibe et al., 2014).

Food texture is affected by many factors such as moisture content, properties of food products (composition, variety or species, maturation or age) and sample dimensions. Texture is also dependent on the drying method, drying conditions (i.e. temperature and time) and pre-treatments applied prior to drying process (Guiné,2018; Rahman and Perera, 2007). Junqueira et al. (2017) evaluated the texture parameters for the fresh cape gooseberry samples and pre-treated dried cape gooseberry ones (pre-treatments: a) liquid nitrogen, b) slow freezing c) ethyl oleate/Na_2CO_3). Fresh samples had higher hardness values than the dried cape gooseberries. Between the dried fruits, the untreated and samples and those pre-treatment with ethyl oleate /Na_2CO_3 had the highest hardness values. The samples subjected to fast (liquid nitrogen) and slow freezing presented the softest textures. During the freezing, followed by the thawing, the cellular structure is affected by the size and the quantity of ice crystal. Liquid nitrogen (fast freezing) usually leads to the formation of numerous and smaller ice crystal,

while the slow freezing leads to the formation of fewer and larger ice crystals, both inducing historical damages.The dried samples (untreated and pre-treated) had higher adhesiveness is related to surface characteristics and depend on effect of adhesive and cohesive forces, the viscosity and viscoelastic properties and the cohesiveness indicate how the sample could be deformed before rupture. Lower hardness, adhesiveness and gumminess values were found with freezing pre-treatments. Adiletta et al. (2016) investigated the effect of rehydration process on changes in texture for fresh and rehydrated eggplants dried at 50 and 60°C. Many differences in firmness were observed: after rehydration process, the firmness had a huge increase up to 67 and 56%, respectively, for both untreated and treated samples with dipping in a solution of trehalose and NaCl compared to the fresh ones. Among the rehydrated eggplant samples, the firmness of the treated samples was much lower than the untreated dried ones indicating the higher elasticity of the pre-treated ones. No differences were found the treated rehydrated samples dried at 50 and 60°C. Dias da Silva et al. (2016) investigated the effect of different pre-treatments (ultrasound, vacuum and combination of ultrasound and vacuum) on the texture evaluation of melon slices at dried 60°C by convective air dryer. Hardness of melon samples decreased in comparison with the fresh ones after drying process, which indicates that the texture of dried melon become softer. Among the dehydrayed pre-treated melon samples, the ultrasound (US) and combination of ultrasound and vacuum (USCS) had a lower firmness and there were no significant differences among these samples. There were also no significant differences among melons pre-treated with sucrose using a vacuum and the dried fruits without pre-treatment. Deng and Zhao (2007) demonstrated that ultrasound pre-treatment increased hardness in air-dried and freeze-dried apples. This increment in hardness is a positive attribute that leads to improved crispiness of dried apple slices which is a main factor in final consumer acceptance. Duan et al. (2008) reported that sea cucumber samples subject to ultrasound pre-treatment prior to microwave freeze drying. Treated dried samples with ultrasound presented low hardness and higher springiness which implied good chewiness properties.

The structure of the food products is a very important quality factor influencing the water diffusion ability during the drying process. Gonzales-Fesler et al. (2008) remarked the blanching pre-treatment of apple tissue resulted in breakage of membranes and in great damage in cell walls after drying, which appeared with interruptions in many areas. Cell walls of calcium impregnated apples appeared overall less disrupted than in only blanched ones. This would indicate that Ca^{+2} penetration improved cell wall structure of apple tissue. Ramirez et al. (2011) evaluated the effect of four pre-treatments (immersion in boiling water, vacuum impregnation, freezing/thawing, and uni-axial compression) on apple microstructure. The quantitative structure analysis showed that freezing/thawing and

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compression pre-treatments caused more damage to the apple structure, yielding larger cell cavities in comparison to vacuum impregnation and immersion in boiling water and control. Moreover, the freezing/thawing and immersion in boiling water were described the most aggressive pre-treatments. During an inappropriate freezing condition in quality terms, usually cellular damage may occur due to ice crystal formation which break cell membrane and walls. Hence, the cells appeared irregular in shape and tissue distortion was observed due to the growing of ice crystals with cell separation. In case of vacuum impregnation, this pre-treatment had the lowest values of microstructural features for apple tissue in terms of cell cavity size. These results showed that the presence of calcium in the matrix helps maintain the integrity of the cell wall due to cross-linking of pectic polymers and increase the degree of cell-to-cell contact. As a consequence of waxy solubilization by ethyl oleate, a non-uniform distribution of the waxy components on the grape surface resulted. After drying process, the presence of cracks on the peel surface of ethyl oleate pre-treated grapes appeared which are not evident for those untreated pre-treated by abrasion. These authors explained that the micro-fissures in the grape peel were formed by using carbonate solution and ethyl oleate solution. In addition, the increased drying rate by chemical pre-treatments was due to the formation of micropores on the surface of peel. Nowacka and Wedzik (2016) investigated the influence ultrasound pre-treatments on the carrot tissue microstructure which were subjected to ultrasound with a frequency of 21 and 35 kHz at the time of 10, 20 and 30 min. It was found that ultrasound pre-treatment had a significant effect on the structural characteristics of carrot tissue.The untreated carrot tissue had a smaller cavities, a high density and irregular shape. In examined samples after sonification, the cells of carrot tissue were distorted, numerous of them were damaged or merged together forming larger spaces. These changes were noticed especially after 30 min of application of ultrasound at a frequency of 21 kHz and after 20 and 30 min of pre-treatment with ultrasound at a frequency of 35 kHz. The alteration of cell structures was greatest when the longest ultrasound treatment time was applied. The ultrasound pre-treatment is known as the physical pre-treatment methods which may cause changes in the structure of carrot tissue. These changes in microstructure were probably caused by a sponge effect ultrasound pre-treatment. The cavitation affected enhance mass transport phenomena and also may influence on the cellular structure of carrot.

The next physical parameter describing the dried food is the rehydration compliance. Rehydration is an important property used for understanding the quality of dried fruit and vegetables. It can be considered as a measure of the physical and chemical changes that occurred in the material during the drying process (Aral and Beşe, 2016). If pre-drying treatments and drying process would not induce any changes in the food products, rehydration could be treated as a process reversible to dehydration. Usually, most of the

changes are irreversible and rehydration can not be considered simply as a reversible to dehydration. Rehydration capacity is affected significantly by drying conditions, pre-treatments applied before the drying and textural characteristics of dried food products. Drying can diminish the osmotic properties of cell walls; as a result, an increase in water absorption and volume occurs due to the swelling of hydrophilic materials such as starch, cellulose, and pectic materials (Vega Galvez et al., 2009; Lewicki, 1998). Doymaz (2012) stated that rehydration ratios of the blanched persimmon slices were higher than those of control ones dried at 50, 60 and 70°C, which means the structural damage and cell shrinkage occurred less during the drying process. Also, rehydration experiments showed that the rehydration ratios of blanched and control samples at 60°C were higher than those of dried at other temperatures. An increase in temperature above 60°C had an adverse effect on the final rehydration ratio value, which decreased with increasing drying temperature. This may be indicative of a change in the product induced by higher temperature and maybe a loss of solid during the rehydration process. The cape gooseberry samples pre-treated with ethyl oleate and Na_2CO_3 showed that the highest rehydration capacity value. This is likely due to the rapid drying of pre-treated these samples (Junqueira et al., 2017). Similar results reported by Adiletta et al. (2016b) during the rehydration of grapes subjected to alkaline ethyl oleate. They also found higher rehydration capacity in chemical pre-treated grape samples in comparison with untreated ones. The reduced processing time when pre-treatment is employed decreases the cellular damage, promoting greater rehydration capacity and lower shrinkage (Khawas et al., 2015). Zhao et al. (2016) reported that 15% ethanol dipping pre-treatment improved the rehydration ration of dried eggplant slices by 26.74%, compared to untreated ones. Ricce et al. (2016) found that the rehydration of carrot slices subjected to ultrasonic pre-treatment at both 30 and 60 min was greater compared to untreated samples, which can be attributed to higher porosity, and micro-channels formation. This enabled the better entrance of water during the rehydration process. The rehydration rate of carrot slices dried at 60°C (with no differences between 30 min and 60 min of ultrasound pre-treatment) is higher than the ones dried at 40°C (with differences between 30 min and 60 min of ultrasound pre-treatment). Schössler et al. (2012) obtained that the ultrasound did not influence rehydration of freeze-dried red pepper. Moreover, rehydration capacity of convectional and hybrid dried sour cherries increased with the increase in air drying temperature and microwave power. This trend can be due to the higher air temperature and microwave power which caused the tissue and cell damage. This increase could be due to the water retention in the spaces created by the damaged cells. Also, microwave energy caused to intercellular gaps which could absorb a high quantity of water during rehydration and cause an increase in rehydration capacity (Horuz et al., 2017).

I.*6.2 Nutritonal quality attributes*

Over the last decades, increased attention has been given to the concerns with regard to the prevention or minimization of quality degradation of fruits and vegetables during drying. These food products contain an extensive collection of phytochemicals, for instance, vitamins, minerals, antioxidants, pigments and other bioactive compounds, in concern with health benefits. However, phytochemicals usually undergo pronounced degradation during drying, since they are sensitive to heat, light and oxygen. In addition, changes in the nutritional value of dried foods may be due to pre-treatments, drying methods and conditions (i.e. temperature, time, velocity) or storage conditions (after drying). Colour changes are also often monitored because they can be directly related to the retention of pigment nutrients such as carotenoids, chlorophylls, phenols, flavonoids and betalains (Guiné, 2018; Oliveira et al., 2015). Adiletta et al. (2016) observed that treated eggplants by dipping in a solution of trehalose and NaCl had higher total phenolic content than untreated ones dried at 50 and 60°C. There was no significant difference in total phenolic content between the untreated and treated samples dried at 70°C; this result indicated that the temperature of 70°C has damaged the nutritional composition of both eggplant samples with and without pre-treatment. The positive effect of this pre-treatment on the phenolic content of the eggplant samples during drying process up to 60°C could be seen. Junqueira et al. (2017) remarked the drying process (air drying temperature: 60°C) resulted in ascorbic acid losses with ascorbic acid retention ranging from 23 to 46% in dried cape gooseberry samples compared to the fresh ones. A reduction in this vitamin content during the drying as expected because the exposure to light, oxygen, and heat accelerates the oxidation of this compound. Lower ascorbic acid retention was found in untreated dried samples (22.64%) while chemical pre-treatment resulted in the highest ascorbic acid retention (46.20%). This effect was due to shorter drying time of chemical pre-treatment (ethyl oleate) which improved the ascorbic acid retention compared to the freezing treatments and untreated samples. The content of α-carotene, β-carotene and lutein in blanched dried carrots were 51%, 76% and 87% higher than nonblanched samples, due to the inactivation of peroxidase and lipoxidase activity (Lavelli et al., 2007). Tao et al. (2019) evaluated the effect of water blanching pre-treatment, surface contacting ultrasound-assisted air drying and their combinations on the quality changes of white cabbage. Among blanched samples, cabbages dried by air alone contained significantly higher vitamin C content than cabbages dried with the assistance of sonification. Despite that sonication resulted in shorter drying time contacting circulating hot air, it may oxidize ascorbic acid in white cabbage samples. Water blanching pre-treatment preserved the vitamin C content in dehydrated cabbages. Furthermore, both blanching and drying resulted in the decrease of total phenolic content in cabbage samples. The loss of phenolics maybe due

to the leaching of phenolic into cooking water. Meanwhile, total phenolic content in dried cabbages were all lower than in fresh ones. However, there was no clear influence of contacting on the retention of phenolics in dried cabbages. The influence of an abrasive physical pre-treatment and drying temperatures at 50, 60 and 70°C on changes in carotenoids of goji fruits was studied by Fratianni et al. (2018). Small losses of total carotenoids were found after drying 15% and 20% for abrasive treated and untreated samples, respectively (p≤0.05), with no significant differences between the drying temperatures (p>0.05). Sabry et al. (2016) showed that, microwave pre-treatment decreased the total carotenoid content of the carrot slices dried 60°C. Increasing the microwave treatment time to 10 min caused a reduction in the carotenoid content of carrot to 32.08, 31.55, 30.77 and 30.28 (mg/100g) on dry weight at 90, 180, 270 and 360 watt. Microwave power and time had significant influence on the retention total carotenoid content of dried carrot slices. Gamboa-Santos et al. (2014) analyzed vitamin C retention during convective drying of strawberries. It was found that although overall ascorbic acid retention after drying was high, the ultrasound treatment resulted in slightly higher loss of ascorbic acid. Kroehnke et al. (2018) investigated the retention of carotenoids after convective and microwave-convective drying of carrots, with and without ultrasound enhancement. They stated that the ultrasound application positively affected retention in carrot samples being dried convectively and could also improve carotenoid retention in microwave-convective dried carrot. Tao et al. (2016) reported that ultrasound-treated mulberry leaves dried at 60°C had the highest phenolic content than control ones. Also, the antioxidant activity of ultrasound- treated dried mulberry leaves was slightly higher than those of samples without ultrasound pre-treatment. These findings may be explained by the reason that shorter drying time after ultrasound treatment and lower drying temperature (≤ 60°C) can preserve phenolic compounds and antioxidant activity in mulberry leaves. Amami et al. (2017) demonstrated that total phenolic content (TPC) levels were generally decreased in strawberry samples as compared to the fresh ones after drying process. The highest total phenolic content was found in strawberries dried by air drying at 50°C (AD 50°C) and combination of ultrasound assisted osmotic dehydration pre-treatment and convective air drying at 40°C (UOD-AD 40°C) and was 38% lower than those fresh ones. The increase in total phenolic content for UOD-AD 40°C may be relevant to an increased extractability of some of the antioxidant components following ultrasound application who can give rise to pores in strawberry tissue and, consequently, improve the extraction of polyphenols during sample preparation. Žlabur et al. (2019) observed that the among dried honeyberry fruits, the highest vitamin C content was in ultrasound pre-treated samples dried by vacuum at 60°C while the lowest value was in samples dried by conduction at 60°C. Also, the highest total phenol, total flavonoid and

anthocyanin content were obtained honeyberries after applying ultrasound pre-treatment and vacuum dried at 40°C. In this sense both ultrasound pre-treatment and vacuum had a significant positive effect on reducing the degradation of thenutritional properties of honeyberry fruits.

Volatile compounds significantly contribute to the aroma, one the foremost important quality aspect of the vegetables, being determinant regarding the consumer choice and acceptance (Olieveira et al., 2015). The drying process may generate flavour or releases flavour from the food products. In addition, drying process usually changes the composition volatile by evaporating most volatiles and forming new volatile odor compounds by chemical reactions. Such changes in volatile compounds may affect the aroma of fresh fruit and vegetables after drying process, such as off-flavors may be produced in dried products when higher drying temperatures are applied (Rahman and Perera, 2007). Li et al. (2010) found that more volatiles were lost of carrot samples at high temperatures after microwave drying. The intensive volatiles of carrots were mainly lost in the early stages, while charring occurred mainly in the middle drying stages. Best product quality was obtained at drying temperature of 60°C. Inserra et al. (2017) evaluated the influence of sulphuring pre-treatment on the dried apricot fruits.Sulphuring treatment significantly affected the aroma compound profile of dried apricots. In general, the levels of aroma compounds in sulphured apricots were significantly lower than those of unsulphured ones. They stated that, the sun drying process without sulphur pre-treatment resulted in an increase in aroma concentration and formation of new aroma compounds by autoxidation and the Maillard reaction. Cheng et al. (2017) found that carbon dioxide (CO_2) caused an accumulation of acetaldehyde and ethanol, and changed the aroma composition of dried Chinese jujube samples.

ChapterII
Aims of thesis

The main aims of this research are:

- ✓ to investigate the effect of different pre-treatments (i.e. innovative and natural dipping pre-treatments) and physical pre-treatments (i.e. microwave and ultrasound applications)and drying process conditions on the drying behavior and quality properties of selected dried food products (i.e. colour, shrinkage, total phenolic, antioxidant activity, microstructure, texture, aroma, preliminary sensorial evaluation and rehydration kinetics etc.)
- ✓ to optimize the used pre-treatments'conditions and drying process conditions for obtaining the high quality dried products
- ✓ to develop new and appropriate mathematical models
- ✓ to valorize different agricultural products for both producers and consumers
- ✓ to obtain healthy-nutritive dried snacks with low calorie after drying process

Chapter III
Materials and Methods

III.1 Raw material definition: 'Annurca' apple

The 'Annurca' apple (*Malus* x *domestica* Borkh. cv Annurca Rossa del Sud) is the traditional cultivar of Southern Italy, especially, in the Campania Region. An official designation of Protected Geographic Indication (PGI) from the European Council was acquired (Commission regulation (EC) No. 417/2006) about the geographical origin, authenticity, quality of 'Annurca' apple and its specific cultivation area (D'Abrosca et al., 2017). Among the other and known apple varieties, 'Annurca' apple is characterized by its own typical and specific properties such as typical reddening treatment, crispy flesh, pleasant flavour, and acidic taste because of its high acid/sugar ratio (Fratianni et al., 2007). 'Annurca' apple is one of the major sources of bioactive compounds such as polyphenols, flavonoids and anthocyanins which high content significantly contributes to its colour, taste, aroma and nutritional values.

III.1.1 Raw material preparation

'Annurca' apples (*Malus* x *domestica* Borkh. cv Annurca Rossa del Sud) were purchased from local market in Campania Region, Italy, after typical treatment to obtain redness. In details after harvest, the apples are put on a straw layer and daily sprayed with water. When the exposed surface of fruit becomes red, it is turned so as to redden the opposite side. The reddening treatment is extended for 20-30 days, depending on weather conditions and type of fruit (Lo Scalzo et al., 2001).

Freshness, uniform size and absence of any mechanical damage were used as the selection criteria for the samples. The samples were stored in a refrigerator at +4°C until analysis. Prior to drying the apples were washed with tap water and peeled by using knife. Cylindrical slabs with diameter of

30 ± 0.22 mm and thickness of 5 ± 0.01 mm were prepared using several raw materials. The average moisture content of fresh apples was determined according tothe AOAC official method (AOAC, 1997) and was found to be as 83.85% ± 1.44 on the wet basis (5.19 kg water/kg db).

Two kinds of samples were compared in this study: untreated apple slabs (UTR) and apple slabs after pre-treatment (TR). Specifically, after cutting some apple slabs were immediately immersed in dipping solution, which consists of a carbohydrate/salt solution containing 0.8% trehalose, 0.1% NaCl and 1.0% sucrose, as reported by Albanese et al. (2007) with some modifications: dipping at 25°C and for 15 min.

(a)   (b)

Figure III.1. *'Annurca' apple (a) and 'Annurca' apple slab (b)*

III.1.2 Drying experiments

Drying experiments of these two types of samples: untreated (UTR) and treated (TR) apple slabs were carried out in a convective dryer (Zanussi FCV/E6L3) The dryer consists of a stainless steel drying chamber with dimensions of 86 cm × 86 cm × 76 cm equipped with an electric heater to heat the air, a centrifugal fan driven by a 0.19- kW motor to supply the air flow and circulate the air flow and an escape vapor was placed on the top of oven (Figure III.2).

The treated and untreated apple slabs were put on a tray in the convectional drier, and dried at four different temperatures 50, 55, 60 and 65°C at 2.3 m/s centrifugal fixed air velocity until the constant mass was reached (about 0.04 kg water/kg db). The air-drying temperatures were chosen according to the literature (Doymaz, 2010, Vega-Galvez et al., 2012)

For drying kinetics, the weight of three slabs for each type was continuously recorded by means of a weight sensor (Phidgets INC., Canada). This sensor (Figure III.3) is a load cell made of a transducer that converts mechanical force into electrical signals. The weight loss of the samples was recorded online every 5 min. The results were reported in terms of M_t/M_0 vs time (min), where M_t was the moisture content (kg water/kg db) at a given

drying time and M_0 was its initial value. Drying experiments were repeated for each temperature in three sets independently, each set having three replicates and the average were reported with a standard deviation (18 experiments totally).
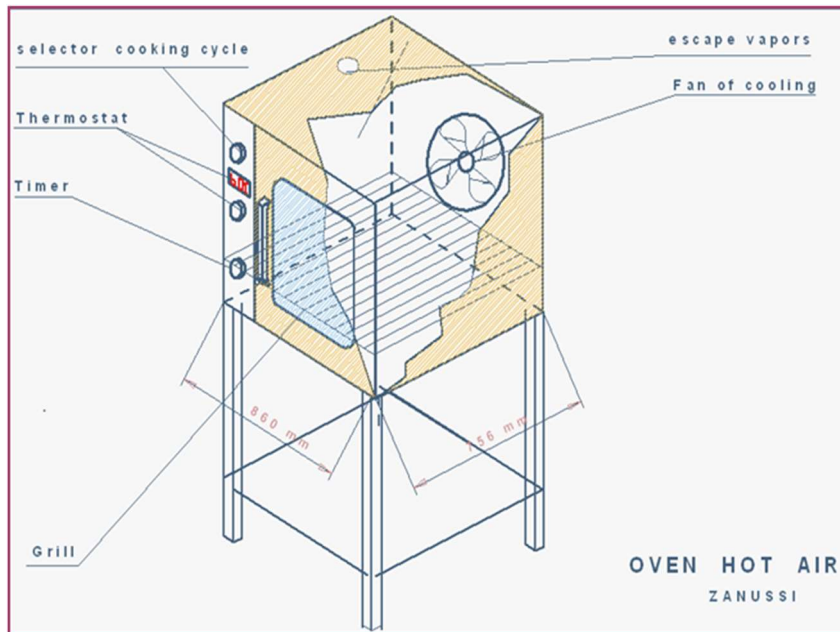
Figure III.2 *Convective dryer – oven (ZanussiFCV/E6L3)*
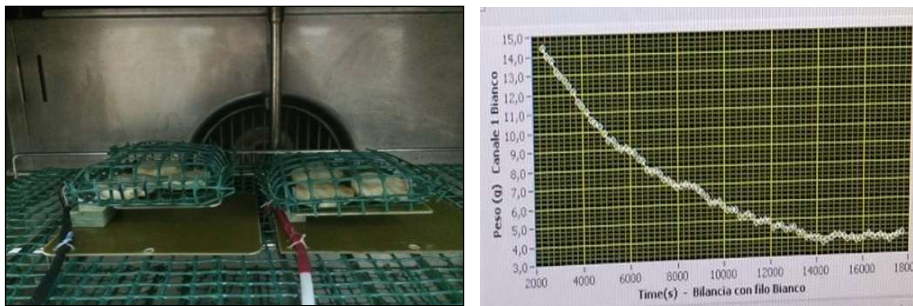
Figure III.3 *Balance sensor*

Chapter III

III.1.3 Mathematical modelling

Drying is a very complex process characterized by two phenomena occurring simultaneously:

- A heat transport phenomenon, characterized by a heat flow coming from the environment towards the food matrix, which causes waterevaporation.
- A material transport of water that moves through the solid matrix (in liquid form or vapour form) towards the environment.

Moreover, water removal causes structural changes of the matrix, both at microscopic (porous) and macroscopic (shrinkage) levels which have to be taken into account.

The mathematical model was obtained from the following simplified hypotheses:

- Material transport is essentially through the base of slab;
- the shape and size characteristic (thickness) is constant;
- the material is homogenous and isotropic;
- the initial moisture content of the samples is uniform (C_0);
- the concentration of water vapor in the surrounding environment is negligible ($C\infty \rightarrow 0$);
- energy transport is much faster than material transport.

The following equation describes the evolution of water content in the material:

$$\frac{\partial c}{\partial t} + \frac{\partial uc}{\partial x} = \frac{\partial}{\partial x}\left(D\frac{\partial c}{\partial x}\right) \qquad (1)$$

where:
c is the dimensionless water content in the differential volume
t is the temporal coordinate (s)
x is the spatial coordinate along which material transport takes place (cm)
D is the effective coefficient diffusion (cm^2/s)

The parameter *u* in eq (2) is the shrinkage velocity, here introduced to take into account shrinkage phenomena (Carslaw and Jaeger,1959). According to Brasiello et al. 2013, 2017, it is possible to write this term as a function of the local dimensional water content:

$$u = u_0 \frac{\partial c}{\partial x} \tag{2}$$

where:
u_0 is the shrinkage velocity coefficient (cm/s).

The initial and boundary conditions (uniform initial water content profiles are imposed):

$$\text{I.C.} \, t = 0 \quad c(x,0) = 1 \quad x = \left[-\frac{L}{2} ; \frac{L}{2} \right] \tag{3}$$

$$\text{B.C.}_1: x = 0 \quad \frac{\partial c}{\partial x} = 0 \tag{4}$$

$$\text{B.C.}_2: x = \frac{L}{2} \quad \frac{\partial c}{\partial x}\bigg|_{x=\frac{L}{2}} = -k_m c\big|_{x=\frac{L}{2}}$$

(5)

where:
L is the size of sample;
k_m is the overall transport coefficient

The I.C.(initial conditions) is related to the assumption of uniform and constant initial distribution of water content, B.C.$_2$(boundary condition) represents the interface condition.

Model parameters were estimated by the non-linear regression method using Matlab Software. For validation purpose normalized residuals and confidence intervals of the parameters were also computed.

III.1.4 Quality analysis

III.1.4.1 Water activity

The water activity of each sample was determined using a water activity meter (Testo 650, Testo Inc., USA) at 25°C. Approximately 3 gr of each sample was put in the sample holder. After equilibration, a_w values were recorded. Each measurement was done in triplicate.

III.1.4.2 Surface colour measurement

Surface colour measurements of fresh (UTR and TR),dried (UTR and TR) apple slabsand rehydrated (UTR and TR) ones at 30 and 70°C were

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determined through a colourimeter (Chroma Meter II Reflectance CR-300 triple flash mode aperture 10 mm Minolta, Japan).The instrument was standardized before the measurements by using a white ceramic plate. In order to analyze the colour change of all samples (fresh and dried), CIE lab colour values (L*, a* and b*) were measured and the average values were calculated for each samples. The lightness value (L*) indicates the darkness/lightness of the sample, a* index represents greenness/redness of the sample and b* index represents blueness/yellowness.

White index (WI) represents the degree of whiteness of fruits and vegetables and was calculated as follows (Adiletta et al., 2016b):

$$WI = 100 - \sqrt{(100 - L*^2) + (a*^2) + (b*^2)} \qquad (6)$$

Total colour difference (ΔE) was determined using eq. (7) (Vega-Gálvez et al., 2012).

$$\Delta E = \sqrt[2]{(\Delta L*)^2 + (\Delta a*)^2 + (\Delta b*)^2} \qquad (7)$$

The fresh apple slices (untreated and treated) were used as a reference, and higher values of ΔE indicate more colour changes from the reference material.

III.1.4.3 Determination of total phenolic content

Fresh and dried apple samples were extracted according to D'Abrosca et al. (2017) with some modifications. Fresh samples (5 g ± 0.01)and each dried and rehydrated samples (3 g ± 0.01) were cut into small pieces and 10 mL of methanol was added to all samples (80% v/v) (CHROMASOLV®, for HPLC, ≥99.9, Sigma-Aldrich, USA). The mixture was homogenized with Ultraturrax at 10 rpm and then stirred for 10 min by a vortex. Supernatant was filtered through a Whatmann No:2 filter paper. All extractions were carried out in triplicate.

The total phenolic content in fresh and dried apple (as treated and untreated) extracts were determined by Folin-Ciocalteu colourimetric method (Singleton et al., 1999). The apple extracts were oxidized by the Folin – Ciocalteu reagent in sodium carbonate solution. The absorbance of the reaction mixture was measured at 765 nm after 2 h in dark at room temperature by using UV-Vis spectrophotometer (Lambda Bio 40; Perkin Elmer, Waltham, MA, USA). The results were expressed as milligrams of gallic acid equivalents per 100 g of dry basis (mg of GAE / 100 g db).

III.1.4.4DPPH radical scavenging activity

The extracts obtained with the procedure described in III.1.4.2paragraph were also used to determine the antioxidant activity. The total antioxidant activity of fresh and dried apples was measured by the DPPH radical scavenging method (Adiletta et al., 2018b). Different volumes of the extracts were mixed with 3.5 mL of 6×10^{-5} M of DPPH methanol solution in cuvettes. The reaction mixture was shaken properly and kept for 30 min at room temperature in the dark. The change of colour from purple to yellow indicated the progress of reaction due to the reduction of 1,1-diphenyl-2-picrylhydrazyl (DPPH) solution complex, as recorded by using UV-Vis spectrophotometer at 517 nm (Lambda Bio 40; PerkinElmer, Waltham, MA, USA). Methanol was used as the blank and the control sample was prepared without adding any extract. Percentage of inhibition of DPPH radical was calculated as the following equation (Deng et al., 2018; Wang et al., 2018):

$$\% \text{ Antioxidant Activity} = \frac{\text{Abs}_{control} - \text{Abs}_{sample}}{\text{Abs}_{control}} \times 100 \tag{8}$$

where

$\text{Abs}_{control}=$ the absorbance of control
$\text{Abs}_{sample}=$ the absorbance of sample

The results were expressed as the EC_{50} value, which was defined as the sample concentration (mg mL^{-1}) required to inhibit 50% of the DPPH radical scavenging activity. EC_{50} was determined from a graph of antioxidant capacity (%) versus extract concentration (mg mL^{-1} sample).

III.1.4.5Shrinkage evolution

The initial volume of untreated (UTR) and treated (TR)apples (V_0) was calculated for each sample (10 slabs)by measuring the diameter and thickness by using digital Vernier calliper (0.01 m accuracy) (Adiletta et al., 2016a). At given times during the drying experiments, the dimensions such as diameter and thickness of the same slabs were measured and the volume (V_t) was calculated. In order to reduce the measurement error during drying, both slab dimensions were measured at different positions, and specifically, the diameter in four positions symmetrically placed along the perimeter of the slab and the thickness in eight positions (Ponkham et al., 2012).For the evaluation of the shrinkage during drying, the volume ratio (V_t/V_0) was reported as a function of the relative moisture ratio (M_t/M_0).
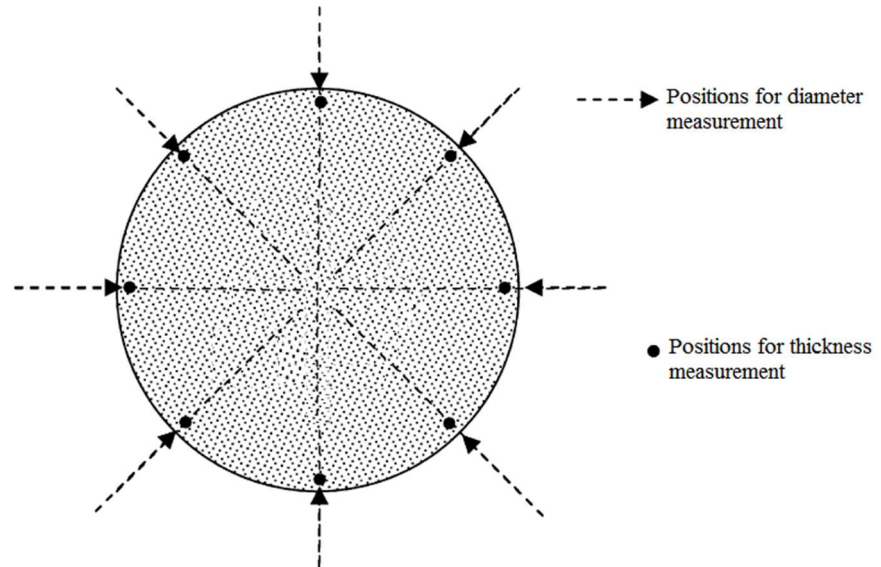
Figure III.4 *Positions for diameter and thickness measurements*

III.1.4.6 Microstructure analysis

Images of dried apple were captured using a scanning electron microscope (TESCAN MIRA3). The area observed was obtained by cutting the slabs along the thickness (longitudinal direction) and then coated with a very thin layer of gold under high vacuum conditions before being examined with the microscope. Micrographs were taken at 10kV accelerating voltage, 21 mm working distance and 300 × magnification (Proietti et al., 2018).

III.1.4.7 Preliminary sensory evaluation

Thesensory evaluation of untreated and treated dried apple slabs was carried out by 15 untrained panelists recruited at the University of Salerno. A hedonic scale of five points which ranged from 1 "dislike extremely" to 5 "like extremely" with 3 as "neither like nor dislike" was used during sensory evaluation for the following quality attributes: colour, flavour, taste, sweetness, acidity and overall acceptability. The panelists' scores were calculated for each sensory attribute and average values were reported with the standard deviation. Acceptability of dried apples was considered when the average score is equal or greater than "3.0" (Moreira et al., 2015).

III.1.4.8 Rehydration experiments

III.1.4.8.1 Rehydration capacity and rehydration indices

Apple slabs dried at 50, 55, 60, and 65°C were rehydrated in distilled water at the specified rehydration temperatures (30°C and 70°C) by a using water bath to estimate their rehydration capacities (Barrera et al., 2016; Doymaz, 2010). The approximate ratio between the weight of dried fruits and water was kept 1:100. Samples were weighted every 15 min in the initial phase of rehydration process (up to 120 min) and then every 30 min. At these fixed time, samples were removed from rehydration media, dried gently with tissue paper and weighted by using an electronic balance.

The total rehydration time at 30°C and 70°C for untreated and treated dried samples was determined as 270 and 210 min, respectively. This process was replicated in triplicate for each dried sample. The rehydration capacity described as percentage water gain (Adiletta et al., 2016a), was calculated from the sample weight difference before and after the rehydration as follows:

Weight gain (%) =
$$\frac{\text{(weight of rehydrated samples-weight of dried samples)}}{\text{(weight of dried samples)}} \times 100$$

$$(9)$$

To explain rehydration ability (RA), three indices were calculated by using eq. (10)- eq.(12) which describe the behaviour of foods subimitted to rehydration (Barrera et al., 2016). These indices are water absoprtion capacity (WAC), dry matter holding capacity (DHC), rehydration ability (RA) as follows:

$$WAC = \frac{M_R . x_R^w - M_D . x_D^w}{M_0 - M_D} \qquad (10)$$

$$DHC = \frac{M_R .(1-x_R^w)}{M_D . (1- x_D^w)} \qquad \mathbf{(11)}$$

$$RA = WAC . DHC \qquad (12)$$

where: M is the total mass in (g) and x^i is the mass fraction of component in g/g. 0, D and R state the product before drying, the completely dried product and rehydrated samples, respectively which were written as subscripts. Superscript, i state water (w) or soluble solids (ss).

It was measured the water holding capacity (WHC) of the rehydrated structure from the soluble solids content of its liquid phase (z_R^{ss}) and the amount of liquid (M_{CF}) removed by centrifuging it at 4000 rpm for 10 min(eq.(13)).

$$WHC =$$
$$\frac{M_R . x_R{}^w - M_{CF} . (1 - z_R{}^{SS})}{M_R - X_R{}^w} \qquad (13)$$

III.1.4.8.2Diameter and thickness evolution

In order to determine best reconstitution properties and evaluate the recovery of volume of all dried apples, diameter and thickness increase were measured at every 30 min during the rehydration process at 30 and 70°C. The diameter and thickness of untreated and treated apples were calculated by measuring for each sample (5 slabs) by using image analysis program NH/ Image J software 1.8.0. Both dimensions were measured at different positions of the sample and their average valueswere considered. In particular the measurement positions on rehydrated apple slabs were as follows: four position diameters (along two perpendicular axes and two diagonal axes) and eight position thicknesses (Ponkham et al., 2012).

III.1.5Statistical analysis

All results were reported as the mean ± standard deviation (S.D).One way analysis (ANOVA) and Tukey's test were applied for comparing mean values by using SPSS 24 Software statistics program (SPSS Inc., Chicago, USA). Any statistical difference was considered significant with $p< 0.05$ and was indicated with different letters. Principal component analysis (PCA) was used to identify the principal components contributing to most of the variations within the dataset, evaluating the effect of pre-treatment, drying/rehydration temperatures on the quality parameters of untreated and treated rehydrated apples. All analyses were carried out using the SPSS software package, version 24 (SPSS Inc., Chicago, USA).

III.2 Raw material definition: 'Terzarola Gialla' peach

'Terzarola Gialla' (*Prunus Pesica* L. Bastsch. cv) is known the cultivar of peach. In the provinces of Naples and Caserta, 'percoca' are still cultivated. It is a category of peach with yellow pulp, very solid and not a freestone. The variety of "Terzarola Gialla" that mature from the end of summer are called "terzarola" (the third age) in Neapolitan. There are many ecotypes of terzarola: yellow, pink, gold and white; the most famous one is "terzarola col pizzo". Terzarola ripens, as the name says, late, from the end of August to October. Like all peaches, also this variety is hand picked and is used a lot for the production of peaches in syrup and are eaten also in pieces in the wine.

III.2.1 Raw material preparation

Fresh 'Terzarola Gialla'peach fruits were purchased from local market (Campania Region, Italy) and were kept in a refrigerator +4°C prior to use. Freshness, uniform size and absence of any mechanical damage were used as the selection criteria for the samples. Prior to drying the fruits were washed with tap water and peeled by using a knife. Cylindrical slabs with a diameter of 29 ± 0.16 mm and thickness of 6 ± 0.05 mm prepared and all trials were carried out using several raw materials. The average initial moisture content of fresh peach samples was determined according to AOAC (1997) and was found to be as 87.44 ± 1.31 on the wet basis.

Untreated peach slabs (UTR) and treated peach slabs (TR) were compared: Treated peach slabs (TR)were prepared with natural–innovative aqueous solution at 25°C. This pre-treatment was diluted solution with natural components in ppm concentration, and it did not contain any chemical substances (this new solution was created by Prof. Marisa Di Matteo, under the patent evaluation).

(a)   (b)

Figure III.5 *'Terzarola Gialla' peach (a) and peach slab (b)*
III.2.2 Drying experiments

Drying experiments of two types of samples: untreated (UTR) and pre-treated (TR) peach slabs were carried out in a convective dryer (ZanussiFCV/E6L3) at four different temperatures(45, 50, 55 and 60°C)ata fixed air velocity of 2.3 m/s until the water content plateau was reached. The treated and untreated apple slabs were put on the tray in convectional dryer. The same convective dryer also was used for drying experiments of peach samples. The some descriptions of oven were presented in part III.2.1 and the scheme of convective dryer was given in Figure III.2. During the drying process, the weight loss of slabs was recorded every 60 min by using a digital electronic balance (mod. Gibertini E42, Italy). Moisture ratio (M_t/M_0) was calculated as the ratio between the actual (M_t) and the initial (M_0) moisture content on dry basis. The results were reported in terms of M_t/M_0 vs time (min), where M_t was the moisture content (kg water/kg db) at a given drying time and M_0 was its initial value. They were reported as the average of three sets of experiments.

III.2.3 Mathematical modelling

A diffusion model was developed to describe the drying process of both untreated (UTR) and treated (TR) peaches. A three-dimensional model of mass transfer that assumes fruits as an isotropic, homogenous and continuous solid phase was adopted. In this model, an isothermal condition was also considered, since in the drying conditions here analyzed, the characteristic time of thermal transient was far lower than that of mass transport. Peaches can be considered as a cylinder, so that the equation that describes the mass diffusion phenomenon (i.e. water during drying) is eq.(14) in cylindrical coordinates.

$$\frac{\partial M}{\partial t} = \frac{1}{r}\left\{\frac{\partial}{\partial r}\left(rD_{eff}\frac{\partial M}{\partial r}\right) + \frac{\partial}{\partial z}\left(rD_{eff}\frac{\partial M}{\partial z}\right)\right\}$$

(14)

where D_{eff} is the diffusion coefficient (m^2/s) and M is the content of moisture on a dry basis (kg/kg$_{d.b.}$).

The initial condition is

$$M(r,z,t=0)=M_0 \quad 0<r<R_0 \quad 0<z<h$$

(15)

The boundary conditions are the following:

$$\frac{\partial M(r=0,z,t)}{\partial r} = \frac{\partial M(r,z=0,t)}{\partial z} = 0 \text{ for } t>0 \tag{16}$$

and at $r=R_0$, $z=h$ and for $t>0$

$$-D_{eff}\rho_s \frac{\partial M}{\partial r} = h_m \rho_s (M_{sur} - M_e) \tag{17}$$

$$-D_{eff}\rho_s \frac{\partial M}{\partial z} = h_m \rho_s (M_{sur} - M_e) \tag{18}$$

where R_0 is the radius of the sample (m); ρ_s is the solid density (kg/m^3) and it is kept constant, h_m is the moisture transfer coefficient (m/s); M_{sur} is the moisture at the surface and M_e is the equilibrium moisture content (kg/kg$_{d.b.}$) (i.e. the moisture content necessary to maintain equilibrium with the surrounding atmosphere).

Introducing the following dimensionless variables:

$$\bar{r} = \frac{r}{R_0}, \ \bar{z} = \frac{z}{R_0} \text{ and } \bar{M} = \frac{M}{M_0} \tag{19}$$

The eq. 14 becomes:

$$\frac{\partial \bar{M}}{\partial \tau} = \left(\frac{\partial^2 \bar{M}}{\partial \bar{r}^2}\right) + \left(\frac{\partial^2 \bar{M}}{\partial \bar{z}^2}\right) \tag{20}$$

whereis τ the dimensionless time $\tau = \frac{tD_{eff}}{R_0^2}$

Furthermore, the initial and boundary conditions in dimensionless form for cylinder are:

$$\bar{M}(\bar{r},\bar{z},\tau=0)=1 \text{ for } 0 < \bar{r} < 1, \ 0 < \bar{z} < \frac{h}{R} \tag{21}$$

$$\frac{\partial \bar{M}(\bar{r}=0,\bar{z},\tau)}{\partial \bar{r}} = \frac{\partial \bar{M}(\bar{r},\bar{z}=0,\tau)}{\partial \bar{z}} = 0 \text{ for } \tau > 0 \tag{22}$$

and at $\bar{r}=1$, $\bar{z}=\dfrac{z}{R}$, for $\tau > 0$

$$\frac{\partial \bar{M}}{\partial \bar{r}} = -Sh\left(\bar{M}_{sur} - \bar{M}_e\right) \tag{23}$$

$$\frac{\partial \bar{M}}{\partial \bar{z}} = -Sh\left(\bar{M}_{sur} - \bar{M}_e\right) \tag{24}$$

The convective mass transfer coefficient and the effective diffusion coefficient are correlated with the dimensionless Sherwood number:

$$Sh = \frac{h_m \cdot R_0}{D_{eff}} \tag{25}$$

where
R_0 is the radius of the sample (m). Sherwood number (Sh) is the ratio of the convective mass transfer to the rate of diffusive mass transport.

To consider the effect of shrinkage, a law of variation of the volume was introduced in the model. This law was expressed as an exponential decay law, equation (26).

$$\frac{V}{V_0} = y_0 + a\exp(-b\tau) \tag{26}$$

The volume of the sample was adjusted at each time step during the calculation of the mass governing equation, and an adaptive grid was used for simulations.

The finite element method was applied to solve the non-linear partial differential equations (eq. 20).with the initial and boundary conditions (eqs. 21-24).The convergence criterion assumed at each node of the computational domain was $|\bar{M}_k - \bar{M}_{k-1}| \ll 10^{-8}$ (where k represents the k-th iteration).

To determine the optimum value of the D_{eff}, the coefficient of determination of the fit (R^2), the reduced χ-square of the fit (χ^2) and the root mean square error of the fit (RMSE) were used as targets.

III.2.4 Quality Analysis

III.2.4.1 Water activity

The water activity of each sample was determined using a water activity meter (Testo 650, Testo Inc., USA) at 25°C. Approximately 3 gr of each

sample was put in the sample holder. After equilibration, a_w values were recorded. Each measurement was done in triplicate.

III.2.4.2 Surface colour measurement

The colour parameters of fresh and dried peach slabs (as untreated and treated) were measured using a colourimeter (Chroma Meter II Reflectance CR-300 triple flash mode aperture 10 mm Minolta, Japan), calibrated previously with a white standard ceramic plate. To analyze the colour change of fresh and dried samples, CIE lab colour coordinates (L*, a* and b*) were recorded and the average values were calculated for each sample. The lightness value (L*) indicates the lightness/darkness of the sample, a* index represents green when negative and red when positive, and b* index represents blue when negative and yellow when positive.

White index (WI) represents the degree of whiteness of fruits and vegetables and was calculated as follows (Adiletta et al., 2016b):

$$WI = 100 - \sqrt{(100 - L*^2) + (a*^2) + (b*^2)} \qquad (27)$$

The Hue angle (H), which indicates how an object's colour is perceived by human eye: red, orange, green or blue, was calculated as follows (Adiletta et al., 2016b):

$$H = \tan^{-1} b*/a* \qquad (28)$$

III.2.4.3 DPPH radical scavenging activity

Fresh and dried peach samples were extracted according to Oliveira et al. (2012) with some modifications. Fresh samples (5 g $\pm$ 0.01) and each dried samples (2 g $\pm$ 0.01) were cut into small pieces and 10 mL of methanol was added to all samples (80% v/v) (CHROMASOLV®, for HPLC, ≥99.9, Sigma-Aldrich, USA). The mixture was homogenized with Ultraturrax at 10 rpm and then stirred for 10 min by a vortex. Supernatant was filtered through a Whatmann No:2 filter paper. All extractions were carried out in triplicate.

The total antioxidant activity of fresh and dried peaches was measured by the DPPH radical scavenging method (Adiletta et al., 2018b). Different volumes of the extracts were mixed with 3.5 mL of 6×10^{-5} M of DPPH methanol solution in cuvettes. The reaction mixture was shaken properly and kept for 30 min at room temperature in the dark. The change of colour from purple to yellow indicated the progress of reaction due to the reduction of 1,1-diphenyl-2-picrylhydrazyl (DPPH) solution complex, as recorded by using UV-Vis spectrophotometer at 517 nm (Lambda Bio 40; PerkinElmer, Waltham, MA, USA). Methanol was used as the blank and the control sample was prepared without adding any extract. Percentage of inhibition of

DPPH radical was calculated as the following equation (Deng et al., 2018; Wang et al., 2018):

$$\% \text{ Antioxidant Activity} = \frac{\text{Abs}_{\text{control}} - \text{Abs}_{\text{sample}}}{\text{Abs}_{\text{control}}} \times 100 \qquad (29)$$

where
$\text{Abs}_{\text{control}}$ = the absorbance of control
$\text{Abs}_{\text{sample}}$ = the absorbance of sample

The results were expressed as the EC_{50} value, which was defined as the sample concentration (mg mL^{-1}) required to inhibit 50% of the DPPH radical scavenging activity. EC_{50} was determined from a graph of antioxidant capacity (%) versus extract concentration (mg mL^{-1} sample).

III.2.4.4 Shrinkage evolution

The initial volume (V_0) of untreated and treated peaches was calculated by measuring for each sample (10 slabs) the diameter and thickness by using a digital Vernier caliper (0.01 m accuracy) (Adiletta et al., 2016a). At given times during the drying experiments, the dimensions such as diameter and thickness of the same slabs were measured and the volume (V_t) was calculated. In order to reduce the measurement error during drying, both slab dimensions were measured at different positions, and specifically, the diameter in four positions symmetrically placed along the perimeter of the slab and the thickness in eight positions (Ponkham et al., 2012).For the evaluation of the shrinkage during drying, the volume ratio (V_t/V_0) was reported as a function of the relative moisture ratio (M_t/M_0).

III.2.4.5 Preliminary sensory evaluation

Thesensory evaluation of treated and untreated dried peach slabs was carried out by 15 untrained panelists recruited at the University of Salerno. A hedonic scale of five points which ranged from 1 "dislike extremely" to 5 "like extremely" with 3 as "neither like nor dislike" was used during sensory evaluation for the following quality attributes: colour, flavour, taste, sweetness, acidity and overall acceptability. The panelists' scores were calculated for each sensory attribute and average values were reported with the standard deviation. Acceptability of dried peaches was considered when the average score is equal or greater than "3.0" (Moreira et al., 2015).

III.2.4.6 Volatile organiccompounds (VOCs)

Peach fruits were subjected to GC/MS analysis to identify the volatile organic compounds (VOCs), whose extractions were carried out using a SPME fiber of divinylbenzene/carboxen/polydimethylsiloxane (Supelco,

Bellefonte, PA). Conditioning of the fiber was done at a temperature of 250°C for 30 min, which was then subjected to an exposure step for 30 min at 40°C to the headspace of the sample vial. The GC/MS equipment, column and conditions as described by Corona (2010)were used for analysis; 1-heptanol solution (35 mg/L 1-heptanol in 20% ethanol aqueous solution) was used as internal standard. The methodology was described by Alfonzo et al. (2016) and Martorana et al. (2016) was applied for the identification of the compounds. Determinations were carried on untreated(UTR) and treated(TR) fresh and dried samples.

III.2.4.7 Rehydration capacity

Rehydration experiments of peach slabs, previously dried, were performed at arehydration temperature of 30°C in distilled water by using a water bath to evaluate rehydration capacities (Doymaz, 2014; Önal et al., 2019). The approximate water to peach ratio was 100:1 (weight basis). At a specific time, the samples were taken out from liquid, blotted with tissue paper and measured by using an electronic balance. The slabs were weighted every 15 min in the initial phase of the rehydration process(up to 120 min) and then every 30 min. At these fixed times, samples were removed from rehydration media, dried gently with tissue paper and weighted by using an electronic balance.

The rehydration capacity described as percentage water gain (Adiletta et al., 2016a), was calculated from the sample weight difference before and after the rehydration as follows:

$$\text{Weight gain (\%)} = \frac{(\text{weight of rehydrated samples-weight of dried samples})}{(\text{weight of dried samples})} \times 100$$

(30)

III.2.5 Statistical analysis

All results were reported as the mean ± standard deviation (S.D).One way analysis (ANOVA) and Tukey's test were applied for comparing mean values by using SPSS 24 Software statistics program (SPSS Inc., Chicago, USA). Any statistical difference was considered significant with $p < 0.05$ and was indicated with different letters.

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III.3 Raw material definition: 'Rocha' Pear

'Rocha' pear (*Pyrus communis* L.) is the main cultivar produced in Portugal, and is classified as protected designation of origin (PDO). As it has a good storage potential, its distribution is possible for the market almost throughout the whole year, which is an important aspect that differentiates it competitively against other varieties.Pear production represents a significant economic activity in Portugal (c.a. 190.000 tonnes per year), and the cultivar Rocha pear represents 95 % of the National Production (Santos et al., 2014).

III.3.1 Raw material preparation

Fresh pears were obtained from local market (Porto, Portugal) and stored in a refrigerator +4°C prior to use. The selected pears showed uniform colour and regular shape. Before the drying process, the fruits were washed with tap water and peeled by using a knife. Cylindrical slabs with a diameter of 38 ± 0.12 mm and thickness of 6 ± 0.05 mm were prepared and all tries were carried out using several raw materials. The average initial moisture content of fresh pear samples was determined according to AOAC official method (AOAC,1998) at 105°C for 24 h and was found to be as 86.52±0.66 on the wet basis (6.42 kg water/kg db).

(a)   (b)

Figure III.6 'Rocha pear' (a) and pear slab (b)

Three pear processing conditions were used in this research: a) control (C) (without application of ultrasound and microwave pre-treatment), b) ultrasound pre-treatment (US), c) microwave pre-treatment (MW).

The pear samples (12 slabs/replicate) were placed in 3 L of distilled water into an ultrasonic bath (Bandelin Sonorex RK 255H, 300 mm (L) x 150 mm (W) x 150 mm (H)) with an ultrasound frequency of 35 kHz and a power of 160 - 640 W. The experiments were conducted for ultrasonic processing time of 10 min and the process temperature of 28°C. After the ultrasoundtreatment, the pear slabs were removed from ultrasound bath, dried with absorbent paper and subjected to drying process.

An important point of the methodology is that ultrasound pre-treatment was applied to packed in a vacuum. The vacuum-packed pears were used in order to prevent direct contact with a liquid medium and to avoid leaching substances contained in the pear samples. In this way, vacuum packaging might reduce the effect of cavitation over the pear samples (Nowacka and Wedzik, 2016).

Figure III.7 Ultrasound pre-treatment of pear slabs

The microwave pre-treatment was applied to pear slabs using a laboratory scale microwave oven (Beko 20 litre, P.C.R, dimensions: 454 mm (W) × 330 mm (D) × 262 mm (H)) at a frequency of 2450 MHz and a microwave power of 539 W for 4 min.

After the ultrasound and microwave pre-treatments, the pear slabs were immediately subjected to drying process.

III.3.2 Drying experiments

Drying experiments of these three types of samples: control (C), microwave (MW) and ultrasound pre-treated (US) pear slabs were carried out ina pilot plant convective tray drier (Armfield UOP8, Ringwood, England) at three different temperatures of 50, 55 and 60°C and fixed air velocity of 0.75 m/s until the water content plateau was reached.The pilot plant convective tray dryer with forced air and controlled temperature and velocity was shown in Figure III.8. Total sample mass, air drying temperature, air relative humidity and air velocity were monitored during the drying process.On-line acquisition of mass was recorded every 3 min by a digital balance (Sartorius, Goettingen, Germany) attached to the drying trays and connected to a computer (Hawlett Packard Vectra, California, USA) equipped with a recording program. Air temperature was monitored with a squirrel data-logger (Grant Instruments 1023, Cambridge, England) and three thermocouple wires attached to each of the three upper trays. Air velocity was measured regularly with a vane anemometer (Airflow LCA 6000, Buckinghamshire, England). Dry and wet bulb temperatures of the outlet air were registered in order to calculate the air relative humidity values, by an online psychometric chart calculator (Jayes, 2015). Average air

relative humidity values were found $33.97 \pm 0.81\%$, $31.67 \pm 0.46\%$, $27.27 \pm 0.75\%$ for drying temperatures of 50, 55 and 60°C respectively.

Moisture ratio (M_t/M_0) was calculated as the ratio between the actual (M_t) and the initial (M_0) moisture content on dry basis. The results were reported in terms of M_t/M_0 vs time (min), where M_t was the moisture content (kg water/kg db) at a given drying time and M_0 was its initial value. They were reported as the average of three sets of experiments.
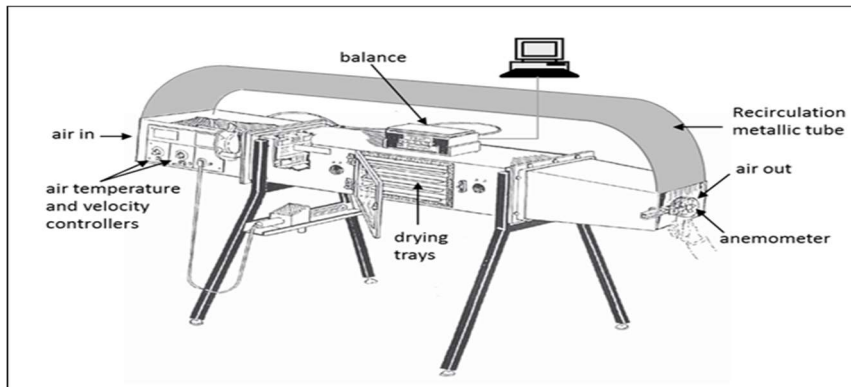
Figure III.8 Scheme of the pilot plant tray dryer
III.3.3 Mathematical Modelling: Empirical Models

Empirical models that are commonly applied to fruit materials were here utilized (Table III.1). Several models were tested to fit drying data of 'Rocha' pear samples, including Henderson & Pabis model, Page model and Modified Page model.

Table III.1 *Mathematical models applied to the drying curves of pear samples*

Model	Equation	References
Henderson & Pabis	$$\frac{X - X_e}{X_0 - X_e} = a \exp(-k\,t)$$ (31)	Doymaz (2005) Yaldız et al. (2001)
Page	$$\frac{X - X_e}{X_0 - X_e} = \exp(-k\,t^N)$$ (32)	Mahmutoğlu et al. (1996)
Modified Page	$$\frac{X - X_e}{X_0 - X_e} = \exp(-(k\,t)^N)$$ (33)	Lahsasni et al. (2004) Yaldız et al. (2001)

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where

X is the average water content on dry basis (kg water kg dry matter $^{-1}$), X_0 the average initial water content, X_e the average equilibrium water content, k the drying constant, a and N the drying coefficients, and t the time (min).

The empirical constants for the drying models were determined from experimental drying curves at each drying temperature and final moisture content of dried pear samples based on the eq. (34).

$$X_f = \frac{m_f\,(1+X_i)}{m_i} - 1 \tag{34}$$

where

X_i and X_f are initial and final moisture content of a given time interval; m_i and m_f are initial and final mass(g) obtained from the online recording balance.

The average equilibrium water content value of the control, microwave and ultrasound treated pear samples was determined by the GAB equation (35), using data from pear sorption isotherms presented by Vazquez et al. (1999).

$$\frac{X_e}{X_m} = \frac{C\,K\,a_w}{(1 - K\,a_w)(1 - K\,a_w + C\,K\,a_w)} \tag{35}$$

X_m is the water content on a dry basis corresponding to the monolayer value, C the Guggenheim constant, a_w the water activity and K a factor correcting properties of the multilayer molecules with respect to the bulk liquid (Bizot, 1983). C and K reflect the temperature effect.

A non-linear regression analysis was used to estimate the coefficients of the given models by SPSS 24 Software statistics program (SPSS Inc., Chicago, USA). The 95% standard error of the parameter (SE) and statistical indicators of the quality of the regression (coefficient of determination (R^2) and standard deviation of the experimental error (s)] were also calculated (Box et al., 1978). The evaluation criterion for selecting the best model was the according to the higher coefficient of determination (R^2)and the lower standard deviation of the experimental error (s).

III.3.4 Quality Analysis

III.3.4.1 Water activity

The water activity of each sample was measured in triplicate at 22-23°C with a water activity meter (Aqualab, Model Series 3 TE, Decagon Devices,

Inc.), previously calibrated with a standard solution of a water activity of 0.760.Three measurements were performed for each sample.

III.3.4.2Surface colour measurement

The colour values (L*, a* and b*) of fresh and dried (control, microwave and ultrasound pre-treated) pear slabs were assessed using a Minolta CR-400 colorimeter (Konica-Minolta, Osaka, Japan), calibrated with a white standard tile.The color brightness coordinate L* measures the whiteness value of a colour and ranges from black at 0 to white at 100. The chromaticity coordinate a* measures red when positive and green when negative, and the chromaticity coordinate b* measures yellow when positive and blue when negative (Olieveira et al., 2015)

White index (WI) represents the degree of whiteness of fruits and vegetables and was calculated as follows (Adiletta et al., 2016b):

$$WI= 100 - \sqrt{(100 - L*^2) + (a*^2) + (b*^2)} \qquad (36)$$

Total colour difference (ΔE) was determined using eq. (37) (Önal et al., 2019). The reference value for total colour difference (ΔE) was the fresh 'Rocha' pear.

$$\Delta E = \sqrt[2]{(\Delta L*)^2 + (\Delta a*)^2 + (\Delta b*)^2} \qquad (37)$$

III.3.4.3 Determination of total phenolic content

The methodology used for the extraction of fresh and all dried pears was adapted from Salta et al. (2010) and Önal et al. (2019). Fresh samples (5 g ± 0.01)and each dried sample(2 g ± 0.01) were cut into small pieces and 30 mL of methanol and 35 mL of methanol were added to fresh and dried pears, respectively (100% v/v) (CHROMASOLV®, for HPLC, ≥99.9, Sigma-Aldrich, USA). The mixture was homogenized with Ultraturrax (Ika digital T25, IKA-Werke GmbH & Co. KG) at 10 rpm and then stirred for 10 min by a vortex. Supernatant was filtered through a Whatmann No:2 filter paper. Three sample extracts were prepared for each sample and three measurements were performed for each replicate. These extractions were also used for the determination of total antioxidant activity (DPPH radical scavenging activity).

The total phenolic content in fresh and dried pears' (as control, microwave and ultrasound pre-treated) extracts were determined by the Folin-Ciocalteu colourimetric method (Singleton et al., 1999). The pear

extracts were oxidized by the Folin – Ciocalteu reagent in mixture of sodium carbonate solution and distilled water. The absorbance of reaction mixture was measured at 765 nm after 1 h in dark at room temperature by using a visible spectrophotometer (Novaspec II, Piscataway, NJ). The calibration curve was constructed with standard Gallic acid. The results were expressed as milligram of gallic acid equivalents per 100 g dry basis (mg of GAE / 100 g db).

III.3.4.4 DPPH radical scavenging activity

The total antioxidant activity of fresh and dried pears was measured by the DPPH radical scavenging method (Önal et al., 2019). Different volumes of the extracts were mixed with 3.5 mL of 6×10^{-5} M of DPPH methanol solution in cuvettes. The obtain solution was shaken properly and left for 30 min at room temperature in the dark. The absorbance of solution was measured at 517 nm by using a visible spectrophotometer (Novaspec II, Piscataway, NJ). Methanol was used as the blank and the control sample was prepared without adding any extract. Percentage of inhibition of DPPH radical was calculated as follows:

$$\text{Antioxidant Activity \%} = \frac{\text{Abs}_{control} - \text{Abs}_{sample}}{\text{Abs}_{control}} \times 100 \qquad (38)$$

where
$\text{Abs}_{control}$ = the absorbance of control
Abs_{sample} = the absorbance of sample

The results were expressed as the EC_{50} value, which was defined as the sample concentration (mg mL^{-1}) required to inhibit 50% of the DPPH radical scavenging activity. EC_{50} was determined from a graph of antioxidant capacity (%) versus extract concentration (mg mL^{-1} sample).

III.3.4.5 Shrinkage evolution

The initial (V_0) and final (V) volumes of control, microwave and ultrasound pre-treated pears were calculated by measuring the diameter and thickness of each sample (4 slabs) by means of digital Vernier caliper (0.01 m accuracy) (Önal et al., 2019).In order to reduce the measurement error during drying, both slab dimensions were measured at different positions, and specifically, the diameter in four positions symmetrically placed along the perimeter of the slab and the thickness in eight positions (Ponkham et al., 2012). After the final time of drying process, the shrinkage (%) was calculated (Aral and Beşe, 2016) as follows:

$$S \% = \frac{V_0 - V}{V_0} \times 100$$
(39)

III.3.4.6 Texture

Texture profile analysis (TPA) was performed using a texture analyzer (TA. XT-Plus, Stable Microsystems Ltd. Surrey, UK) equipped with a 50kg.f load cell and a cylindrical probe with a diameter of 5 mm.The cylindrical sample (38 ± 0.12 mm) was placed on a flat aluminium base and compressed (50% compression). The pre-test speed was of 2 mm/s, the test speed of 1 mm/s, the post-test speed of 1 mm/s.Sample, probe and base were not lubricated.

All operations were automatically controlled bythe texture analyzer. The instrument automatically recorded theforce–displacement or force–time curve. Six or seven replicateswere conducted for each type of pear fruits.
From the force–displacement or force–time graph of two compression–decompression cycles, the following attributes were determined: hardness, springiness, cohesiveness, chewiness.

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Figure III.9 *Texture profile analyser*

Hardness is the maximum compression force during the first cycle in the TPA curve.Cohesiveness is expressed as the dimensionlessquotient of the areas represented by the work to be done for two bites.Springiness (elasticity) is defined as the proportion of compression distancerecovered between the first and the second compression.Chewiness is theproduct of hardness, cohesiveness and springiness.
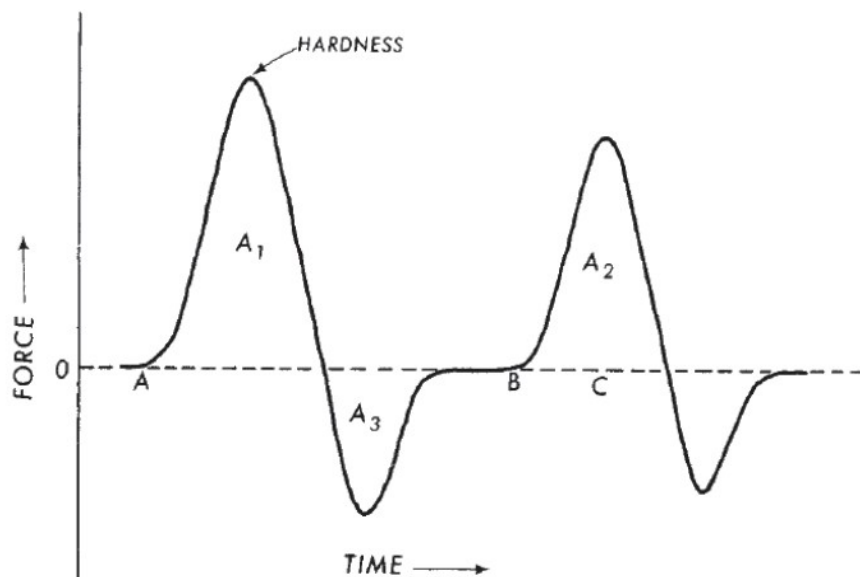
Figure III.10 *Texture profile analysis (TPA) curve (Friedman et al., 1963)*

III.3.4.7 Rehydration capacity

Rehydration experiments of dried pears were performed at 30°C in distilled water for 210 min by using a water bath until the constant water

content was reached. About 2±0.05 gr dried samples were placed in glass beakers containing water in the ratio 1:100. Samples were weighted every 15 min in the initial phase of the rehydration process (up to 120 min) and then every 30 min. At these fixed times, samples were taken out from rehydration media, blotted with tissue paper and weighted by using an electronic balance. The rehydration experiments were repeated in three sets independently three replicates and averages were reported with standard deviation.

The rehydration capacity described as percentage water gain (Adiletta et al., 2016a), was calculated from the sample weight difference before and after the rehydration as follows:

$$\text{Weight gain (\%)} = \frac{\text{(weight of rehydrated samples-weight of dried samples)}}{\text{(weight of dried samples)}} \times 100$$

(40)

III.3.5 Statistical analysis

All results were reported as the mean ± standard deviation (S.D).One way analysis of variance (ANOVA) and Tukey's test were performed for comparing mean values using SPSS 24 Software statistics program (SPSS Inc., Chicago, USA).The significance level assumed in all situations was 5% ($p< 0.05$) and was indicated with different letters.

Chapter III

ChapterIV
Results and Discussion

IV.1 Results of 'Annurca' apple slabs

IV.1.1 Drying kinetics and influence of pre-treatment on drying time

Air drying temperature and pre-treatments are important factors influencing drying kinetics, quality attributes and safe storage conditions of dried foodstuffs (Vega-Gálvez et al., 2012). In order to determine the effect of pre-treatment combined with different drying temperatures on the drying kinetics 'Annurca' apples, the curves of moisture ratio M_t/M_0 vs. drying time (min) were shown in Figure IV.1(a-d). The drying kinetic curves consist of a short period of initial transient followed by falling rate period for all investigated temperatures except at 55°C where initial transient and two falling rate periods occurred. According to these results, the falling rate period was characterized by mass transfer phenomena and diffusion was the dominant physical mechanism governing moisture transfer in the apple slabs.

As it was appeared fromFigure IV.1(a-d), the pre-treatment had a significant influence on apple drying behaviour, especially on drying time and on equilibrium moisture content. The average water activity of fresh apples was 0.98 ± 0.004. Apple slabs were dried up to final moisture of 0.04 ± 0.01 kg water/kg (db) and to the water activity of lower than 0.40 ± 0.03. The drying times needed to reach equilibrium moisture content were found equal to 600, 490, 420 and 210 min at the air-drying temperatures of 50, 55, 60, and 65°C, respectively, for the untreated samples. Time values for the treated samples were, respectively, 415, 325, 315 and 180 min at the same respective temperatures. At all the temperatures investigated a faster moisture loss for treated apples in comparison with untreated ones appeared during the drying process. Moreover, alower equilibrium moisture content was reached after pre-treatment. From this viewpoint, this dipping pre-

treatmentcontributed to increase the permeability of the cell membranes of apple slabs, thus improving external and internal water diffusivity.

In comparison with other studies reported in the literature, it is possible to observe that the results of current study were promising. Doymaz (2010), analyzing the effect of the blanching and citric acid pre-treatments on the apple slices in similar process conditions to present work (slice thickness of 5 mm, drying temperatures varying from 55 to 75°C, constant velocity of 2 m/s) observed that pre-treated apples with citric acid solution had shorter drying time than blanched and control ones. On the contrary, as reported by Kowalski and Mierzwa (2013), the application of osmotic pre-treatment (sucrose, glucose and fructose) did not have significant effect on the drying kinetics of convective dried apples. The drying time at 55°C of dried apple slabs with osmotic solutions was similar or even longer in comparison to untreated samples. This behaviour may be explained by the presence of a visible layer of sugar was probably formed during penetration of the soluble solid to the cell interior which may hinder moisture and heat transfer during the drying process. Applied pre-treatments prior to drying influence not only water diffusion but also product quality. Thus, the selection of suitable pre-treatments is important critical point in terms of drying process and high quality dried products.

a)

b)
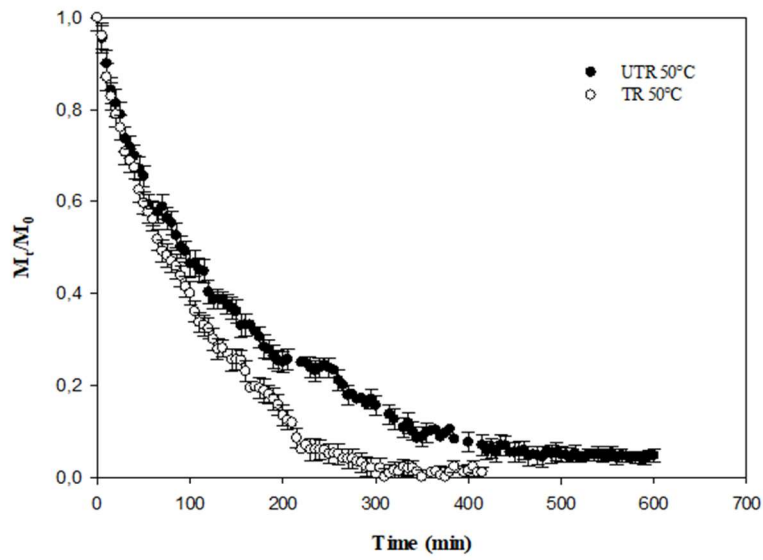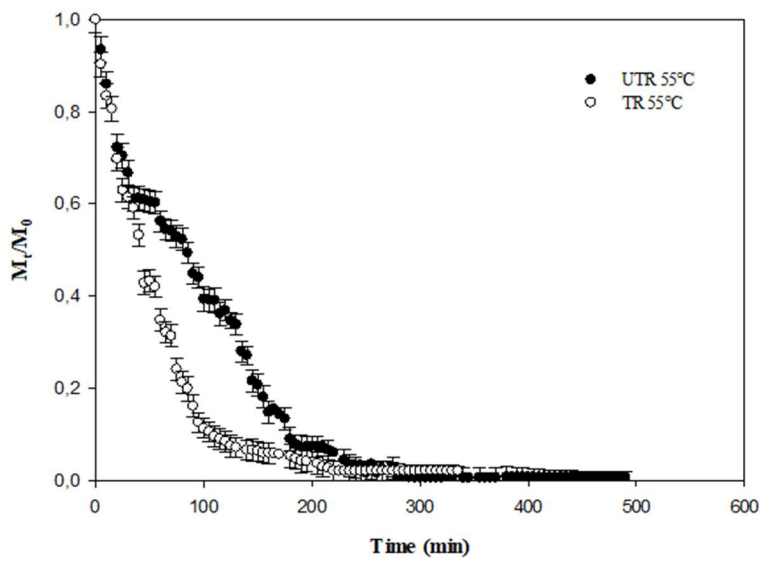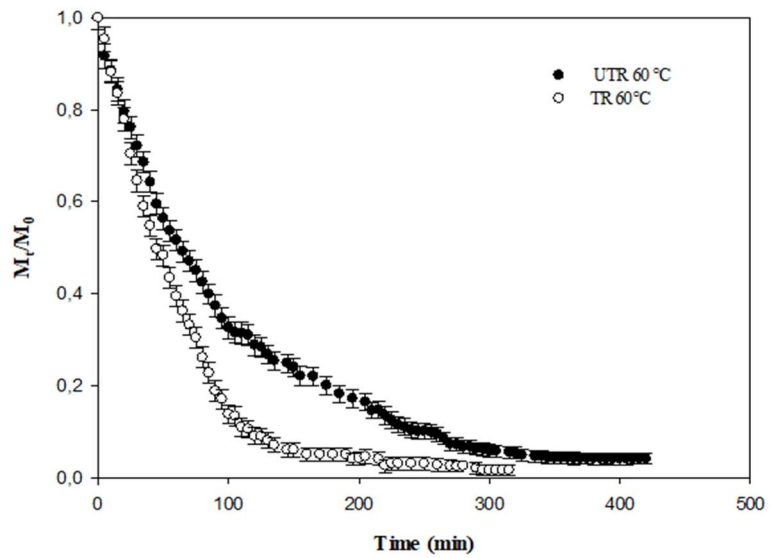
c)
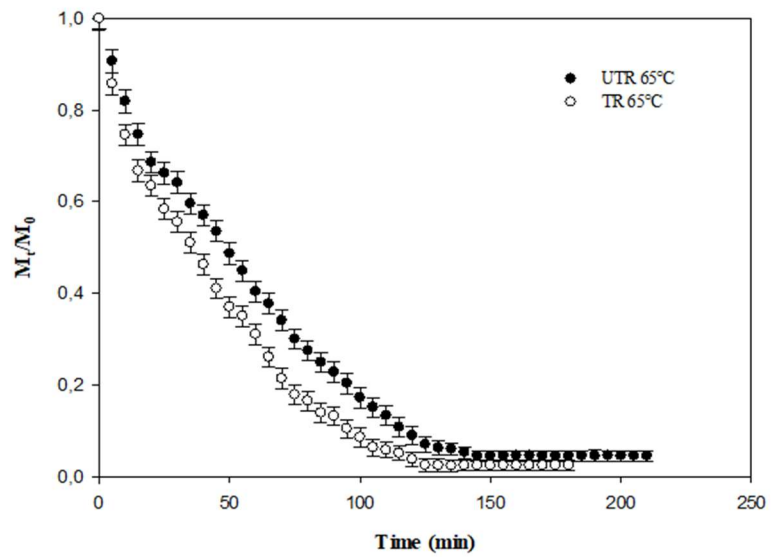
d)

Figure IV.1 *Experimental drying curves of untreated (UTR) and treated (TR) samples at 50°C (a), 55°C (b), 60°C (c), 65°C (d)*

IV.1.2 Mathematical Modelling

The parameter values of dehydration model with confidence intervals were reported in Table IV 1-3. As can be observed the confidence intervals are very close to the estimated values.

Table IV.1 *Model's parameters of apple drying*

Samples	K_m (1/cm)	Confidence Interval	D_0 (cm²/s)	Confidence Interval	E/R (K)	Confidence Interval
UTR	9.69×10^{-1}	8.79×10^{-1}-1.06	21.73	19.67-23.79	4.01	3.94-4.04
TR	9.70×10^{-1}	8.79×10^{-1}-1.06	13.61	12.15-15.08	3.68	3.63-3.72

Table IV.2 *Shrinkage velocity values of apple drying*

	u (cm²/s)							
	50°C	C.I.	55°C	C.I.	60°C	C.I.	65°C	C.I.
UTR	6.08×10^{-2}	5.68×10^{-2}- 6.49×10^{-2}	5.38×10^{-2}	4.83×10^{-2}- 5.92×10^{-2}	4.25×10^{-2}	3.60×10^{-2}- 4.80×10^{-2}	5.57×10^{-3}	4.02×10^{-3}- 7.13×10^{-3}
TR	1.94×10^{-2}	1.59×10^{-2}- 2.29×10^{-2}	1.44×10^{-1}	1.35×10^{-1}- 1.53×10^{-1}	1.02×10^{-1}	9.11×10^{-2}- 1.12×10^{-1}	9.53×10^{-4}	6.79×10^{-4}- 1.23×10^{-3}

Table IV.3 *Effective diffusion coefficient values (D_{eff}) of UTR and TR samples at different drying temperatures*

Temperature (°C)	D_{eff} UTR (m²/s)	D_{eff} TR (m²/s)
50°C	9.15×10^{-5}	1.54×10^{-4}
55°C	1.10×10^{-4}	1.83×10^{-4}
60°C	1.33×10^{-4}	2.17×10^{-4}
65°C	1.58×10^{-4}	2.56×10^{-4}

The results of nonlinear regression also indicated that the pre-treatment was more effective. It can be observed that the values of the velocity coefficient (*u*) changed with temperature for both treated and untreated cases. In particular the lowest value was observed for both cases at 65°C. This result means that at such temperature the drying process is close to a pure diffusive phenomenon. The parameter *u,* in fact, states the difference between the actual process and a pure diffusive one, due to shrinkage phenomenon occurring during drying. Hence, we can assess that a low value of the parameter *u* corresponds to low shrinkage.

Generally, diffusion is assumed as the dominant transport mechanism during drying and the rate of moisture movement is therefore described by an effective diffusivity value, D_{eff}. The results of effective diffusion

coefficient of UTR and TR samples at different temperatures were demonstrated in Table IV.3. Values of D_{eff} indicate an increase with increasing drying temperature.The effective diffusion coefficient of apple slabs was to found to be range between 9.15×10^{-5}- $1.58 \times 10^{-4} m^2/s$ untreated samples and for 1.54×10^{-4}– $2.56 \times 10^{-4} m^2/s$ for treated samples at different drying temperatures. Based on these results, pretreated samples had the highest effective diffusion coefficient compared to untreated ones. The values of D_{eff} are comparable with the reported values of 2.27-4.97×10^{-10} m^2/s mentioned for organic apple slices (5-9 mm) drying ina temperature range of 40-60°C (Sacilik and Elcin,2006), 2.46 to 8.15×10^{-9} m^2/s for Granny Smith apple slices (5 mm) at 50-80°C (Velic' et al., 2007), 0,48 to $5,63 \times 10^{-9}$ m^2/s for apple cubes (10mm × 10mm × 10 mm) at 40-65°C (Dikbasan, 2007). The differences between the results could be due to the differences in the varieties type of product, maturation time, pre-treatments, temperature and drying equipments.

The residuals, referring to experiments carried out at 50, 55, 60 and 65°C, are also reported in Figure IV.2(a-d) for UTR samples and Figure IV.3(a-d) for TR samples. Data are uniformly dispersed and very close to zero as in good fitting.
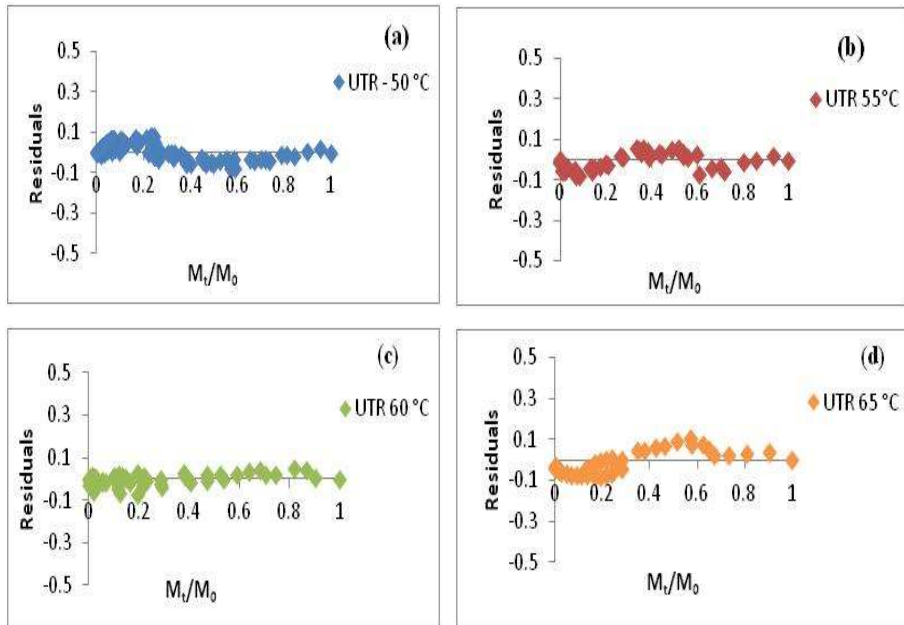
Figure IV.2 *Residual analysis, data refer to drying (a) 50°C, (b) 55°C, (c) 60°C, (d) 65°C for untreated(UTR) samples*
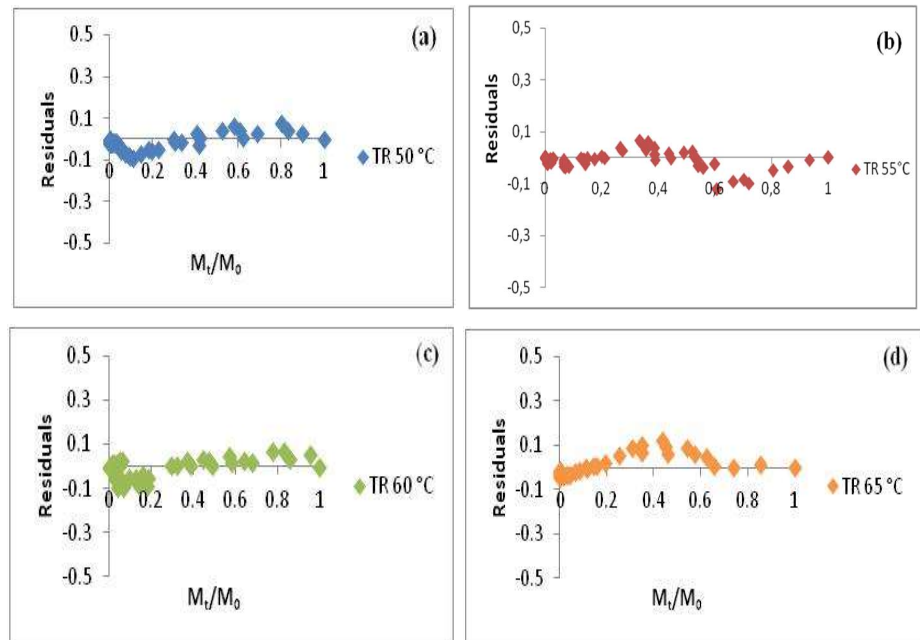
Figure IV.3 *Residual analysis, data refer to drying (a) 50°C, (b) 55°C, (c)60°C, (d) 65°C for treated (TR)samples*

The evolution of theoretical and experimental water content (M_t/M_0) is compared in Figure IV.4(a-d)and Figure IV.5(a-d). The model was able to predict with sufficient accuracy the evolution of water for both samples at each temperature. Figure IV.4 and Figure IV.5 showed that a very good agreement at each drying temperature.

a)

b)
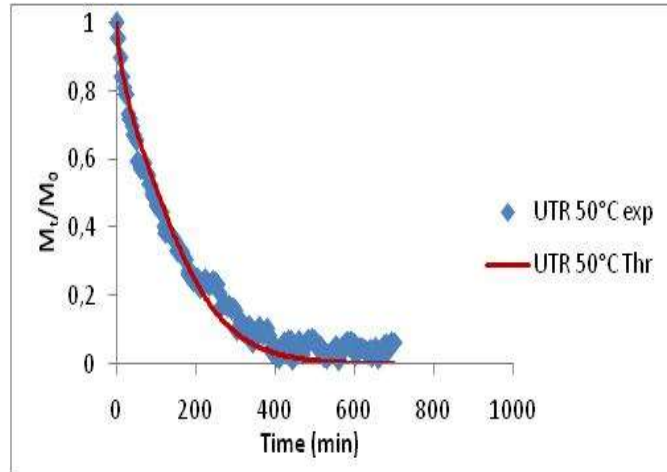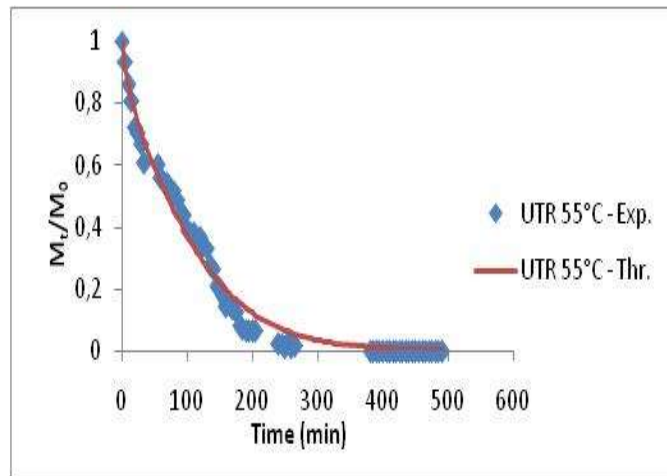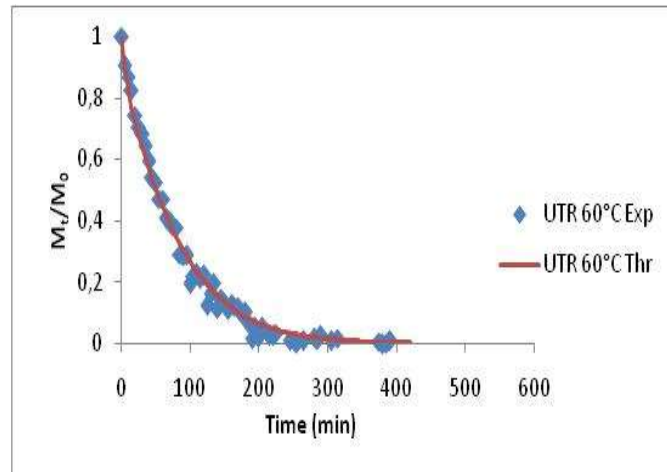
c)
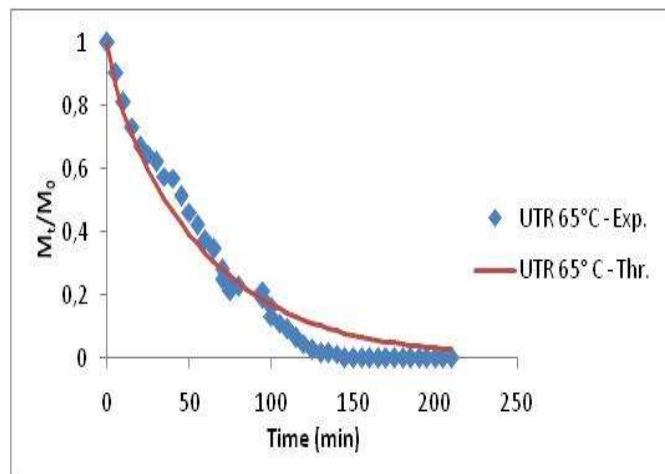
d)

Figure IV.4 *Experimental and theoritical total dimensionless water content temporal profiles drying at (a) 50°C (b) 55°C (c) 60°C (d) 65°C for untreated (UTR) samples*
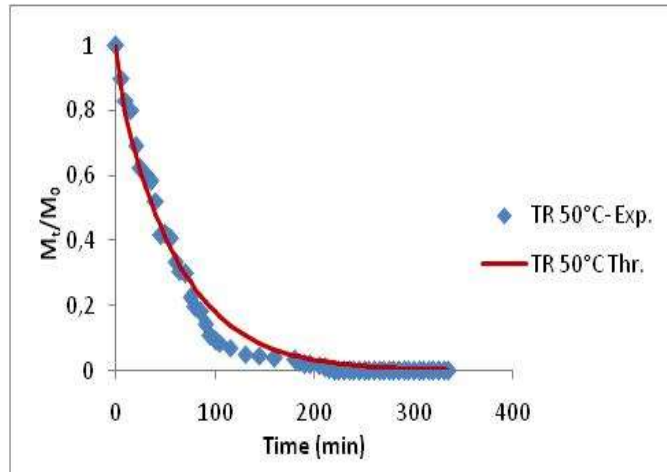
a)
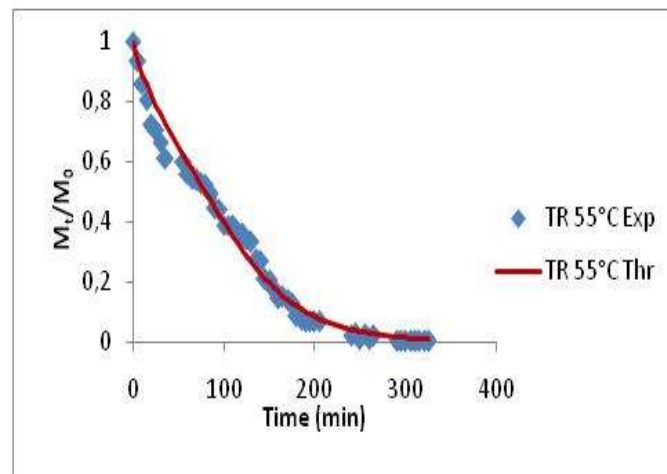
b)
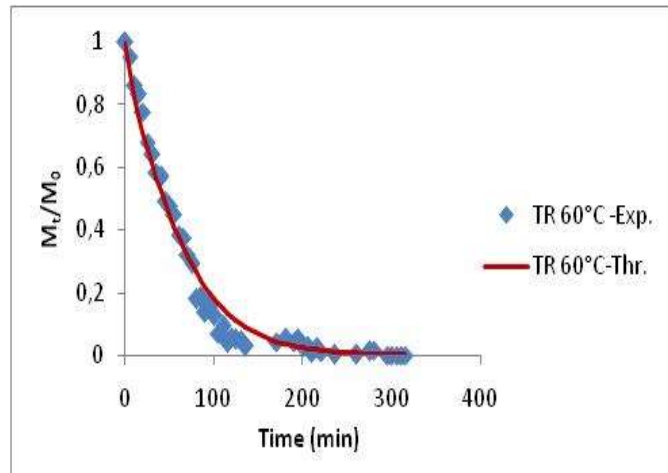
c)
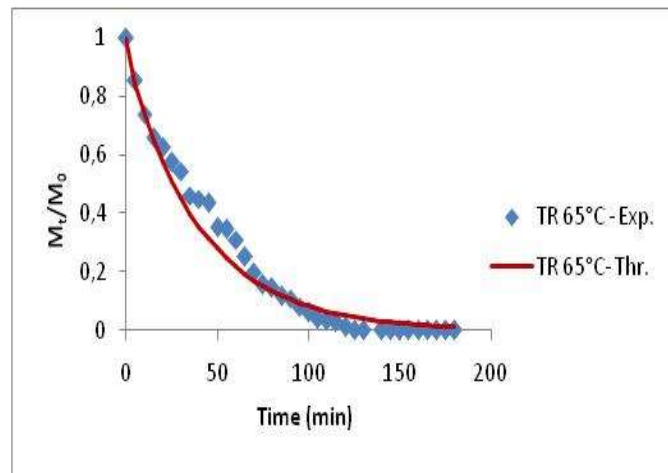
 d)

Figure IV.5 *Experimental and theoritical total dimensionless water content temporal profiles drying at (a) 50°C (b) 55°C (c) 60°C (d) 65°C for treated (TR) samples*

IV.1.3 Colour evaluation

Colour is one of the most important quality indices of dried food products for their marketing value and consumer choices. The changes in colour of dried fruits may be explained as a function of the chemical, biological and physical reactions that occur during thermal processing. The fresh fruits, especially apples with high content of polyphenols, polyphenol oxidase (PPO) and peroxidase (POD) are prone to enzymatic browning. Non-enzymatic browning includes a wide number of reactions such as the Maillard reaction, caramelisation and chemical oxidation of phenols (Deng et al., 2017).

The effects of pre-treatment, drying process and temperatures on surface colour of fresh and dried samples were shown in Table IV.4. Lightness (L*), white index (WI) and total colour difference (ΔE) were reported. Results demonstrated that pre-treatment and drying process had aremarkable effect on the colour parameters of fresh and dried apples. After drying, the L* value decreased in all untreated samples, indicating a decrease of brightness compared to fresh apples. But treated samples had higher (L*) values than untreated ones at all drying temperatures. The difference in brightness showed that all untreated samples were darker than treated ones.

As regards the white index (WI), a significant decrease with respect to fresh sample was determined in all the untreated samples which can be explained by the outbreak of browning on the surface of the dried apples (Albanese et al., 2007). In particular, in the analyzed range of temperatures no significant differences in terms of WI were found between all the untreated samples. On the contrary, treated samples showed similar WI values to those of fresh ones (p>0.05).

The highest quality dried apples would be those whose colour is the closest to the original one of fresh apples, since low values of colour changes (ΔE) were desired for consumer acceptance. A significant colour difference (ΔE) was found in dried apples also in some cases after pre-treatment (p<0.05). According to the results in Table IV.4, the lower values of ΔE were observed in treated samples dried at 60°C and 65°C, demonstrating that the pre-treatment is able to maintain the colour of the final product and contributed to the homogeneous colour distribution on the apple surface (Figure IV.6a-d). On the contrary, long drying times at low temperatures could promote apple discoloration associated with formation of browning products by simultaneous heat and mass transfer (Vega-Gálvez et al., 2012).

Table IV.4 *Colour parameters for untreated (UTR) and pre-treated (TR) fresh and dried 'Annurca' apples. Different superscript letters (a,b,c, etc.) in the same column mean significant differences (p<0.05)*

Sample	L*	WI	ΔE
UTR Fresh	81.73 0.66[abc]	72.13 ± 1.79[bc]	-
TR Fresh	84.79 ± 2.89[bc]	76.91 ± 1.02[c]	-
UTR 50°C	78.68 ± 2.44[a]	63.10 ± 1.17[a]	17.41 ± 0.99[f]
TR 50°C	81.71±0.61[abc]	73.68 ± 2.30[bc]	11.39 ± 1.02[d]
UTR 55°C	79.70 ± 1.33[a]	59.85 ± 0.73[a]	16.20 ± 0.71[f]
TR 55°C	82.92 0.78[abc]	70.24 ± 0.00[b]	12.48 ± 1.24[de]
UTR 60°C	80.04 ± 1.90[ab]	63.36 ± 3.06[a]	13.61 ± 0.96[d]
TR 60°C	84.70 ± 0.93[bc]	72.26 ± 0.29[bc]	5.46 ± 0.82[b]
UTR 65°C	80.16 0.78[abc]	62.17 ± 0.93[a]	7.90 ± 0.68[c]
TR 65°C	84.88 ± 0.25[c]	76.85 ± 0.21[c]	3.49± 0.43[a]

Figure IV.6 *Annurca apple slabs: fresh untreated (a), fresh treated (b), dried untreated (c) and dried treated (d) at 65°C*

IV.1.4 Total phenolic content (TPC)

The results of the effect of pre-treatment and air-drying temperatures on total phenolic content (TPC) were summarised in Figure IV.7. The TPC of fresh untreated and treated apple slabs was 419.46 and 499.80 mg GAE/100 g db. It can be possible to see from Figure IV.7 that untreated samples after drying had a significant reduction of TPC respect to fresh ones, but the increase of drying temperature did not significantly affect the polyphenols content.

In contrast, there were no statistical differences (p>0.05) between treated fresh samples and treated dried ones up to 60°C. Only at the highest temperature (65°C) a significant reduction in TPC was observed.

These results agree with those reported by Vega-Gálvez et al. (2012) found that an increase in air drying temperature caused a degradation of TPC in dried apples with respect to fresh ones. However, Rodríguez et al. (2014)

observed lower TPC loss at higher temperature, specifically: 38.7±1.6% at 30°C, 26.5±1.9% at 50°C and 19.7±1.6% at 70°C in apples during the convective drying. The reduction of phenolic compounds of samples may be explained by three possible mechanisms, including the release of bound phenolic compounds; partial degradation of lignin, leading to release of phenolic acid derivatives and the beginning of thermal degradation of the phenolic compounds, as reported in literature (Méndez-Lagunas et al., 2017).

On the contrary, Lutz et al. (2015) reported that the total phenolic content of hot air dried apples (ranging from 40°C to 130°C) were higher than fresh ones. This increment of TPC after convective drying may be attributed to the release of polyphenolic compounds from the food matrix. (Dalmau et al., 2017) or the formation of Maillard reaction products which could cause new phenolic compounds to form from precursors (İzli, 2017).

In summary, literature results demonstrate that the different drying methods, drying temperatures and applied pre-treatments effect the constancy of thermolabile phenolic compounds (Horuz et al., 2017). In our case, the proposed pre-treatment assured their conservation up to 60°C.
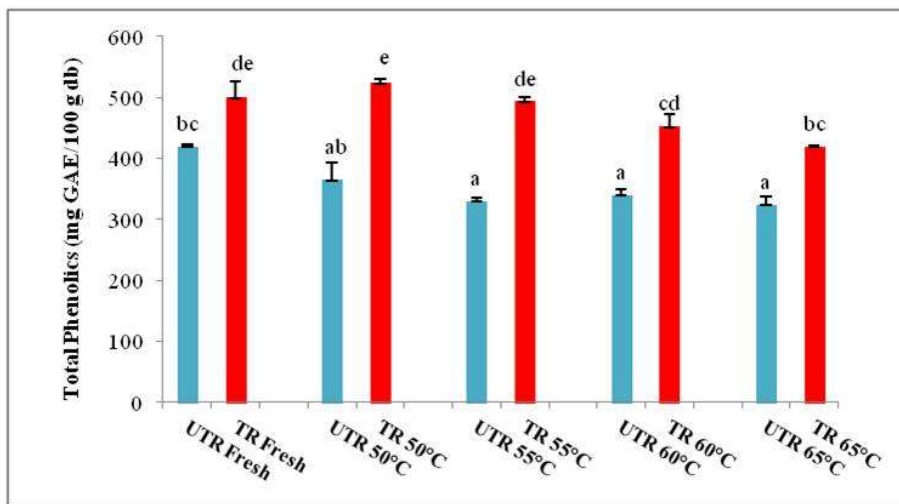
Figure IV.7 *Total phenolic content in fresh and dried apple samples (50, 55, 60 and 65°C)*

IV.1.5 DPPH radical scavenging activity

The radical scavenging activity of fresh and dried apple slabs for both untreated and treated samples was given in Figure IV.8 at different drying temperatures. The lowest EC_{50} values (the highest antioxidant activity) were determined as 16.06 and 18.66 mg/mL db in treated and untreated fresh

apples, respectively. For treated samples at lower drying temperatures (50 and 55°C) the antioxidant activity was better retained than at higher drying temperatures. This trend of antioxidant activity may be associated with modifications of the chemical structure of the main antioxidant compounds in apple (i.e, chlorogenic acid, quercetin, gallic acid, α- tocopherol etc.) or interactions between antioxidant compounds and other apple constituents, such as proteins at higher temperatures.Similar results were reported by Vega-Gálvez et al. (2012) for apple, Adiletta et al. (2016a) for grape, and İzli (2017) for convective dried dates. No statistical differences (p >0.05) were found between the untreated samples dried at 50°C and 55°C. Analogous result was found for treated samples.In contrast to these findings, Rodríguez et al. (2014),analysingthe effect of the drying temperatures (30, 50 and 70°C) on the antioxidant activity of air dried apples, observed that higher the drying temperature and lower is the antioxidant activity loss.

Drying temperature and pre-treatments play an important role in determining the quality of the final product, especially in the way of its antioxidant potential and health-promoting effects. On the basis of the results in this study, the used pre-treatment and the lower drying temperatures (50 and 55°C) can better preserve the antioxidant activity of apple slabs. However, these results are not in agreement with those obtained for total phenolic content which decreased increasing temperature (Figure IV.8). This behaviour can be explained considering that during the drying process, activation of oxidative enzymes, such as polyphenoloxidase and peroxidase, may lead to the loss of phenolic compounds. In addition, binding of phenolic compounds to proteins, changes in chemical structures or low extraction efficiencies are other factors related to the loss in total phenolic content (Gumusay et al., 2015). With regards to antioxidant activity, recent researches suggested that the high antioxidant activity in fruits and vegetables after drying might be related to the fact that partially oxidized polyphenols have greater antioxidant activity than unoxidized polyphenols (Nora et al., 2014). In addition, increases in antioxidant capacity after drying may be related to the Maillard reaction products (MRPs), which can be formed as a consequence of heat treatment or prolonged storage and, which generally exhibit strong antioxidant properties (Kamiloglu and Capanoglu, 2014).
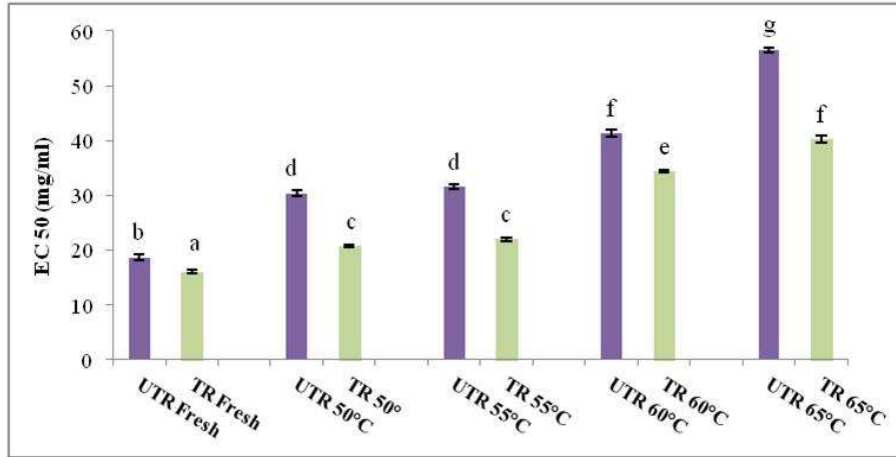
Figure IV.8 *Antioxidant activity of fresh and dried apple samples (50, 55, 60 and 65°C)*

IV.1.6 Shrinkage

One of the most important physical changes which influences the quality of dehydrated foodstuffs is known as shrinkage. Shrinkage may occur as a consequence of change in shape, collapse of cells and pores, loss of volume and hardness increase; all these phenomena in many cases may cause a negative impression on the consumers (Brasiello et al., 2013; Mayor and Sereno, 2004). The effect of pre-treatment and different air drying temperatures on shrinkage of apple slabs was investigated and the change in volume ratio (V_t/V_0) as a function of the moisture ratio (M_t/M_0) was reported in Figure IV.9(a-d). Based on the experimental results, the shrinkage of apple slabs increased with the removed water content. During drying process, at the same moisture content, treated apples were less shrunk than untreated ones at all investigated temperatures. Therefore, it can be underlined that this innovative pre-treatment had a positive effect on the shrinkage of apple slabs probably due to protection of dried fruit structure. These observations are in good agreement with earlier results reported for grapes (Adiletta et al., 2016a), cape gooseberry fruits (Junqueira et al., 2017) and apple disks (Moreira et al., 2000). Moreover, Nowacka et al. (2012) demonstrated that the ultrasound pre-treatment of apples before convecting drying resulted in between 9% and 11% higher shrinkage when compared to untreated samples.

In addition, shrinkage decreased with increasing drying temperature from 50 to 65°C. This behaviour can be explained by the increasing drying rate at the higher drying temperatures which leads to mechanical stabilization of the apple surface and limited degree of shrinkage. This is in agreement with the

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findings of Lewicki and Jakuzcyk (2004), who reported for dried apples that decreasing drying temperature from 80 to 50°C caused the gradual increase of volumetric shrinkage from 0.49 to 0.58 (more than 18%). Moreover, they observed that the shrinkage of apple slices was not isometric: the height of apple slices shrank more than diameter.Sturm et al. (2012) mentioned that the increasing both air temperature (35-85°C) and air velocity (2.0- 4.8 m/s) resulted in a reduction of shrinkage in hot air dried apples. On the contrary, some studies have reported that shrinkage is reduced by lowering the drying temperatures because of the slow drying rate and uniform moisture distribution in fruits such as chrysanthemum (Wang et al., 2018a, b) papaya slabs (Udomkun et al., 2016).

From this viewpoint, shrinkage was affected by many factors such as food materials, drying rate, drying air temperature, used pre-treatment-its availability and drying conditions.

a)

b)
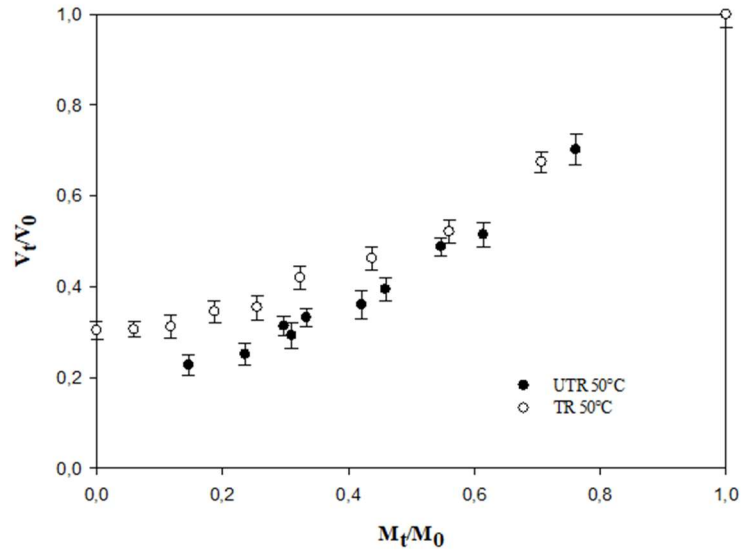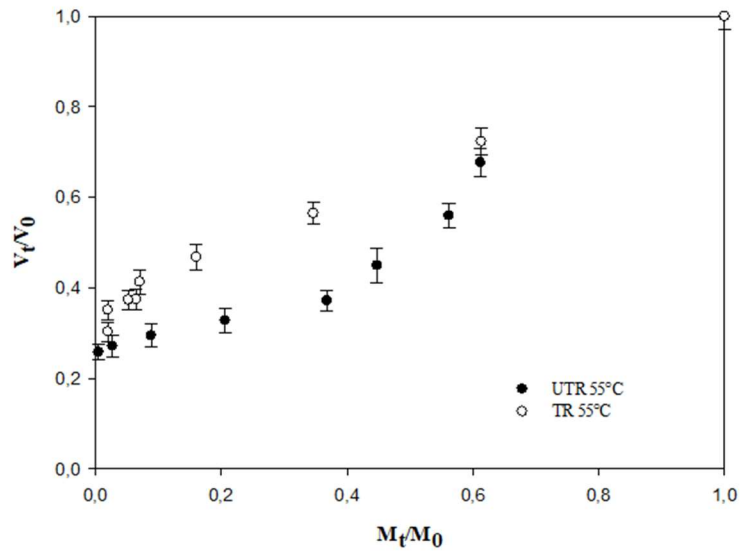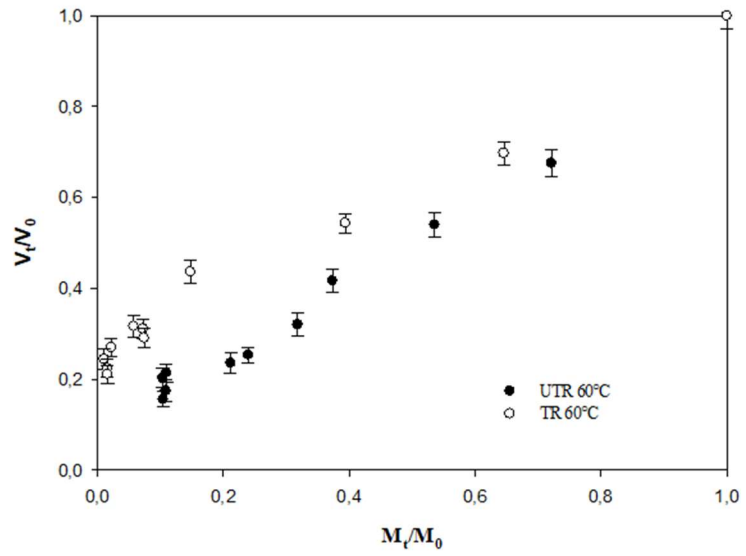
c)
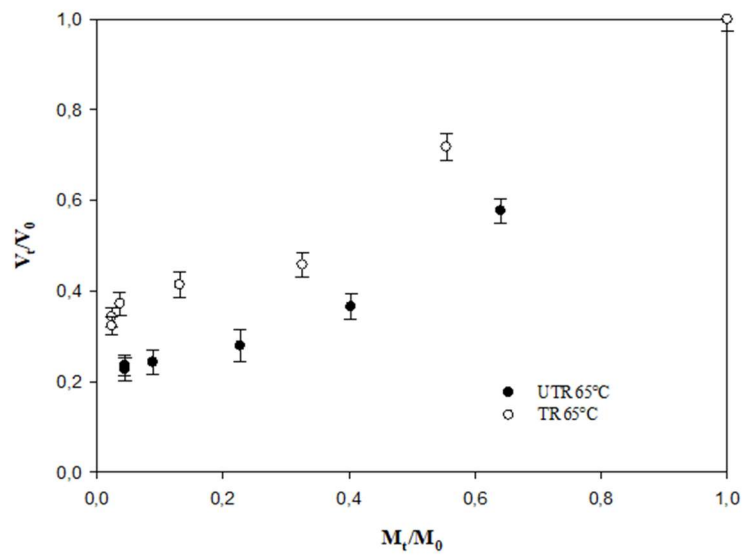
d)

Figure IV.9 *Experimental data of volume shrinkage of untreated (UTR) and treated (TR) apples during drying at 50°C (a), 55°C (b), 60°C (c) and 65°C (d)*

IV.1.7 Microstructure analysis

SEM analysis was used to examine the structure of both untreated and treated dried apples. Typical SEM images of apples dried at 50, 60 and 65°C were reported in Figure IV.10(a-f) for untreated and treated samples, respectively.

According to SEM images, a cell breakage was observed in the untreated dried samples, especially at higher temperature, emphasizing loss of turgor and rupture of cell membranes from the breakage zone. This turgor loss is due to degradation of cell walls (mainly attributed to pectins) and causes a possible shrinkage phenomena in the cell walls. In general, hot air convective drying of fruits and vegetables is characterized by structural changes as macroscopic (shrinkage) and microscopic (porous) levels. These mentioned structural changes could be followed by collapse of tissue and surface cracking in consequence of loss of cell turgor (Brasiello et al., 2013; Vallespir et at., 2018). The untreated dried apples at 50°C presented a contracted-closed microstructure, broken and non-uniform pores (both larger and smaller pores) with respect to the treated dried samples. These phenomena may be explained by the loss of the initial water present into the food matrix and occur when the solid matrix is not sufficiently strong to retain its structure (Adiletta et al., 2016b). At higher temperature (65°C) the cell volume reduction and tissue collapse caused the formation of a lamaller structure (Figure IV.10e), which resulted more porous and with larger pores after pre-treatment (Figure IV.10f).

Therefore, it can be concluded that the observed effect of pre-treatment may be attributed to the ability maintaining the solid structure of the apples by protecting the cell walls. A similar trend of the microstructure was reported by Adiletta et al. (2016b) for dried eggplant, Maldonado et al. (2010) for dried mango ad Rojas and Augusto (2018) for dried pumpkin.
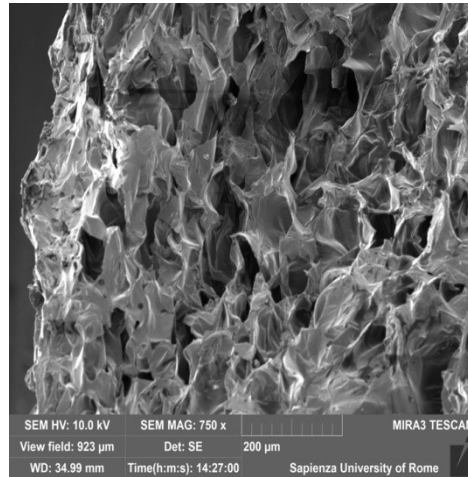
a) UTR 50°C
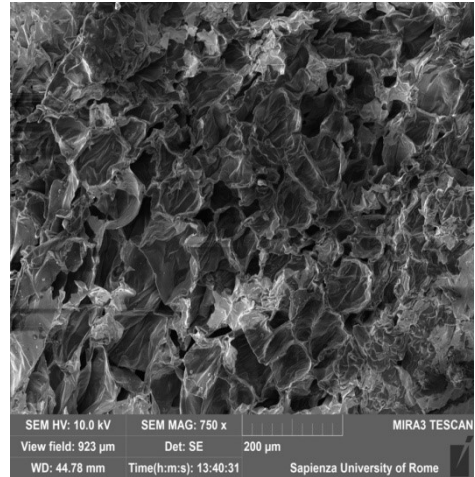
b) TR 50°C
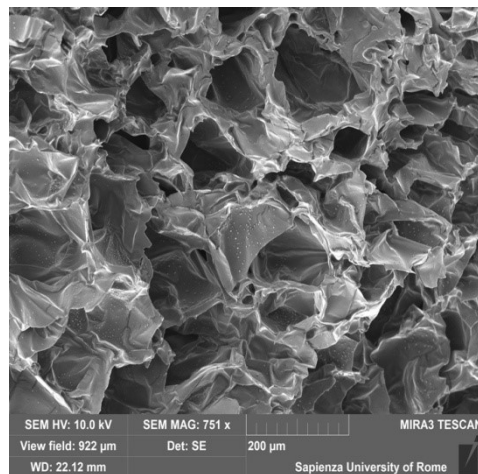
c) UTR 60°C   C
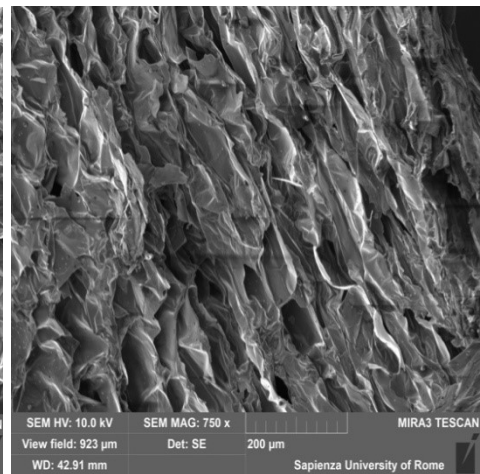
d) TR 60°C
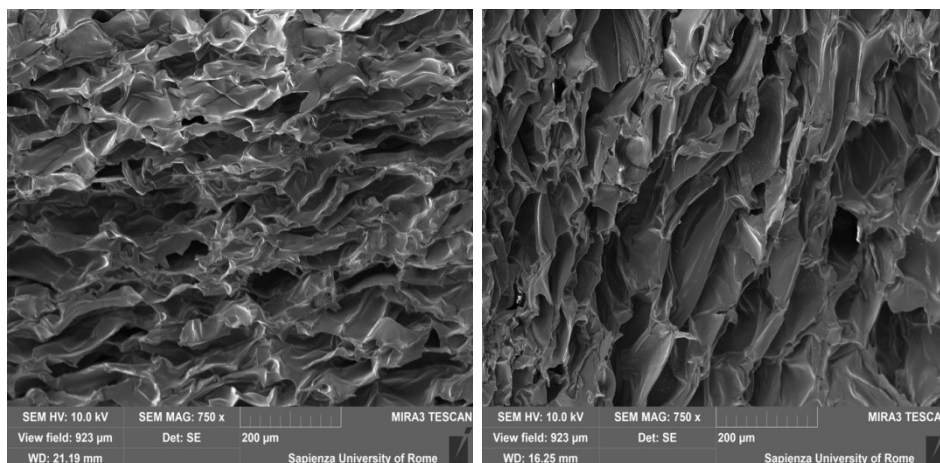
e) UTR 65°C   f) TR 65°C

Figure IV.10 *SEM images of untreated (a,c,e) and treated (b,d,f) apples dried at 50°C (a,b), 60°C (c,d) and 65°C (e,f)*

IV.1.8 Preliminary sensory evalaution

Sensory quality attributes of dried fruits play an important role in consumer satisfaction and their marketing value. Table IV.5 reported the effect of pre-treatment and air-drying temperatures on the sensory quality of the dried apple slabs. The pre-treated samples received the highest scores except for acidity in comparison with the untreated ones. Colour is one of the most important quality attributes for consumer preferences. Changes of colour are usually accompanied by flavour changes (Agustus Leon et al., 2002). The colour of dried treated samples was not significantly ($p > 0.05$) affected by the different air-drying temperatures. These results were mainly related to the great impact of the pre-treatment on the colour characteristics of dried apples which made possible an enhancement in lightness and reduced undesirable browning reactions. Concerning flavour, no significant differences ($p > 0.05$) were observed between treated dried samples, except those at 50°C.

Both treated and untreated samples dried at 50°C obtained the lowest flavour scores. This loss of volatile flavour compounds in dried apples at 50 °C may be due to the inactivation of volatile forming enzymes or to chemical reactions (oxidation, browning, Maillard etc.) which contributed to flavour deterioration (Agustus Leon et al., 2002; Nijhuis et al., 1998). The best results for taste were attributed by the panel members to treated samples at higher temperatures (60°C and 65°C) which were characterized by higher

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sweetness and taste. Therefore, the higher air-drying temperature (65°C) determined the better overall acceptability.

Table IV.5 *Preliminarysensory evaluation for quality attributes of untreated (UTR) and treated (TR) dried apples (50, 55, 60, 65°C). Values with different letters in a given column are significantly different (p<0.05)*

Sample	Colour	Flavour	Taste	Sweetness	Acidity	Overall Acceptability
UTR 50°C	1.60 ± 0.48^a	2.14 ± 0.69^a	2.86 ± 0.37^{ab}	3.57 ± 0.53^{ab}	1.43 ± 0.53^a	2.86 ± 0.38^{ab}
TR 50°C	4.17 ± 0.37^c	3.14 ± 0.38^b	3.57 ± 0.53^{bcd}	3.43 ± 0.53^{ab}	1.43 ± 0.53^a	3.86 ± 0.38^{cde}
UTR 55°C	2.29 ± 0.48^{ab}	2.57 ± 0.53^{ab}	2.50 ± 0.53^a	2.71 ± 0.49^a	1.57 ± 0.53^a	2.43 ± 0.53^a
TR 55°C	4.14 ± 0.38^c	4.14 ± 0.38^c	3.57 ± 0.53^{bcd}	3.29 ± 0.49^{ab}	1.43 ± 0.53^a	3.57 ± 0.53^{bcd}
UTR 60°C	2.78 ± 0.44^b	2.89 ± 0.33^{ab}	3.33 ± 0.50^{ab}	3.00 ± 0.50^{ab}	1.56 ± 0.53^a	3.33 ± 0.50^{bc}
TR 60°C	4.56 ± 0.52^c	4.11 ± 0.60^c	4.22 ± 0.64^{cd}	3.89 ± 0.60^b	1.22 ± 0.44^a	4.22 ± 0.44^{de}
UTR 65°C	2.92 ± 0.51^b	2.85 ± 0.69^{ab}	3.54 ± 0.52^{bc}	3.42 ± 0.67^{ab}	1.17 ± 0.39^a	3.42 ± 0.52^{bc}
TR 65°C	4.46 ± 0.52^c	4.07 ± 0.64^c	4.31 ± 0.48^d	4.08 ± 0.64^b	1.15 ± 0.38^a	4.31 ± 0.48^e

IV.1.9 Rehydration capacity and rehydration indices

Rehydration is a complex process of moistening of dry products and it cannot be interpreted as a reversible process of drying. Such a complex phenomenon is influenced by many factors, for example, drying method and conditions, pre-treatments, structure of tissue and chemical composition. During reconstitution of dehydrated products, the amount and rate of water absorption are important points that affect the quality properties. The rehydration features of a dried product are accepted as a quality index and indicate the physical and chemical changes during drying (Horuz et al., 2017; Lewicki, 1998).

The results of this work referred to untreated and treated apples dried at 50 – 65°C and rehydrated at 30°C and 70°C until the moisture content reached equilibrium, in order to evaluate the damageof drying and the effect of rehydration temperature on the rehydration behaviour.

The weight gain (%) during rehydration was shown in Figure IV.11(a,b). and Figure IV.12(a,b) for both untreated and treated dried apples at temperatures of 30 and 70°C, respectively.

It was observed that at any rehydration temperature all the samples showed the same trend. All rehydration curves showed a clear logarithmic tendency, and as expected, the rehydration time decreased when the temperature increased from 30 to 70°C.

At the end of the experiments at 30 and 70°C, untreated and treated samples reached almost the same water gain of 420% and 460%, respectively. As rehydration time progressed, there was a decrease in the driving force for water transfer and the system slowly attained equilibrium.

According to rehydration temperatures, the rapid initial water uptake lasted different times: for both samples at 70°C the water uptake was faster for the first 30 min with respect to 30°C where it was for 100 min.

This rapid initial water uptake may be due to the filling of capillaries and cavities, which are near the surface. The lower water gain during the late stage of experiments indicated a decrease in the water transfer when the cavities are filled up and then the system slowly attained equilibrium (Önal et al., 2019).

It was found that 65°C was the optimal drying temperature in terms of the main quality parameters (i.e, drying behaviour, colour, shrinkage, sensory evaluation). For this reason, in Figure IV.13 and Figure IV.14 it was reported the comparison between treated and untreated samples at 30 and 70°C, respectively.

The treated samples exhibited a higher rehydration capacity with respect to the untreated ones at both rehydration temperatures. Less and slower rehydration of untreated samples was probably due to the tissue collapse by longer drying times. The structure of untreated samples was probably significantly modified by drying. In other words, the carbohydrate/salt solution containing trehalose here proposed had a remarkable effect on the protection of apple structure during the rehydration process. This is in agreement with the findings of Adiletta et al. (2016a,b), Atarés et al. (2008), Doymaz (2010), Junqueira et al. (2017) and Vásquez-Parra et al. (2013) used different pre-treatments which contributed to the improvement of structure preservation as indicated by the greater rehydration capacity during the reconstitution of dried food products.

In conclusion, during the reconstitution of dried apples, the pre-treatment contributed to the improvement of structure preservation as indicated by the greater rehydration capacity and lower shrinkage.
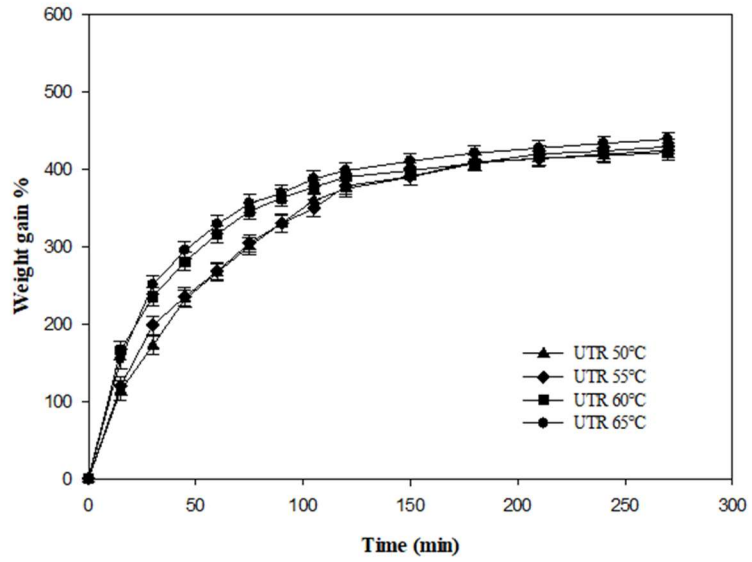
a)
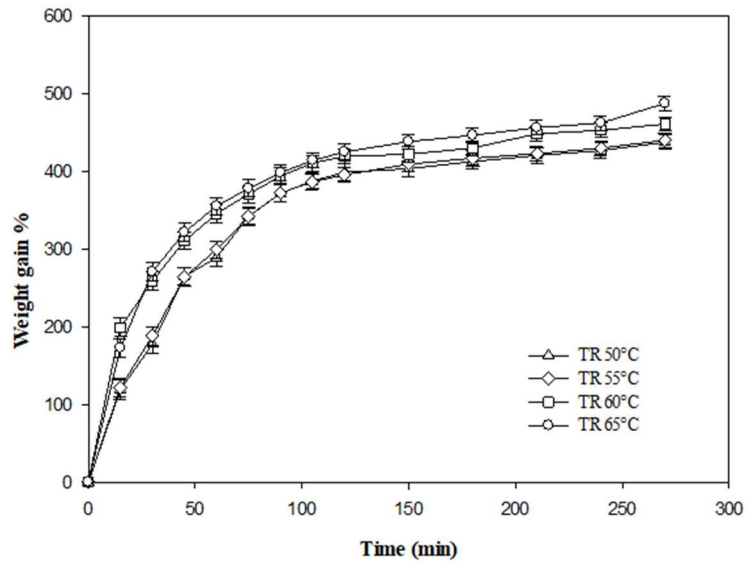
b)

Figure IV.11 *Rehydration kinetics of untreated (a) and treated (b) dried samples (50, 55, 60 and 65°C) at rehydration temperature of 30°C*
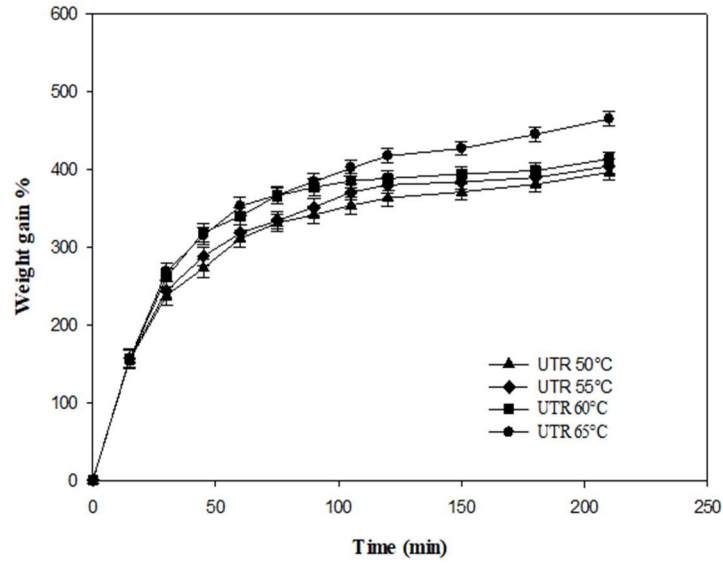
a)
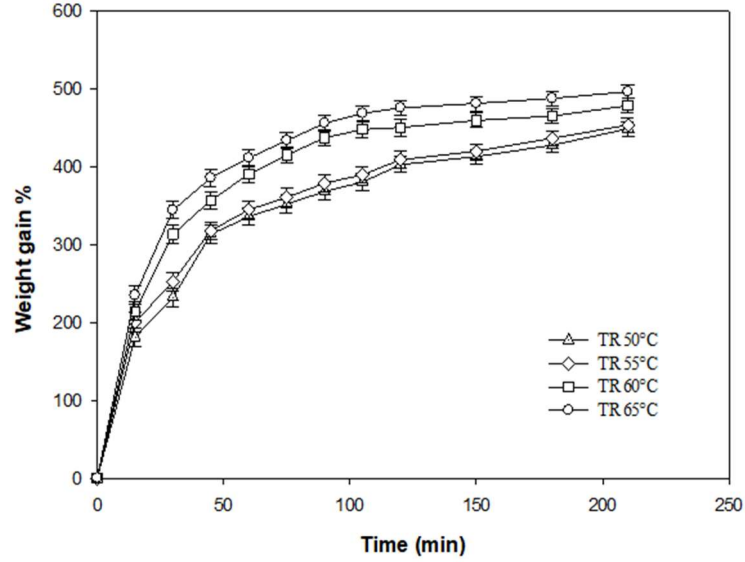
b)

Figure IV.12 *Rehydration kinetics of untreated (a) and treated (b) dried samples (50, 55, 60 and 65°C) at rehydration temperature of 70°C*
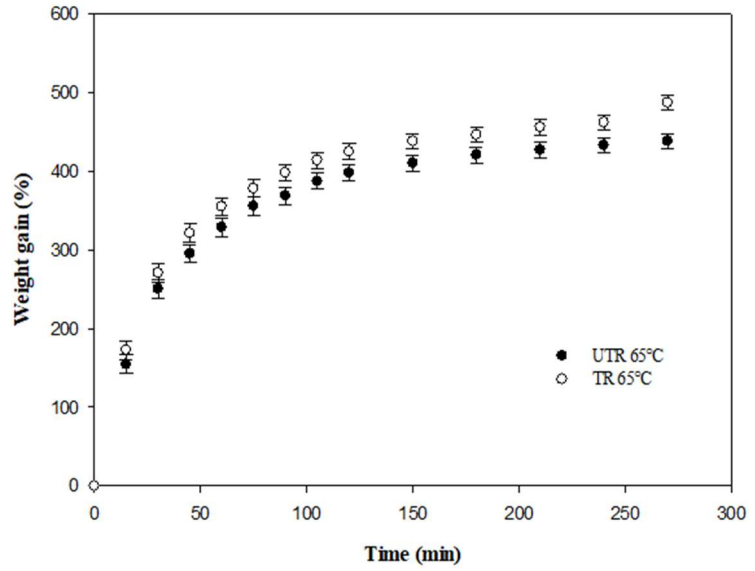
Figure IV.13*Rehydration kinetics at 30°C of both untreated (UTR) and treated (TR) samples dried at 65°C*
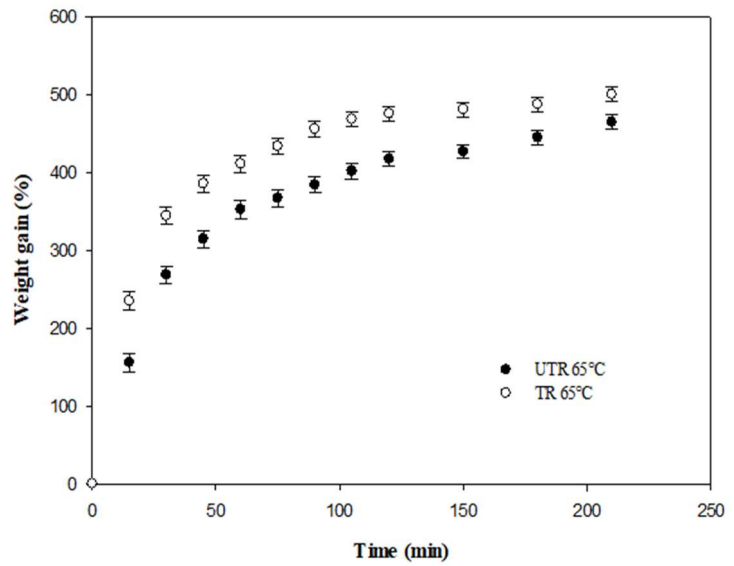
Figure IV.14*Rehydration kinetics at 70°C of both untreated (UTR) and treated (TR) samples dried at 65°C*

In order to have more information about the amount of absorbed water, the amount of removed solutes, and the degree of cellular and structural disruption during rehydration, four quality indices were investigated (Lewicki,1998; Barrera et al., 2016), as follows: (1) water absorption capacity, WAC; (2) dry matter holding capacity; DHC, (3) rehydration ability, RA; and (4) water holding capacity, WHC. The WAC index explains the ability of food material to absorb water that replaces the water removed during drying. The DHC index is a measure of food material's ability to hold soluble solids after rehydration; it gives information on tissue destruction and on tissue permeability to solutes. The RA index describes the rehydration ability of dried product, and it indicates the total tissue destruction caused by both drying and rehydration conditions (Maldonado et al., 2010). The WHC index measures the ability of product to maintain its own and added water during the rehydration process. Also, WHC has an important role in the food structure modifications (Zayas, 1997).

In Table IV.6 and Table IV.7, the rehydration quality indices (WAC, DHC, RA and WHC) of both untreated (UTR) and treated (TR) dried samples(50, 55, 60 and 65°C) at rehydration temperatures 30 and 70°C, respectively, were reported. At the rehydration temperature of 30°C, the WAC and DHC indices showed significantly ($p<0.05$) higher values for treated samples dried at 60 and 65°C. This trend indicated that the treated samples were able to absorb more water at low rehydration temperature with regard to the untreated ones. Similar findings were reported by Barrera et al. (2016) which found higher values of WAC and DHC indices in rehydrated apples treated previously with vacuum impregnation with sucrose solution. They stated that these higher indices are correlated to the higher rehydration ability of apple samples. Furthermore, the highest ability to rehydrate (RA index) and the highest WHC values were observed in treated samples dried at 60 and 65°C and rehydrated at 30°C. This phenomenon could be explained by the development of a porous structure at higher drying temperatures, which increases the ability to absorb the water during rehydration especially at lower rehydration temperature (30°C). Similar WHC results were obtained by Zura–Bravo et al. (2013), which found that the highest rehydration temperature (60°C) resulted in lower WHC of rehydrated apple slices. Accordingly, increasing rehydration temperature leads to texture damage likely due to the fact that the breaking or the denaturation of polysaccharides of cell wall, promoting a remarkable reduction of mechanical resistance in the apples.

Barrera et al. (2016) revealed that the vacuum impregnation (VI) with an isotonic sucrose solution significantly improved rehydration process of apple. This explanation was confirmed by higher values of WAC, DHC and WHC reached by VI sucrose samples.

After rehydration experiments at higher temperature 70°C, no significant differences ($p > 0.05$) were found in the following indices: DHC, RA, WHC

between all untreated and treated samples; only the treated samples dried at 60°C showed the highest WAC value.

The combination of lower rehydration temperature (30°C) and higher drying temperatures (60 and 65°C) with this novel pre-treatment was proven to be useful to preserve the rehydrated apple structure by reducing cellular damage and promoting the absorption of great amount of water.

Table IV.6 *Rehydration indices of both untreated (UTR) and treated (TR) dried samples (50, 55, 60 and 65°C) at rehydration temperatures of 30°C. Different superscript letters (a, b, c, etc.) in the same column mean significant differences (p < 0.05)*

| Rehydration at 30°C	WAC	DHC	RA	WHC
UTR 50°C	0.781 ± 0.03^a	0.233 ± 0.01^a	0.182 ± 0.02^a	0.794 ± 0.01^a
TR 50°C	0.806 ± 0.02^{ab}	0.243 ± 0.00^a	0.196 ± 0.01^{ab}	0.822 ± 0.02^{ab}
UTR 55°C	0.784 ± 0.02^a	0.240 ± 0.01^a	0.188 ± 0.01^{ab}	0.796 ± 0.06^a
TR 55°C	0.827 ± 0.03^{ab}	0.247 ± 0.00^a	0.204 ± 0.01^{ab}	0.859 ± 0.0^{abc}
UTR 60°C	0.816 ± 0.01^{ab}	0.241 ± 0.01^a	0.197 ± 0.03^{ab}	0.844 ± 0.0^{abc}
TR 60°C	0.847 ± 0.02^b	0.268 ± 0.00^b	0.227 ± 0.02^{ab}	0.902 ± 0.02^c
UTR 65°C	0.831 ± 0.02^{ab}	0.243 ± 0.01^a	0.202 ± 0.01^{ab}	0.877 ± 0.02^{bc}
TR 65°C	0.853 ± 0.01^b	0.274 ± 0.00^b	0.234 ± 0.01^b	0.911 ± 0.02^c

Table IV.7 *Rehydration indices of both untreated (UTR) and treated (TR) dried samples (50, 55, 60 and 65°C) at rehydration temperatures of 70°C. Different superscript letters (a, b, c, etc.) in the same column mean significant differences (p < 0.05)*

Rehydration at 70°C	WAC	DHC	RA	WHC
UTR 50°C	0.767 ± 0.02^a	0.222 ± 0.02^a	0.177 ± 0.01^a	0.807 ± 0.02^a
TR 50°C	0.811 ± 0.02^{ab}	0.236 ± 0.00^a	0.191 ± 0.01^a	0.825 ± 0.02^a
UTR 55°C	0.799 ± 0.03^{ab}	0.213 ± 0.00^a	0.170 ± 0.02^a	0.813 ± 0.14^a
TR 55°C	0.822 ± 0.02^{ab}	0.237 ± 0.02^a	0.195 ± 0.03^a	0.823 ± 0.03^a
UTR 60°C	0.801 ± 0.02^{ab}	0.218 ± 0.01^a	0.181 ± 0.02^a	0.821 ± 0.02^a
TR 60°C	0.830 ± 0.01^b	0.241 ± 0.02^a	0.200 ± 0.00^a	0.870 ± 0.02^a
UTR 65°C	0.790 ± 0.01^{ab}	0.240 ± 0.02^a	0.190 ± 0.00^a	0.843 ± 0.01^a
TR 65°C	0.817 ± 0.01^{ab}	0.239 ± 0.01^a	0.195 ± 0.01^a	0.879 ± 0.01^a

IV.1.10 Diameter and thickness evolution

Diameter and thickness evolution is another important factor that should be analyzed during the rehydration process of apples. The average diameter and thickness of all dried apples were evaluated during the rehydration process at 30 and 70°C. All samples had similar increasing trend of the diameter and thickness during the experiments at 30 and 70°C. In Figure IV.15(a,b) and Figure IV.16(a,b) the diameter and thickness of untreated and treated samples dried at 65°C were presented.Obviously, the fastest increment of diameter and thickness took place in the initial period of the rehydration process. In the further stage of process, water absorption slowed down since rehydrated samples got close to the state of balance with equilibrium moisture content.

There were significant increments of diameter and thickness of both samples up to 120 min of the rehydration process (for 30 and 70°C). Higher increases in diameter and thickness were observed in treated samples than untreated ones at both temperatures. Moreover, the diameter changes were more significant than thickness ones in all investigated samples at both rehydration temperatures. Among the two investigated temperatures, the best temperature was 30°C with regards to the increment of diameter-thickness of the samples.

The reconstitutability of food products depends principally on the internal structure of the dried samples. Increasing rehydration temperature causes a deterioration of the structure that intensifies the damage caused during thermal dehydration and promotes significant loss of mechanical resistance and elasticity behaviours in the samples. It was seen that, treated apples had a homogeneous structure which was protected by the pre-treatment and upon rehydration its functionality was restored. This can be explained considering that the trehalose solution replaces water in membrane and prevent the phase transition in dried apple slabs. Moreover, trehalose has the ability to form glasses, and direct interaction between the sugar and polar groups in proteins and phospholipids would indicate that the cellular structure is maintained and stabilized (Betoret et al., 2015; Crowe et al., 1988; Lewicki, 1998).

Several studies have been investigated the impact of pre-treatments and the drying/rehydration conditions on the dimensional changes of dried fruit and vegetable during their rehydration. Winiczenka et al. (2014) found that the temperature of drying influenced the relative increase of the volume of dried apples during rehydration at 20°C.According to Bilbao-Sáinz et al. (2005), apple cylinders dehydrated in microwave oven with vacuum impregnation showed that the percentage of recovered volume of impregnated samples was higher than the percentage of recovered volume of non-impregnated ones. A justification for this result is that the vacuum impregnation implies the release of the major part of the initial gas (air)

present in the pores and sample gains the mass by the entrance of isotonic solution of the apple juice in the pores.
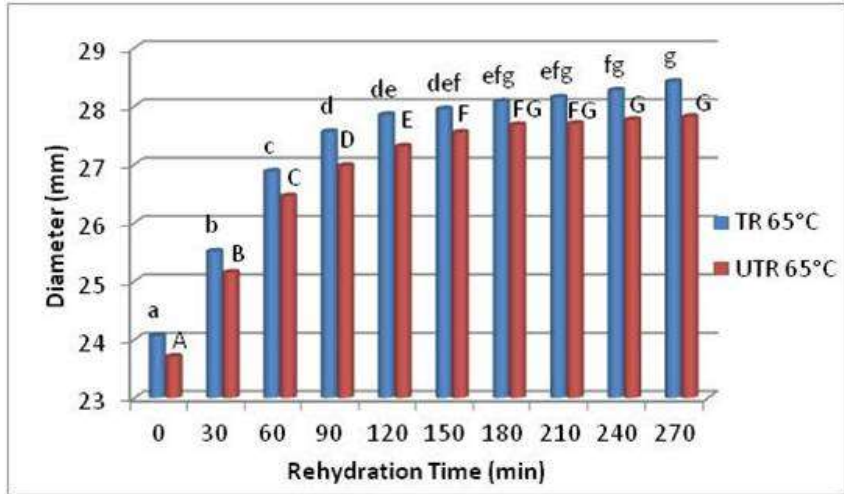
a)
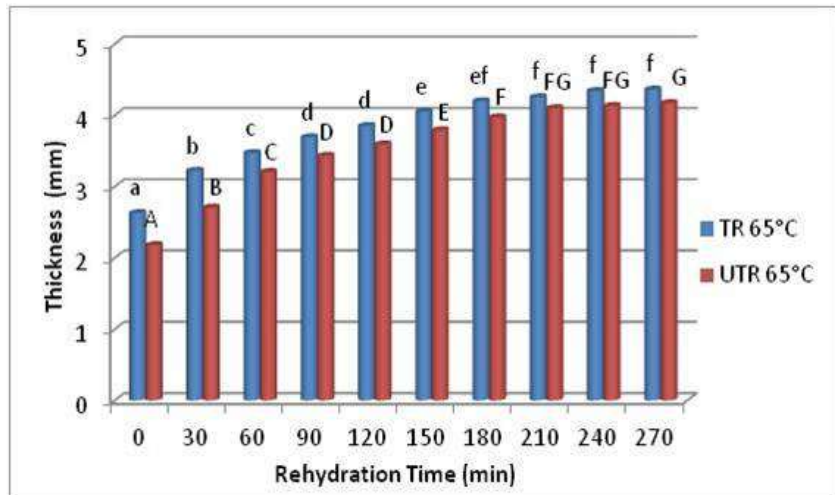
b)

Figure IV.15*Diameter (a) and thickness (b) of untreated (UTR) and treated (TR) rehydrated samples dried at 65°C during rehydration at 30°C.*
Mean values in treated samples with different lower letters (a, b, c....) are significantly different (p < 0.05) during the rehydration time, and mean values in untreated samples column with different capital letters (A, B, C...) are significantly different (p < 0.05) during the rehydration time.
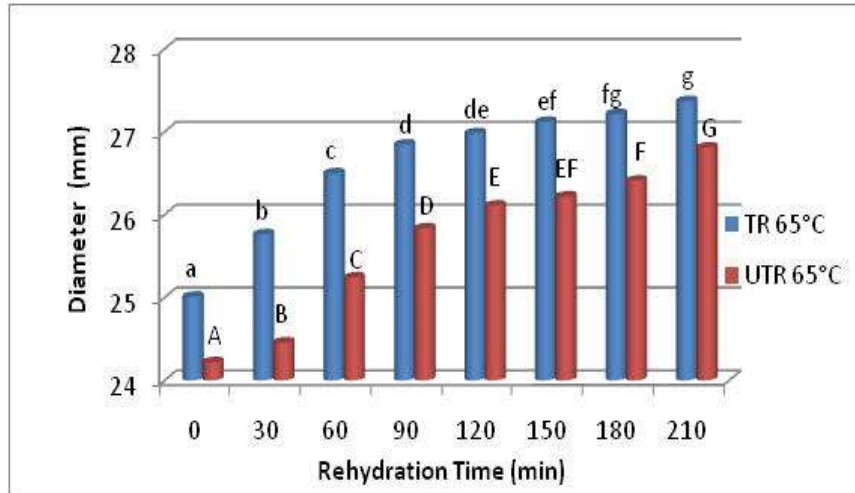
a)
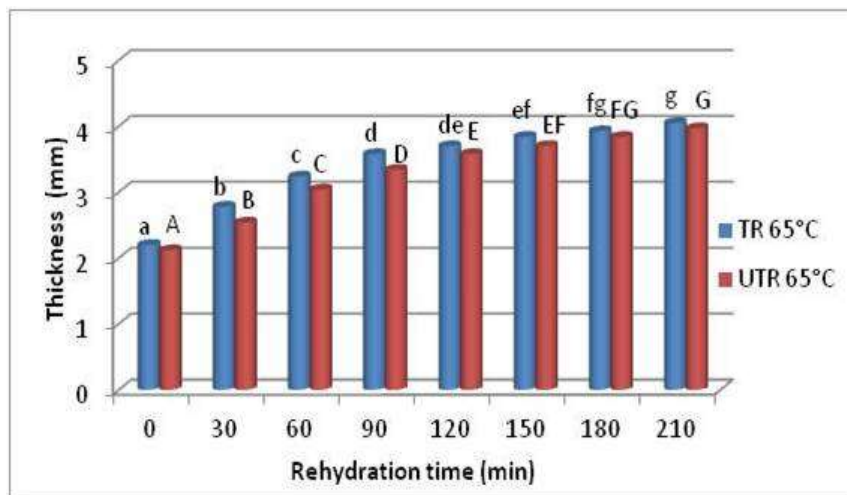
b)

Figure IV.16 *Diameter (a) and thickness (b) of untreated (UTR) and treated (TR) rehydrated samples dried at 65°C during rehydration at 70°C.*
Mean values in treated samples with different lower letters (a, b, c….) are significantly different (p < 0.05) during the rehydration time, and mean values in untreated samples column with different capital letters (A, B, C…) are significantly different (p < 0.05) during the rehydration time.

IV.1.11 Colour evaluation

Colour parameters are also important as quality indices for rehydrated food products and they should closely resemble the colour characteristics of fresh food material to increase consumer acceptability (Cox et al., 2012). The evaluation of rehydration conditions with the aim of minimizing the colour changes during drying/rehydration process has great importance from an economic viewpoint. The effects of pre-treatment and drying/rehydration temperatures on surface colour of fresh and rehydrated apple slabs were shown in Table IV.8. Lightness (L*) and white index (WI) were reported. According to the results, the colour parameters of rehydrated apple slabs were significantly different (p < 0.05) in relation to fresh ones. It was observed a decrease in L* and WI values in all samples, indicating a reduction of brightness compared to fresh apples.This is an unexpected result which may be attributed to the fact that an increase in water gain would normally lead to a higher luminosity (Gowen et al., 2006; Moreira et al., 2008). Based on these results, it is argued that some modifications in the optical properties of the apples occurred during the rehydration. Oxidation processes or other chemical reactions such as Maillard reactions probably led to the formation of browning agents (Deng et al., 2017; Gowen et al., 2006; Lemus – Mondaca et al., 2009; Moreira et al., 2008). Treated rehydrated samples had higher L* and WI values than untreated rehydrated ones at both rehydration temperatures (30 and 70°C), demonstrating that the pre-treatment was able to maintain the colour of the final rehydrated apples. Furthermore, drying temperatures play an important role on colour attributes of dried products, as well as, of rehydrated foodstuffs (Link et al., 2017). The higher drying temperature retained the best colour preservation in terms of lightness and white index at both rehydration temperatures. On the contrary, Moreira et al. (2008) and Zura-Bravo et al., (2013) for rehydrated chestnut and apples, respectively, showed that the values of L* decreased as rehydration temperature increased.

It can be concluded that, the combination of pre-treatment with higher drying temperatures (60 and 65°C) had a significant effect on the colour of rehydrated samples, while both used rehydration temperatures did not significantly influence on the colour changes of rehydrated slabs.

Table IV.8 *Colour parameters for untreated (UTR) and treated (TR) fresh and rehydrated samples (drying temperatures, 50, 55, 60 and 65°C) at rehydration temperatures 30 and 70°C. Values with different letters in a given column are significantly different (p < 0.05).*

Sample	L*	WI
Fresh Apples		
UTR Fresh	81.73 ± 0.66^f	72.13 ± 1.79^g
TR Fresh	84.79 ± 2.89^f	79.61 ± 1.02^h
Rehydrated Apples at 30°C		
UTR 50°C	53.26 ± 1.97^a	45.30 ± 2.00^a
TR 50°C	60.45 ± 1.15^{bc}	59.48 ± 1.31^{cde}
UTR 55°C	53.29 ± 1.29^a	46.82 ± 0.59^a
TR 55°C	65.98 ± 0.77^{de}	58.31 ± 0.90^{cd}
UTR 60°C	57.64 ± 1.53^{ab}	45.68 ± 0.89^a
TR 60°C	66.31 ± 0.62^{de}	62.09 ± 0.34^{ef}
UTR 65°C	59.15 ± 2.79^{bc}	52.55 ± 1.52^b
TR 65°C	66.81 ± 0.25^{de}	62.88 ± 1.60^{ef}
Rehydrated Apples at 70°C	57.55 ± 2.15^{ab}	56.25 ± 1.93^{bc}
UTR 50°C	65.51 ± 1.71^{de}	62.57 ± 0.31^{def}
TR 50°C	59.35 ± 0.61^{bc}	57.27 ± 0.53^c
UTR 55°C	65.39 ± 0.55^{de}	64.74 ± 1.61^f
TR 55°C	62.77 ± 1.59^{cd}	59.71 ± 0.51^{cde}
UTR 60°C	66.12 ± 0.80^{de}	66.01 ± 1.23^f
TR 60°C	60.31 ± 0.50^{bc}	58.13 ± 1.38^{cd}
UTR 65°C	68.13 ± 0.18^e	65.75 ± 1.83^f
TR 65°C		

IV.1.12 DPPH radical scavenging activity

The knowledge of the content and stability of apple antioxidant components after rehydration treatments is essential to assess the nutritional values prior to its consumption (Cox et al., 2012). The radical scavenging activity of fresh and rehydrated apple slabs for both untreated and treated samples was shownin Figure IV.17 at the two rehydration temperatures (30 and 70°C). As expected, the lowest EC_{50} values (the highest antioxidant activity) were determined as 16.06 and 18.66 mg/mL db in treated and untreated fresh apples, respectively.

The antioxidant activity of all untreated and treated rehydrated samples decreased after both rehydration processes at 30 and 70°C. For treated apples

rehydrated at 30°C the antioxidant activity was better retained rather than at higher rehydration temperature (70°C). The pre-treatment combined with the lower drying temperatures (50 and 55°C) and the lower rehydration temperature (30°C) can better preserve the antioxidant activity of rehydrated apple slabs. This behaviour can be explained considering that during the drying and rehydration processes, modifications of the chemical structure of the main antioxidant compounds in apple (i.e, chlorogenic acid, quercetin, gallic acid, α- tocopherol etc.) or interactions between antioxidant compounds and other apple constituents, such as proteins at higher temperatures occurred(Önal et al., 2019). In addition, trehalose –dried and rehydrated foods have a higher nutritional content than conventionally processed foods (Colaça and Roser, 1994). In our case, lower rehydration temperature (30°C) had a positive effect on the antioxidant activity of rehydrated apples. This trend may be explained by higher water temperatures which promoted significant loss of antioxidant compounds also into the water. Similar results were obtained by Moldanado et al. (2010) which reported that increases in temperature above 40°C resulted in higher loss of solid compounds.

In contrast to these findings, Cox et al. (2012) evaluated the effect of the rehydration temperatures (20, 40, 60, 80 and 100°C) on the antioxidant activity of seaweed and reported the highest percentage increase was seen in seaweed rehydrated at 80°C which increased to 100% after 20 min (24.3% increase). Higher rehydration temperatures positively effected on the radical scavenging activity of seaweeds. These results indicated that there was a temperature dependence in the case of antioxidant activity. Zura – Bravo et al., (2013) also found a higher antioxidant activity of rehydrated apple slices (*Granny Smith*) at higher rehydration temperatures (60°C) rather than at lower temperatures (20 and 40°C). This increment at a higher rehydration temperature may be associated with the generation and accumulation of Maillard-derived melanoidins which have a varying degree of antioxidant activity. Thus, the higher temperature promoted the water penetration and immediately more antioxidants were released, resulting in a high DPPH activity (Miranda et al., 2009; Vega-Gálvez et al., 2009).
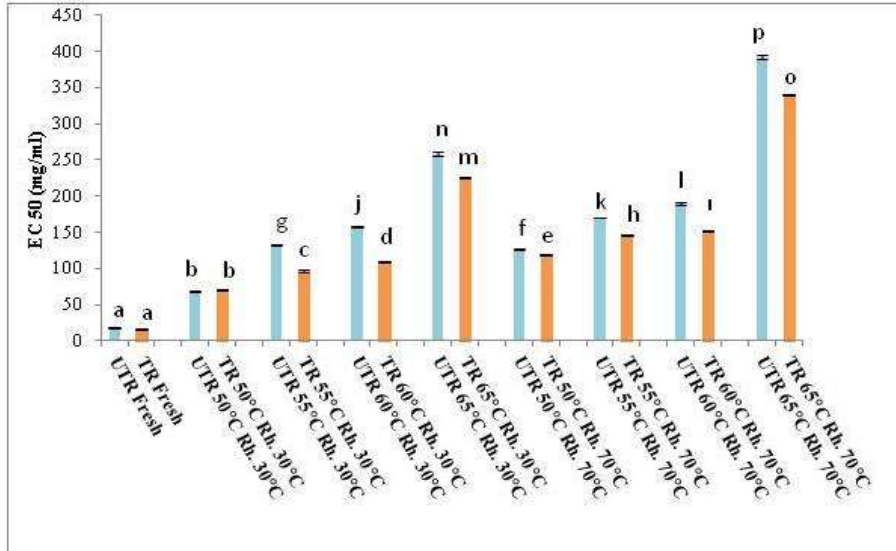
Figure IV.17 *Antioxidant activity of untreated (UTR) - treated (TR) fresh and rehydrated apples (drying temperature, 50,55,60 and 65°C) at 30°C and 70°C*

IV.1.13 Effect of pre-treatment, drying/rehydration temperaturesby

using Principal Component Analysis (PCA)

The effect of novel pre-treatment and drying/rehydration temperatures on the qualitative traits of rehydrated apples were evaluated by PCA analysis. Covariance matrix showed that the eigenvalues accounted for 67.63% of the total variance in the dataset using two principal components (PCs). PC1 explained 38.52% of the variance in the dataset, whereas PC2 explained an additional 29.11% of the variance. All loadings and scores were shown in the same PCA plot (Figure IV.18).

WAC (R^2= 0.844), DHC (R^2=0.851), RA (R^2= 0.831), WHC (R^2= 0.590) were positively correlated to PC1; while L* (R^2= 0.807), WI (R^2=0.876), EC50 (R^2=0.585) indicated a positive correlation to PC2.

As shown in PCA plot, according to the rehydration temperature of 30°C, treated slabs dried at 50 and 55°C were more similar than those dried at 60 and 65°C. Furthermore, those dried at 60 and 65°C were more correlated with quality parameters in terms of WAC, DHC, RA and WHC along PC1, indicating that the drying temperature had significant impact on the ability to reconstitute the water content of apples during the rehydration process. At the same rehydration temperature (30°C), untreated apples dried at 50 and 55°C were closer to each other than untreated apples dried at 60 and 65°C.

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As it is seen from Figure IV.18, these latters (UTR dried at 60 and 65°C) were more correlated with the quality parameters of rehydrated apples along PC2, i.e. colour parameters. It was clear that the higher drying temperatures (60 and 65°C) showed the better rehydration results in both UTR and TR apples.

With regards to the rehydration temperature of 70°C, treated dried samples at 50, 55 and 60°C were similar. Scoring and loading plot enabled to differentiate the behaviour only for treated apples dried 65°C which were more correlated with PC2.Untreated samples rehydrated at 70°C shifted from negative values to positive ones along PC2.

In conclusion, higher rehydration temperature (70°C) negatively affected quality parameters of rehydrated apples. The samples rehydrated at 30°C showed the better correlations with quality parameters.
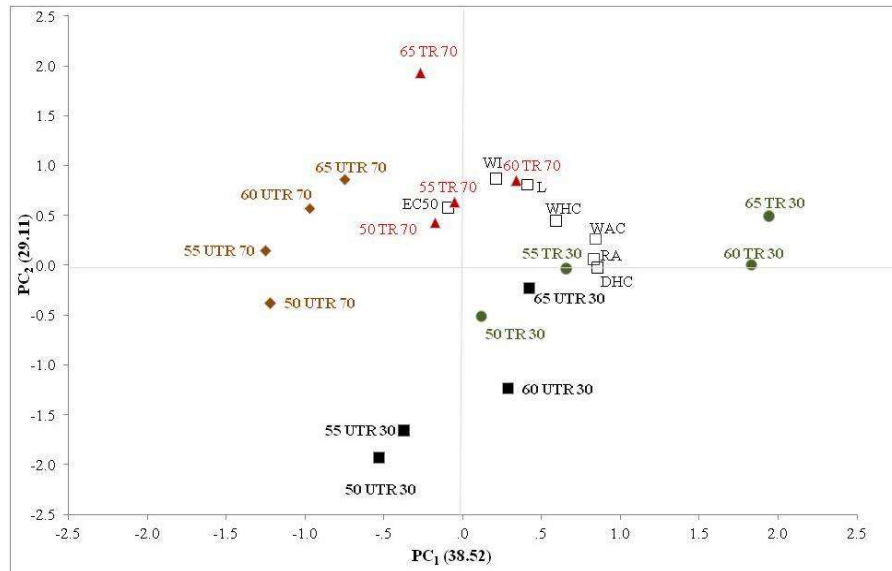
Figure IV.18 *Two-dimensional principal component analysis of rehydration properties for untreated (UTR) and treated (TR) samples dried at 50, 55, 60 and 65°C and rehydrated at 30 and 70°C. (WI = white index; L* = lightness; WAC = water absorbtion capacity; DHC = drying matter holding capacity; RA = rehydration ability; WHC = water holding capacity; EC50 = antioxidant activity).*

IV.1.14 Conclusions

The effect of air-drying temperature (50, 55, 60, and 65°C), combined with a novel dipping pre-treatment including trehalose, on drying behaviour and quality parameters of hot air drying 'Annurca' apple slabs was studied. Apples' drying process was influenced by both pre-treatment and temperature. At fixed drying temperature, treated samples had shorter drying times than untreated ones. Furthermore, the combination between pre-treatment and optimal drying temperature (65°C) allowed to obtain the best quality 'Annurca' slabs which exhibited the highest moisture loss in shorter drying time, the lowest colour changes, the better structure preservation with less shrinkage and higher rehydration capacity. On the contrary, retention of total phenolic content and antioxidant activity of samples decreased by increasing drying temperatures: the highest values were found in treated samples dried at 50°C. On the other hand, sensory evaluation indicated that the solution pre-treatment was also effective to obtain dried apples with better overall characteristics. This study may be useful to provide information about the development of innovative and natural pre-treatment conditions before drying of fruit and vegetables. Therefore, the combination of dipping pre-treatment and hot-air drying at 65°C seems to be a suitable process to produce high quality and healthy dried apple snacks for both consumers and food industry.

In addition, the influence of drying/rehydration process conditions and an alternative novel dipping pre-treatment on rehydration kinetics and quality attributes of rehydrated 'Annurca' apple slabs were studied. Rehydration temperatures (30 and 70°C) did not significantly effect on the weight gain of dried apples. On the other hand, lower rehydration temperature (30°C) with used pre-treatment had a positive effect on the rehydration indices (WAC, DHC, RA and WHC). Higher increases in diameter and thickness were observed in treated rehydrated samples respect to the untreated ones. The pre-treatment at both rehydration temperatures was able to preserve the colour properties. The lower the drying temperature and the lower rehydration temperature, the higher antioxidant activity for both samples. The results clearly highlighted the rehydration process and quality of rehydrated apples are influenced by pre-treatment, drying and rehydration temperatures. The combination between dipping pre-treatment containing trehalose solution and optimal drying temperature (65°C) at a lower rehydration temperature (30°C) allowed to obtain the best overall reconstitution properties of the rehydrated apples in terms of the rehydration characteristics and structure. Thereby, it is recommended to combine an innovative and natural pre-treatment and drying/rehydration process conditions to achieve the high quality attributes of dried/rehydrated apples to meet consumer expectation.

IV. 2 Results of 'Terzarola gialla'peach slabs

IV.2.1 Drying kinetics and influence of pre-treatments on drying time

In order to determine the influence of different drying temperatures and pre-treatment on the drying kinetics of peach slabs, the curves of moisture ratio M_t/M_0 versus drying time (min) for untreated (UTR) and treated (TR) samples were presented in Figure IV.19(a-d).The average water activity of fresh peach was 0.98 ± 0.003. Peach slabs were dried up to the final moisture of 0.02 ± 0.01 kg water/kg (db) and to the water activity of lower than 0.40 ± 0.02. Drying air temperature had an important effect on the drying process. At higher drying temperatures, due to the quick removal of moisture, the drying time was less. The drying curves showed that moisture ratio decreased continuously with drying time. A constant-rate period was not exhibited in drying curves. Therefore, the entire drying process for peach slabs at the investigated drying temperatures (45, 50, 55, 60°C) occurred in the range of falling rate period. These results demonstrated that diffusion was the dominant mechanism governing the moisture movement in the peach slabs. Pre-treatment had a remarkable effect on the peach drying process: at each temperature, the treated samples reached the plateau M_t/M_0 in a shorter time. The drying time decreased significantly when the drying temperature increased. As it was seen from Figure IV.19(a-d), the plateau value of M_t/M_0 and the final water content were obtained at the following time: 540, 480, 465 and 390 min for untreated samples at 45, 50, 55 and 60°C, respectively. In comparison with these samples, at the same temperatures, treated slabs reached the plateau value at reducing time: 480, 390, 360 and 330 min, respectively. According to results, this new pre-treatment enhanced contribution to increase the permeability of the cell membranes of peach slices, leading to an increase in water diffusivity.

Similar results were obtained by different authors on drying process of peach fruit. Doymaz and Bilici (2014) evaluated the effect of air drying temperatures (45, 55, 65, 75°C) and citric acid pre-treatment on the drying process of peach slices. The drying process affected by drying temperatures and pre-treatment. Peach samples dipped in a citric acid solution prior to drying had shorter drying time in comparison to control ones. Also, Kingsly et al. (2007) reported that in both air drying temperatures (55°C and 65°C), pre-treated samples (blanched with 1% potassium meta bisulphite and blanched with 1% ascorbic acid) took less time for drying than the untreated samples. The mass transfer increased after treatment with both chemicals.

a)

b)
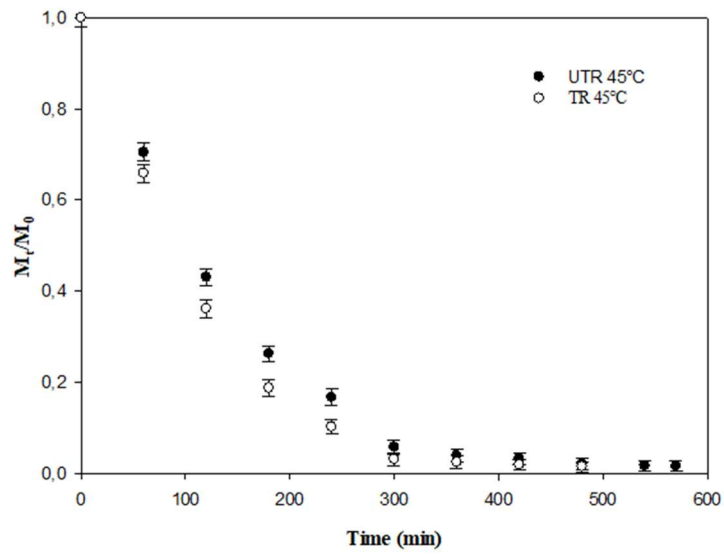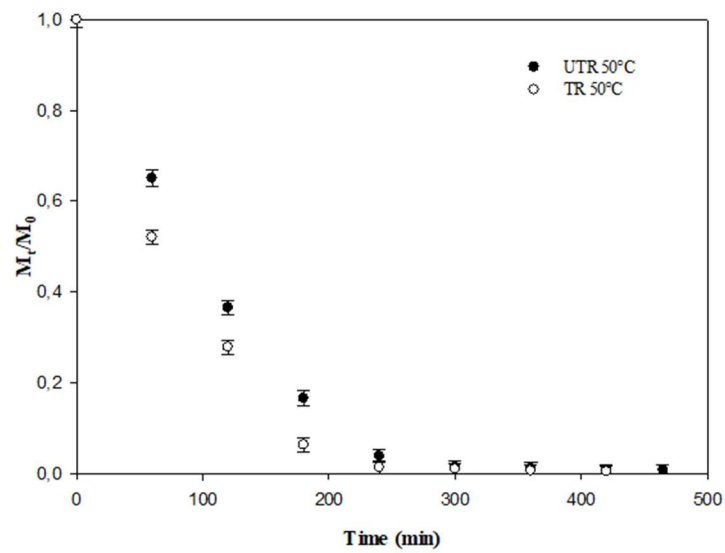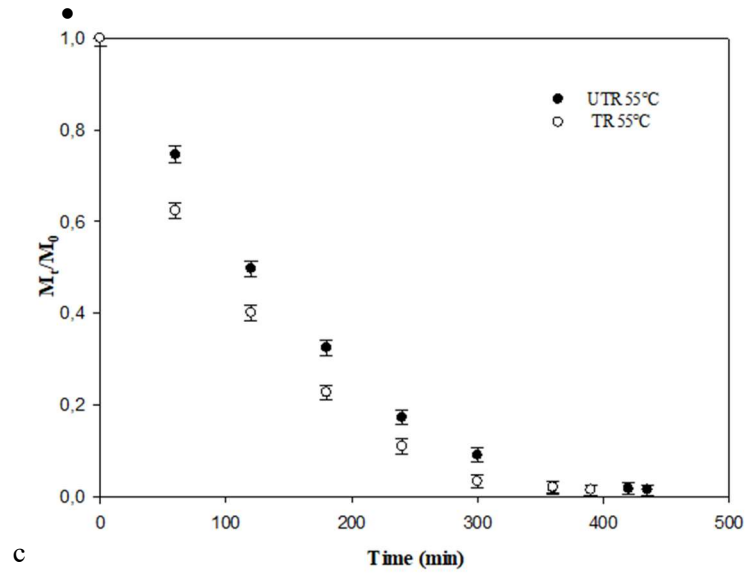
c

c)
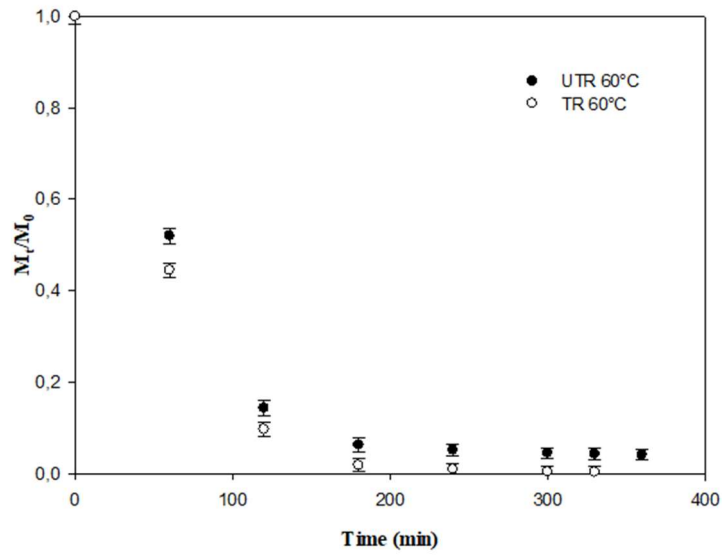
d)

Figure IV.19 *Experimental drying curves of untreated (UTR) and treated (TR) peach samples dried at 45°C (a), 50°C (b), 55°C (c) and 60°C (d)*

IV.2.2 Mathematical Modelling

The drying kinetics of peach slices at different temperatures have reported in terms of moisture ratio in Figure IV.19(a-d) and discussed in part IV.2.1.

The Figure IV.20 showed the comparison between experimental and model results for untreated (UTR) peach slabs at four drying temperatures of 45, 50, 55 and 60°C. The obtained results highlighted that the model was able to describe the experimental drying kinetics at those temperatures of 45, 50, and 60°C. However, at drying temperature of 55°C, there was a behaviour different from the other temperatures of the experimental data. A good agreement was found between the experimental data and predicted results with R^2 values ranging from to 0.95 to 0.99.

The Figure IV.21 reported the comparison between experimental data and simulation results for the treated (TR) samplesat four drying temperatures of 45, 50, 55 and 60°C.The comparison between experimental and prediction results was positive for all temperatures,but in any case the behaviour of drying temperature of 55°C was different from other temperatures. The predicted results demonstrated the moisture ratio values banded along the straight line which enabled the suitability of this model for describing the drying characteristics of treated peach samples.
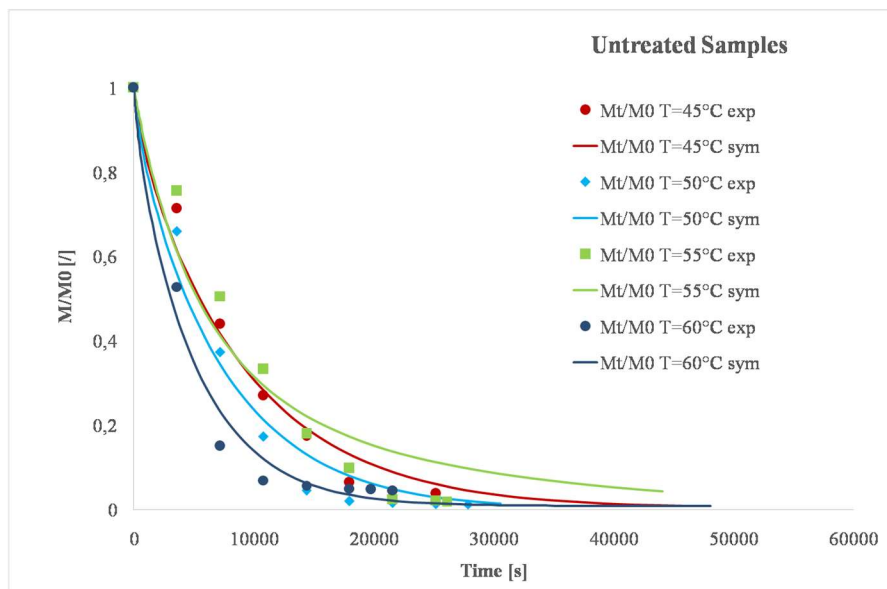
Figure IV.20 *Experimental(symbols) and simulated (lines) drying curves of untreated peach samples dried at 45°C, 50°C, 55°C and 60°C*
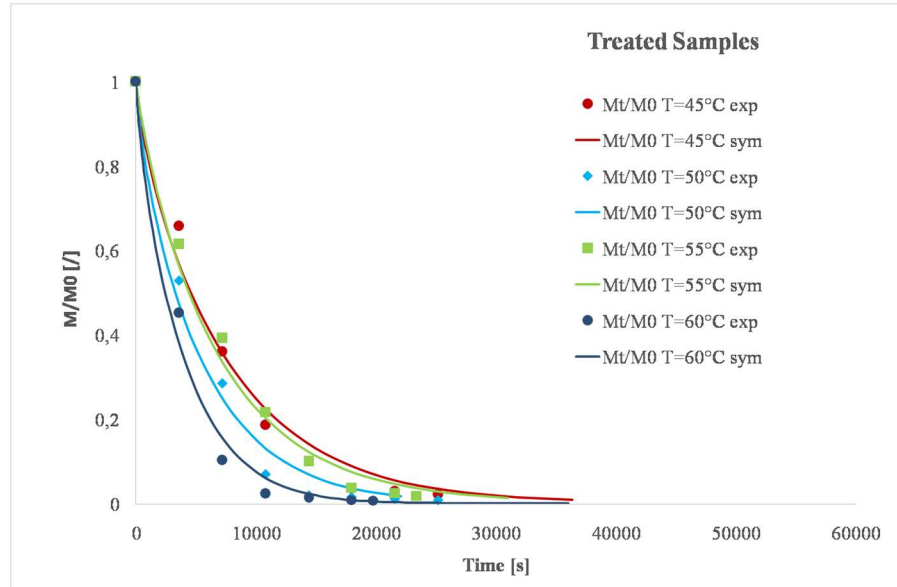
Figure IV.21 *Experimental(symbols) and simulated (lines) drying curves of treated peach samples dried at 45°C, 50°C, 55°C and 60°C*

According to results, a good fitting between the experimental data and predicted values was observed in treated peach samples respect to untreated ones with higher R^2 values ($R^2 > 0.98$).

Table IV.9 – and Table IV.10 reported the values of h_m, D_{eff} estimated by the model and the corresponding values of fitting parameters for untreated (UTR) and treated (TR) peach samples at all investigated temperatures, respectively. The values of the effective diffusion coefficient (D_{eff}) estimated by the model ranged from 1.90×10^{-10} to 3.40×10^{-10} for untreated samples and from 2.40×10^{-10} to 4.40×10^{-10} for treated samples in the temperature range of 45 - 60°C. As it is seen from Table IV.9 – and Table IV.10, D_{eff} values increased with increasing drying temperature. Moreover, the treated peach slices had higher moisture diffusivity values in comparison with the untreated peach samples. The increase of moisture diffusivity values (D_{eff}) in treated peach samples may be attributed to the partial chemical breakdown of the peach skins resulting in higher permeability of water. Another possible explanation could explained by the using of innovative pre-treatment which has affected the internal mass transfer during the drying process.According to the literature, the values of D_{eff} are reported to vary between $3.99 – 7 \times 10^{-10}$ m^2/s for the drying of peach slices in a temperature range of 50-70°C (Johnson and Ali Al Mukhaini, 2016)and 6.66×10^{-10} – 1.10^{-9} m^2/s for peach slices at 60 -80°C (Zhu and Shen, 2014). These results

were in agreement with the estimated D_{eff} values obtained from this study for both untreated and treated peach slices.

Table IV.9 *Effective diffusive coefficient (D_{eff}), h_m and R^2 values of untreated (UTR) peach slices*

Sample	T [°C]	D [m²/s]	h_m [m/s]	R²
UTR	45	1.90×10^{-10}	3.80×10^{-7}	0.99
	50	2.30×10^{-10}	4.60×10^{-7}	0.99
	55	3.00×10^{-10}	6.00×10^{-8}	0.95
	60	3.40×10^{-10}	6.80×10^{-7}	0.98

Table IV.10 *Effective diffusive coefficient (D_{eff}), h_m and R^2 values values of treated (TR) peach slices*

Sample	T [°C]	D [m²/s]	h_m [m/s]	R²
TR	45	2.40×10^{-10}	4.80×10^{-7}	0.99
	50	3.20×10^{-10}	6.40×10^{-7}	0.99
	55	3.60×10^{-10}	2.40×10^{-7}	0.99
	60	4.40×10^{-10}	8.80×10^{-7}	0.99

The effective moisture diffusivity can be related to temperature by Arrhenius-type relationship:

$$D_{eff} = D_0 \ \exp\left(-\frac{E_a}{RT}\right) \tag{41}$$

where D_0 is the effective moisture diffusivity at 273.15K, E_a is the active energy, R is the universal gas constant with 8.314 J/mol·K as its value, T is the drying temperature.

A plot of ln (D_{eff}) against $1/(T + 273.15)$ gave a straight line $R^2 = 0.985$ for untreated samples and $R^2 = 0.976$ for treated samples which was demonstrated in Figure IV.22. The slope (E_a/R) of the straight line was obtained by using Arrhenius relationship. The values of activation energy were found to be35.4 and 34.1 kJ/mol for untreated and treated peach samples, respectively.This behaviour indicated that a little less energy is required for the drying of treated peach samples respect to untreated ones hence, the pre-treatment aids moisture diffusion and evaporation and thus reduces slightly the energy involved in the drying process. These values were similar to those proposed in literature for peach drying as 25.92 kJ/mol (Johnson and Ali Al Mukhaini, 2016) and 42.53 kJ/mol (Zhu and Shen, 2014).
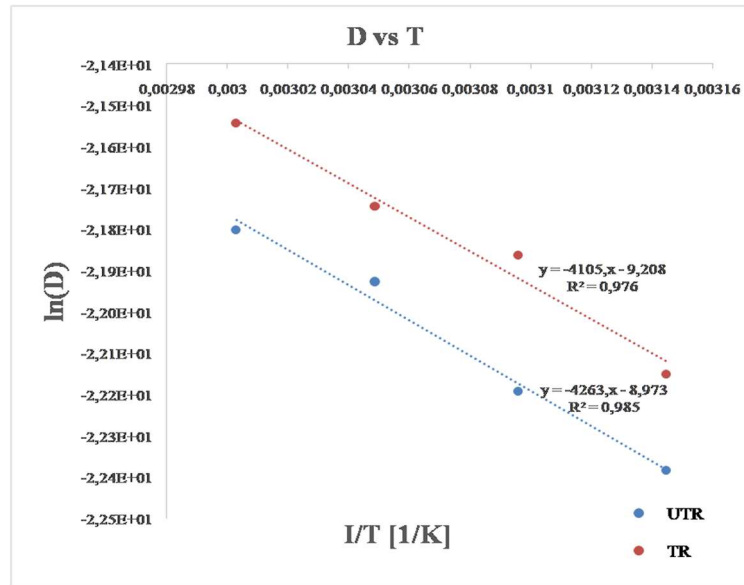
Figure IV.22 *Arrhenius-type relationship between diffusivity and reciprocal of absolute temperature*

IV.2.3 Colour evaluation

Colour is the major appearance attribute of dried fruits and vegetables. It is commonly employed as a tool of product standardization of consumer's satisfaction, as an indicator of biological and/or physico-chemical breakdown, and as a predictor of other quality characteristics (Ek et al., 2017). Unsuitable changes in colour of agricultural products would make low quality and marketing value (Krokida et al., 2000). Colour is also critically important in many dimensions of food choice and influences the perception of the other sensory characteristics by consumers (Taub and Singh, 1998). The effects of pre-treatment and drying temperatures on colour characteristics of fresh and dried samples were demonstrated in Table IV.11. Hunter value (L*) and Hue angle (H°) were presented. According to the L* values, no significant differences (p > 0.05) were observed between fresh and all untreated dried peach slabs. Treated peaches dried at 60°C had the higher L* valuesThere were no significant (p > 0.05) differences among the treated fresh peaches and treated samples dried at 55 and 60°C. On the contrary, the lowest L* value was found in untreated samples dried at 45°C and it is probably caused by longer drying times. These changes in brightness indicated that all the untreated dried peach samples were

darkerthan those treated dried ones. Colour deterioration may result from many reactions, but the most common ones are degradation of pigments, browning reactions and oxidation of ascorbic acid (Maskan, 2001).

The derived values of colour from Hunter values (L*, a* and b*), namely, hue angle and total colour differences provide more information concerning the colour degradation of fresh and dried peaches (Ek et al., 2017; Maskan, 2001). In our case, it seemed that, the most important parameter was determined as Hue angle (H°). Treated peach samples at investigated different temperatures (45,50,55 and 60°C) showed higher H° values in comparison with untreated ones after drying which means that the final untreated samples were browner than the fresh ones. These results may be explained by the long-time exposure to drying process at low temperature (45°C) and the enzymatic browning reactions that took place during the peach drying process, since the processing temperature and the duration of drying are crucial factors leading to colour deterioration.

The combination of novel pre-treatment and higher drying temperatures (55°C and 60°C) can better preserve the colour of peach slabs during the drying process.

Table IV.11 *Colour changes in untreated (UTR) and treated (TR) fresh and dried peaches at 45, 50, 55 and 60°C*

Sample	L*	Hue angle (H°)
UTR Fresh	72.28 ± 2.66[ab]	93.34 ± 2.31[fg]
TR Fresh	74.28 ± 1.53[b]	94.85 ± 1.04[g]
UTR 45°C	65.63± 1.12[a]	78.40 ± 2.81[a]
TR 45°C	72.92 ± 0.80[ab]	84.31 ± 0.89[bc]
UTR 50°C	70.65 ± 0.72[b]	81.18 ± 0.53[ab]
TR 50°C	71.68 ± 1.77[ab]	88.54 ± 2.31[d]
UTR 55°C	73.10 ± 1.49[ab]	82.90 ± 1.82[ab]
TR 55°C	74.84 ± 0.92[b]	89.22 ± 1.19[de]
UTR 60°C	73.08 ± 1.80[ab]	87.34 ± 1.78[cd]
TR 60°C	77.06 ± 0.40[b]	90.65 ± 0.38[def]

Values followed by the same letter, within the same column, significantly different ($p < 0.05$), according to Tukey's test

UTR 45°C   TR 45°C   UTR 50°C   TR 50°C

a)   b)

UTR 55°C   TR 55°C   UTR 60°C   TR 60°C

c)   d)
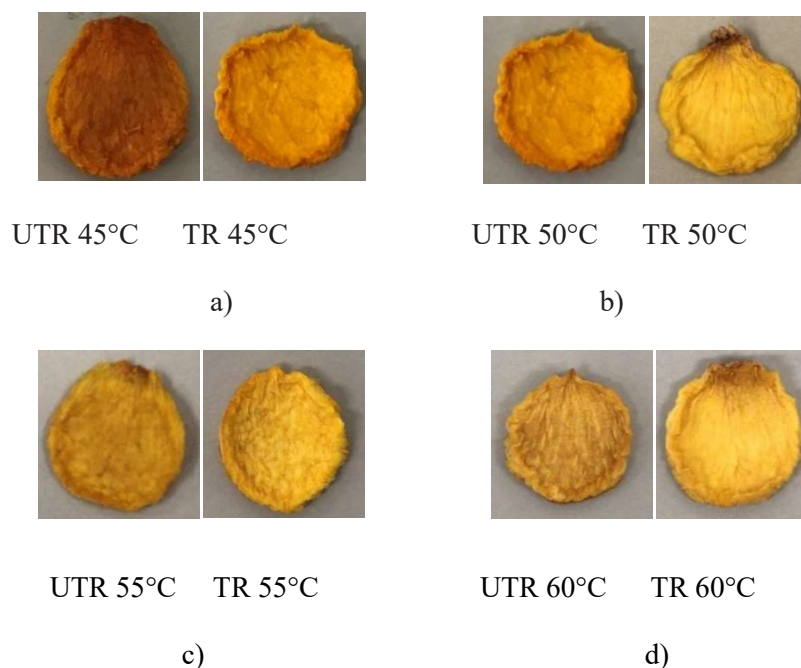
Figure IV.23 *Untreated (UTR) and treated (TR) 'Terzarola gialla' peach slabs dried at (a) 45°C, (b) 50°C, (c) 55°C (d) 60°C*

IV.2.4 DPPH radical scavenging activity

The radical scavenging activity of untreated (UTR)- treated (TR) fresh and dried peach slabs was given in Figure IV.24 at investigated different drying temperatures (45, 50, 55 and 60°C). The lowest EC_{50} values (the highest antioxidant activity) were found as 12.06 and 17.64 mg/mL db in treated and untreated fresh peaches, respectively. The scavenging activity of samples decreased significantly as increasing the drying temperature from 45 to 60°C, possibly due to antioxidant compounds being relatively unstable at higher drying temperatures. These results demonstrated that drying temperature affected adversely the antioxidant activity of peach slabs, especially when the sample is exposed at higher drying temperatures (55 and 60°C). Among the dried peaches, the highest antioxidant activity was observed as 23.29 mg/mL db in treated dried ones at 45°C. It was obviously seen that the treated samples at lower drying temperatures (45 and 50°C) the antioxidant activity was better retained than at higher drying temperatures. Analogous results were found for treated peach samples. This behaviour probably was due to the combined effect of exposure time and temperature: higher temperatures and longer exposure time, especially for untreated samples could cause to degrade phenolic compounds and result in

degradation of antioxidant activity. (Adiletta et al., 2016; Wojdylo et al., 2014). This trend of antioxidant activity could also be related to modifications of the chemical structure of the main antioxidant compounds in peach or interactions between antioxidant compounds and other peach constituents.

On the basis of the results in this study, the used novel dipping pre-treatment and the lower drying temperatures (45 and 50°C) can better improve the antioxidant activity of peach slabs.
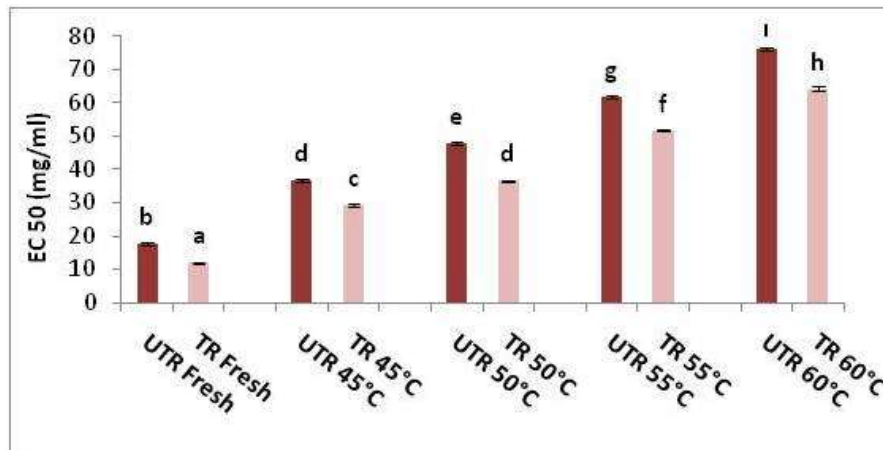
Figure IV.24 *Antioxidant activity of fresh and dried peach samples (45, 50, 55 and 60°C)*

IV.2.5 Shrinkage Evolution

Shrinkage of fruits and vegetables is a common physical phenomenon occurring simultaneously with moisture removal during the drying process and thus can reduce the drying rate as the moisture can be trapped in the dense cells and its movement toward the outer surface for subsequent removal is hindered (Mayor and Sereno, 2004; Zenoozian and Devahastin, 2009). In addition, shrinkage influences the physical properties of dried foodstuffs, such as rehydration capability, texture, appearance, shape, mechanical and elastic properties; all these changes in many cases may cause a negative impression on the consumers (Brasiello et al., 2013; Wang et al., 2018). Consequently, exploration of the shrinkage phenomenon is fundamental for a better understanding of the drying process and to control the product quality. Macroscopic shrinkage is commonly referred to the ratio of the volume of the products at a given drying time to the initial volume. In Figure IV.22(a-d) the variation of V_t/V_0 as a function of the relative moisture content (M_t/M_0) was represented. According to Figure IV.25(a-d), shrinkage

increased in all peach samples as the drying time advanced and more moisture was removed leading to a reduction in samples'volume.When moisture is removed from foodstuffs, a pressure is created between the inside and outside, generating contracting stresses that lead to material shrinkage (Junqueira et al., 2017; Parthasarathi and Anandharamakrishnan, 2014). During the drying process, at the same moisture content, treated peaches were less shrunk than untreated ones at all investigated temperatures (45, 50, 55 and 60°C). In our case, this novel pre-treatment promoted lower shrinkage, demonstrating a positive effect on the maintenance of the structure of the dried peaches.

Moreover, shrinkage decreased with increasing drying temperature from 45 to 60°C. These findings may be due to the higher drying rate at the higher drying temperatures (55 and 60°C) which cause the mechanical stabilization of the peach surface and limited degree of shrinkage.Similar results have been obtained for potato (Abbasi et al., 2011), pomegranate (Horuz and Maskan, 2015), grape (Adiletta et al., 2016a), hawthorn (Aral and Beşe, 2016) gooseberry fruit (Junqueira et al., 2017) and apple (Önal et al., 2019). Another possible explanation may be attributed to prolonged drying periods at lower drying temperatures (45°C) which leads to greater shrinkage (Horuz and Maskan, 2015; Khawas et al., 2015).

a)

b)
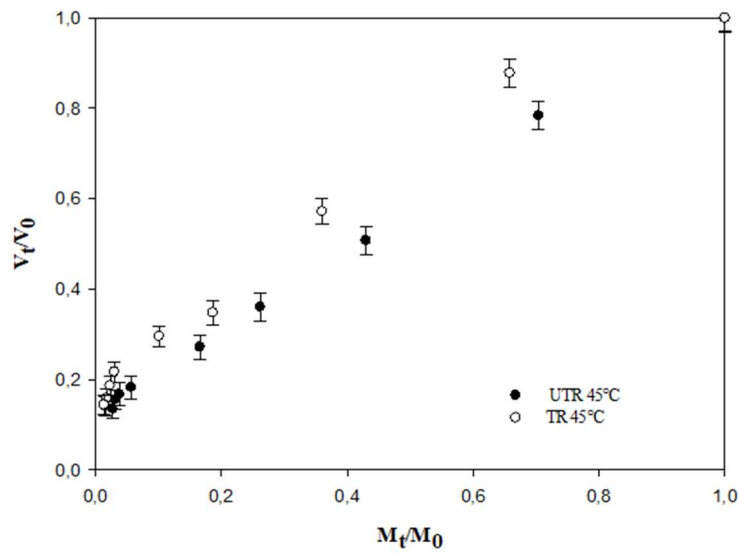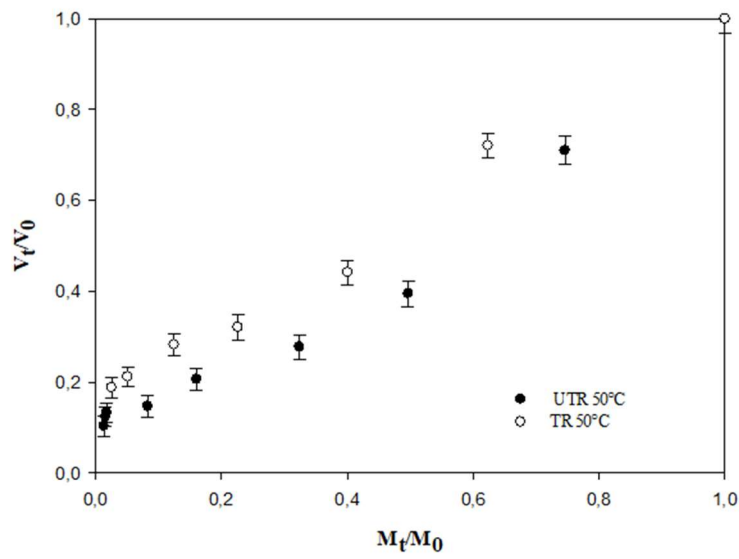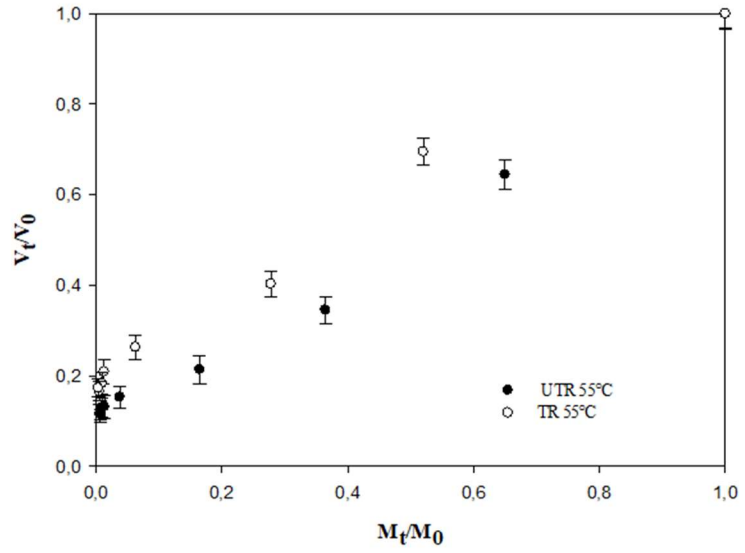
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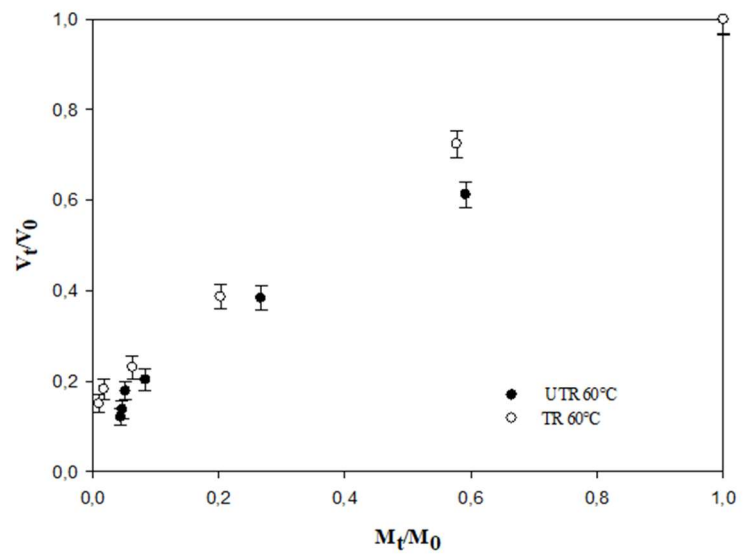
d)

Figure IV.25 *Experimental data of volume shrinkage of untreated(UTR) and treated (TR) peaches during drying at 45°C (a), 50°C (b), 55°C (c) and 60°C (d)*

IV.2.6 Rehydration Capacity

During reconstitution of dried foodstuffs, the amount and rate of water absorption determine to a considerable extent the sensorial properties and preparation time. c (Aral and Beşe, 2016; Horuz and Maskan, 2015).

The typical rehydration curves for peach slabs were reported in Figure IV.26(a-d). All rehydration curves indicated that an exponential trend with high water absorption rates mainly at the beginning of the rehydration process. However, as rehydration time progressed in the last stage of the process, the driving force for water transfer decreased when the pores are filled up and the system slowly attained equilibrium.

Based on these results, the rehydration process was affected by pre-treatment and drying temperatures (45, 50, 55 and 60°C). The treated peach samples exhibited a higher rehydration capacity in comparison with the untreated ones at all drying temperatures. In addition, rehydration capacity of dried samples increased with an increase in drying temperature (from 45°C to 60°C). The rehydration experiments demonstrated that rehydration capacity of treated samples dried at 60°C was higher than those of dried ones at other temperatures. This behaviour may be associated with rapid drying of these peach samples. Faster drying process caused the development of more porous structure in treated peaches at 60°C and this porosity allowed the higher water penetration. This result is similar to that reported by Aral and Beşe (2016), Doymaz and İsmail (2011), Junqueira et al. (2017).Slow or poor rehydrability of untreated samples may be attributed to internal collapse of the peach structure by prolonged drying times. Throughout the drying process, irreversible deformation and dislocation of cells may be observed. This induced a loss of integrity and therefore a dense structure of collapsed capillaries, and strong shrinkage. Such reduced hydrophilic properties would result in lower rehydration capacity of sufficiently absorbing required water for a complete rehydration (Al-Khuseibi et al., 2005).

The results are great interest, since the rehydration process aims to restore the properties of raw material. In our case, these results reflect the combined effect of an innovative solution and higher drying temperature (60°C) had a positive effect on the peach cellular structure during the rehydration process. Treated peaches dried at 60°C may have the best restoration properties that approximate the rehydrated samples to those of an in fresh product.

Chapter IV

a)

b)

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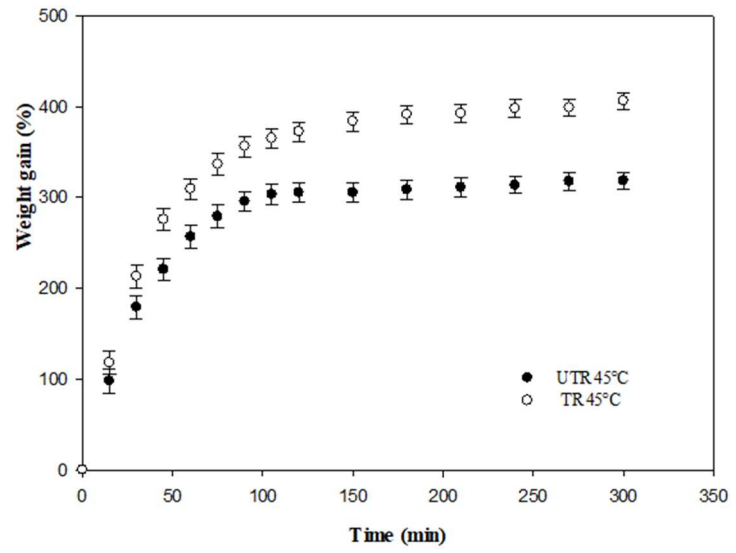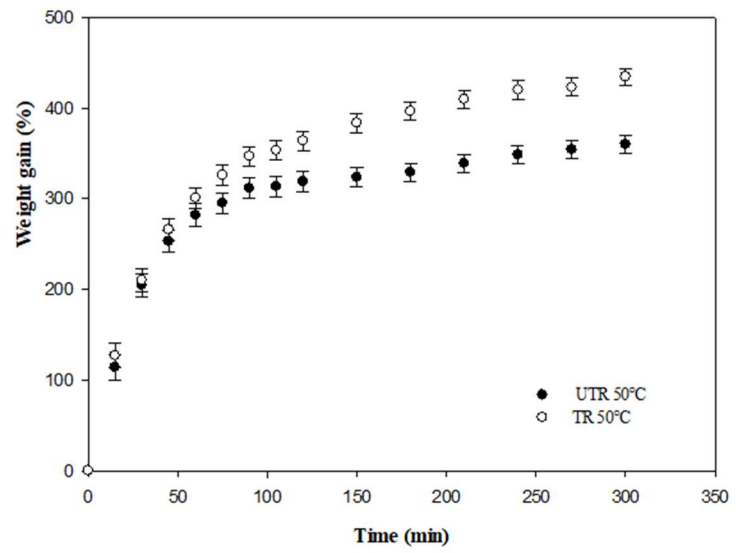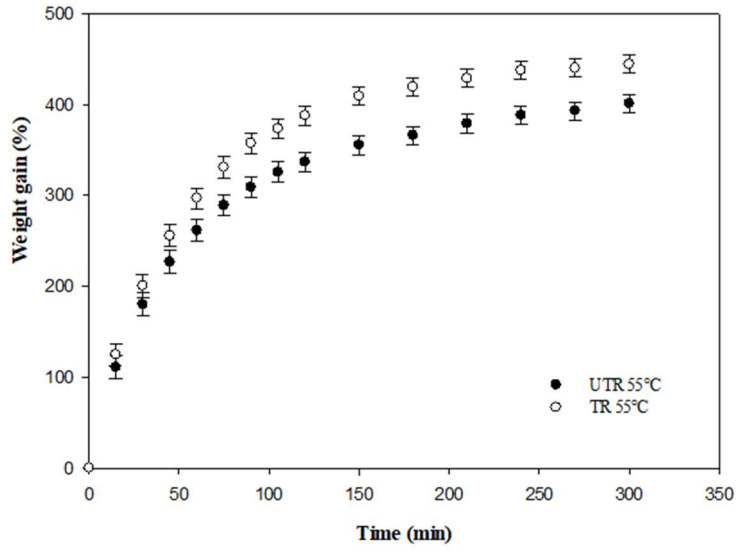
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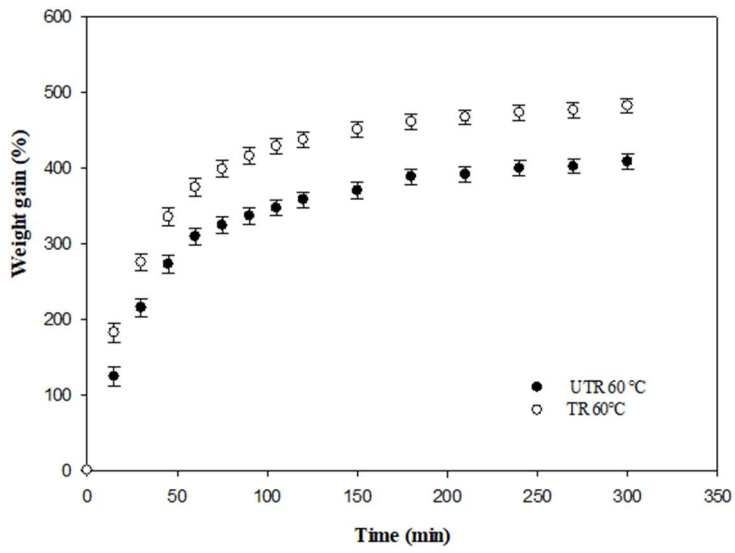
d)

Figure IV.26 *Rehydration curves of untreated (UTR) and treated (TR) peaches dried at 45°C (a), 50°C (b), 55°C (c), 60°C (d)*

IV.2.7 Preliminary sensory evaluation

Preliminary sensory evaluation of untreated (UTR) and treated (TR) peach slabs dried at 45, 50, 55 and 60°C was performed based on attributes of appearance, aroma, taste, texture and overall acceptability. The obtained results were shown in two forms as Table IV.12 and Figure IV.27 which reported the effect of pre-treatment and air-drying temperatures on the sensory quality of the dried peach slabs. Appearance is one of the most important quality attributes for both dried foodstuffs and consumer preference.Related to appearance attribute, there were not significant differences (p > 0.05) among the untreated peaches dried at 50, 55 and 60°C. Untreated peaches dried at 45°C had the lowest appearance scores while both treated samples dried at 55 and 60°C obtained the highest appearance scores. Appearance attributes of dried peaches probably may be accompanied by colour for consumers. These findings were mainly attributed to the great impact of the novel pre-treatment on the colour characteristics of dried peaches which made possible an enhancement in lightness and reduced undesirable browning reactions. Also, at lower drying temperature (45°C) and long drying time promoted more colour changes in untreated dried samples (as browner final dried peaches).

In addition, there were no statistical differences (p > 0.05) in aroma attributes between untreated samples dried at 45 and 50°C and treated ones dried at 50°C and the lowest scores of aroma were determined those dried samples. Also, there were no statistical differences in aroma scores between untreated samples dried at 55 and 60°C and treated samples dried at 45 and 55°C. The highest score of aroma was in treated samples dried at 60°C. Based on these aroma results, it was very difficult to make clear comments on aroma attributes of dried peaches. The combined effect of the higher drying temperature (60°C) and pre-treatment may be useful to preserve the aroma attributes of dried peach samples. Another possible explanation may be explained by exposure to high temperatures promotes the occurrence of non-enzymatic browning reactions, which results in volatile compound formation, such as fission products and Strecker aldehydes, among other compounds, which are responsible of flavour or off-flavour (Dueik et al., 2013).

Concerning both taste and texture, no significant differences (p > 0.05) were observed between untreated and treated dried peaches at each drying temperature (45, 50, 55 and 60°C).

The best results for overall acceptability were attributed by the untrained panel members to treated samples at higher drying temperatures (55°C and 60°C) which mainly characterized by higher scores of appearance (i.e. colour, shape etc.) which influence the overall acceptability of dried peaches.For these reasons, the combination of pre-treatment and higher

drying temperatures (55°C and 60°C) indicated the better overall acceptability in treated dried peaches.

Table IV.12 *Preliminary sensory evaluation for quality attributes of untreated (UTR) and treated (TR) dried peaches (45, 50, 55, 60°C). Values with different letters in a given column are significantly different (p<0.05).*

Sample	Appearance	Aroma	Taste	Texture	Overall Acceptability
UTR 45°C	2.75 ± 0.46^a	2.75 ± 1.16^a	3.25 ± 1.16^a	3.25 ± 0.89^a	2.50 ± 0.53^a
TR 45°C	3.50 ± 1.20^{ab}	3.50 ± 0.53^{ab}	4.00 ± 0.76^a	3.50 ± 0.53^a	3.50 ± 0.53^{ab}
UTR 50°C	3.25 ± 0.46^{ab}	2.75 ± 0.46^a	3.00 ± 0.76^a	3.00 ± 0.76^a	2.75 ± 0.46^a
TR 50°C	4.00 ± 0.00^{ab}	2.75 ± 0.46^a	3.00 ± 0.76^a	3.25 ± 0.46^a	3.50 ± 0.53^{ab}
UTR 55°C	4.00 ± 0.00^{ab}	3.25 ± 0.89^{ab}	3.25 ± 0.89^a	3.75 ± 0.46^a	3.50 ± 0.53^{ab}
TR 55°C	4.50 ± 0.53^b	3.50 ± 0.53^{ab}	4.25 ± 0.46^a	4.50 ± 0.53^a	4.50 ± 0.53^b
UTR 60°C	3.75 ± 0.46^{ab}	3.50 ± 0.53^{ab}	3.75 ± 0.46^a	4.25 ± 0.46^a	3.75 ± 0.46^{ab}
TR 60°C	4.50 ± 0.53^b	4.50 ± 0.53^b	4.75 ± 0.46^a	4.50 ± 0.53^a	4.75 ± 0.46^b
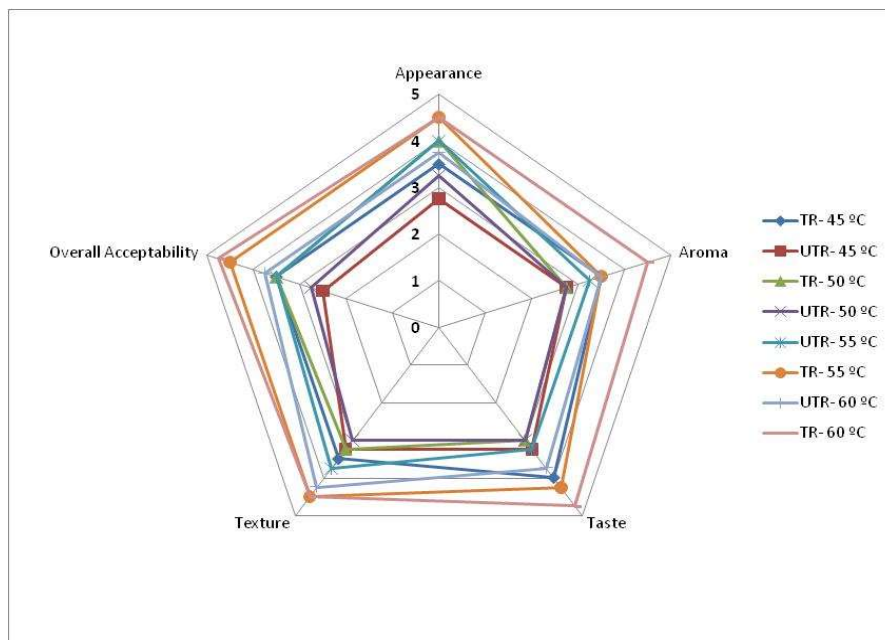
Figure IV.27 *Preliminary sensory evaluation for quality attributes of untreated(UTR) and treated(TR) dried peaches (45, 50, 55, 60°C)*

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IV.2.8 Volatile organic compounds (VOCs)

IV.2.8.1 Volatile compounds in fresh peach

The major VOCs of fresh peach were: benzaldehyde (553 μg/kg d.b.); 4-decalactone (293 μg/kg d.b.); (E)-2-hexenal (131 μg/kg d.b.), cis-3-hexenylacetate (129 μg/kg d.b.); isopentanoic acid (87 μg/kg d.b.) and 2-phenylethylalcohol (75 μg/kg d.b.). Anyway, the contribution of the substances to the odor note of the fruit depends not only in the quantities of the compounds, but also mainly on their perceptionthreshold. VOCs with odor activity value (OAV) > 1, considered to contribute to the aroma of the peach, were: 4-decalactone (OAV=2.92); benzaldehyde (OAV=1.73); (E)-2-hexenal (OAV=1.60) and 2-phenylethylalcohol (OAV=1.21) (Van Gemert, 2003, Zhu et al., 2015). Decalactones are the most abundant lactones in the flesh and contribute to the characteristic fruity smells of peach. (Zhu and Xiao., 2019). Benzaldehyde, characterized by almond, fruity odor notes, was previously observed to correlate positively to firmness (Sánchez et al., 2012), while the concentration of 2-hexenal, responsible of green and fatty aromas was reported to differ within a cultivar (Bianchi et al., 2017).

IV.2.8.2 Volatile compounds in dried peach

After drying, the reduction of benzaldehyde varied from 86% in UTR50°C sample, to 76% in TR60°C. The loss of decalactons was much more accentuated: up to 99% in UTR60°C and always more than 93% in all dried peaches, as the reduction of (E)-2-hexenal, varying from 98% in TR45°C to 85 in UTR55°C samples. Even the decreases in 2-phenylethylalcohol were substantial, up to 99% in TR45°C and always over 90% in the other samples. Therefore, all the compounds with the odorous impact of the peaches, and not just them, were significantly reduced after drying, modifying fruits odorous profiles. On the other hand, several new compounds were formed, because of various chemical transformations, including the Maillard reaction.The Maillard reaction initially gives Amadori products, which further degrade into sugar fragmentation compounds forming Strecker aldehydes, alcohols and ketones (Rizzi 2008), whose concentration play a key role in the overall flavor of dried fruits. Among the volatile compounds in dried peaches, typical of the Maillard reaction, were: furfuraldehyde, with concentrations increasing with rising temperatures (from 10 μg/kg d.b. in TR45°C to 143 in UTR60°C samples) and 5-hydrxoymethylfurfural, with values always below 10 μg/kg d.b. The concentration of nonanal had in all dried samples OAV > 1, with maximum values in TR50°C (OAV=2.43) and minimum values in TR60°C (OAV=1.59). Nonanal is described as having partially green aromas, with citrus characteristics. The concentration of decanal exceeded the peak threshold only in samples TR55°C (OAV=1.41) and UTR45°C (OAV=1.08).

IV.2.8.3 Effect of pre-treatmet on VOCs

The concentration of acids (octanoic, nonanoic, decanoic, hexanoic and acetic acids), alcohols (2-ethylhexanol, 2-octanol and cis-3-hexen-1-ol) esters (4-decalactone, ethyldodecanoate, ethyldecanoate and butyryl lactone) and terpens compounds (D-limonene, linalool, α-terpineol, hotrienol) was greater in TR samples, especially in the sample dried at 45°C. The same behaviour was observed for thealdehydes compounds (nonanal, 2,4-heptadienal, decanal, 5-hydrxoymethylfurfural), especially in the sample dried at 55°C. While the concentration of ketones compounds (acetoin, 6-methyl-5-heptene-2-one,β- ionone) was less in TR samples.

IV.2.8.4 PCA analysis

The first two components of PCA explained 68% of data variability (49.94% and 17.99%, respectively). Alcohol compounds, as 3-octanol, cis-3hexenol, 1-pentanol, 1-octanol, 1-hexanol, are located in the positive quadrants of PC1 while Ketones compounds, as hydroxyacetone, 3-5-octadien2one, cis-geranylacetone and aldehyde compounds, as 5-methylfurfural, nonanal, decanal, are located on the negative side one. The fresh peach showed a clear opposition respect to dried samples compared to the first component. Fresh peach and TR samples showed a good correlation on PC2 axis, whereas the UTR samples were negatively correlated on the second factor of PCA.

IV.2.8.5 Conclusion

The dried samples compared to fresh peaches showed an increase in aldehydes, ketones and especially terpenes and acids. The highest concentration of these compounds was higher in TR samples.
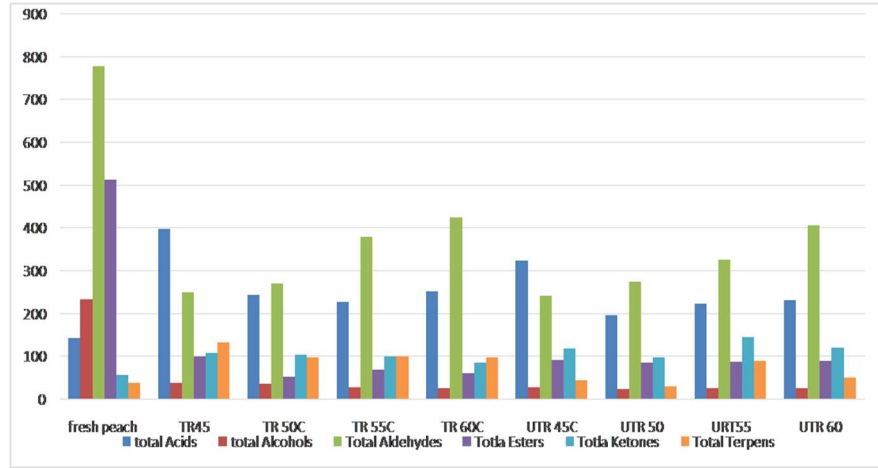
Figure IV.28 *Classification of volatile aroma compounds (VOCs) of fresh and untreated (UTR)-treated (TR) peaches dried at 45, 50, 55 and 60°C*
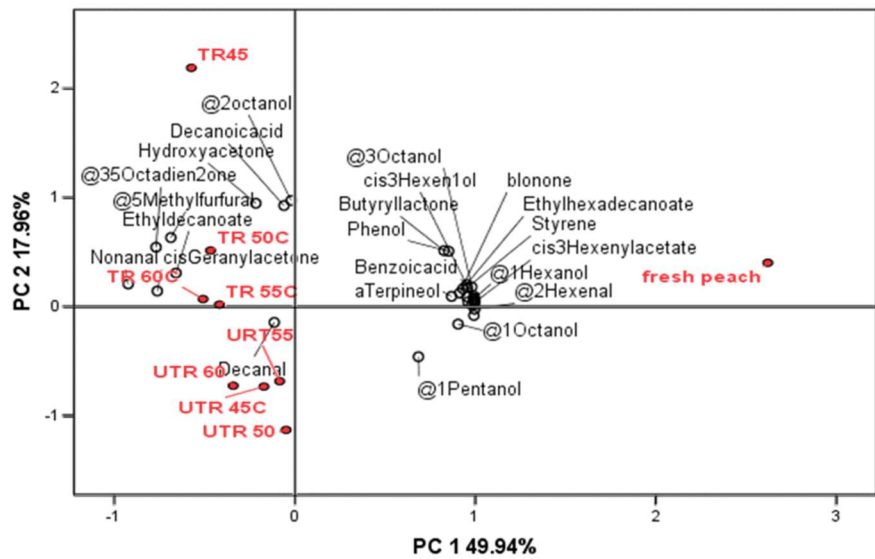
Figure IV.29 *Two-dimensional principal component analysis of volatile aroma compounds (VOCs) for fresh, untreated (UTR) and treated (TR) samples dried at 45, 50, 55 and 60°C*

IV.2.9 Conclusions

The effect of novel dipping pre-treatment and air-drying temperatures (45, 50, 55, and 60°C) on drying characteristics and quality parameters of 'Terzarola giallo'peach slabs were investigated using a convective dryer.The drying behaviour of peach slabs was affected by both the pre-treatment and drying temperatures. Lower drying time was found in pre-treated samples with a novel solution at higher drying temperature (60°C). In addition, this pre-treatment solution improved the quality parameters of dried peaches, including colour, shrinkage, antioxidant activity, rehydration capacity, preliminary sensorial evaluation and volatile aroma compounds (VOCs). The combined effect of pre-treatment and higher drying temperature (60°C) determined the best quality peach slabs with the except of antioxidant activity. Besides, the antioxidant activity of samples decreased by increasing drying temperatures from 45°C to 60°C: the highest values were observed in treated dried peaches at 45°C.The preliminary sensory evaluation showed that this pre-treated samples dried at 55 °C and 60°C had good acceptance for untrained panelists. The peach samples pre-treated with a novel solution presented higher rehydration capacity at 60°C. The findings of this study may be important to provide useful information for the development of novel and natural pre-treatment conditions before drying of peach or other fruits, being an alternative to other chemical pre-treatments and suitable to an industrial context.

IV. 3 Results of'Rocha' pear

IV.3.1 Drying characteristics and effectof pre-treatments on drying process

Figure IV.30(a-c) presented the drying curves of control, microwave and ultrasound pre-treated pears at investigated temperatures of 50, 55 and 60°C. The drying curves of pears samples depended on the applied pre-treatments (i.e. microwave and ultrasound) and drying conditions (i.e. temperature and time).The average moisture content of fresh samples was kg 6.42 ± 0.66 water/kg (db), and the average water activity was 0.98 ± 0.004.Pear slabs were dried up to final moisture of 0.07 ± 0.01kg water/kg (db) and to the water activity of 0.41 ± 0.04. No constant rate period was detected in all samples within the studied drying experimental conditions.In our case, only the falling rate period was observed in all samples at each drying temperature, indicating that the drying process in the tray dryer was controlled by internal mass transfer (moisture migration within pear samples).

The all drying curves of pears showed the similar behaviour: moisture content decreased in all samples as the drying time progressed. Moreover, the drying process was accelerated when the temperature increasing from 50 to 60°C. The drying behaviour with increasing drying temperature was explained by Nascimento et al. (2016) and Tao et al. (2018): The temperature rising can reduce the relative humidity of air, increase the moisture gradient between food materials and air and promote the moisture movement within foods, thus resulting in the enhancement of drying curves.

As it can be seen, microwave pre-treatment (539 W for 4 min) before drying enhanced drying process, because the moisture content in microwave pre-treated (MW) pears decreased faster than in control and ultrasound pre-treated (US) samples. In this way, the microwave pre-treated samples had shorter drying time in comparison with control and ultrasound pre-treated ones at all investigated temperatures. In the meanwhile, the drying times necessary to attain equilibrium moisture content were observed equal to 582, 450 and 393 min for microwave pre-treated pears at drying temperatures of 50, 55 and 60°C. Time values were for control samples, respectively, 900, 696 and 525 min; while for ultrasound pre-treated samples were, respectively, 750, 696 and 577 min at the same respective drying temperatures.This behaviour may be explained by the internal (volumetric) heating caused the moisture migration to the pear surface during microwave pre-treatment. Microwave applications include two mechanisms as ionic polarization and dipole rotation. In ionic polarization, an electric field is applied to make ions move and collide with each other. As a consequence, their kinetic energy is converted into heat inside the food materials. Dipole

rotation occurs when polar molecules (i.e. water)are present in food. The molecules change their direction which affected by an electrical field, interact with their surround molecules and as a result, produce heat. Therefore the higher number of polar molecules results in the higher heat production.(Deghannya et al., 2019).

Less drying time in the microwave and its combined applications may be associated with the rapid mass transfer within food materials during microwave heating. Heat is generated within the food due to the absorption of microwave energy, and it creates high internal pressure, temperature and concentration gradients. Therefore, the flow rate of the liquid through the food to the boundary is increased. Similar results were obtained from different studies in which time of microwave drying was found to be significantly shorter than hot air drying time of fruits and vegetables (Aydogdu et al., 2015; Sharma and Prasad, 2001; Sumnu et al., 2005).

As expected, the drying temperature affected the drying curves of ultrasound pre-treated pears which became faster with increasing drying temperature from 50 to 60°C. The use of ultrasound as pre-treatment prior to drying process of fruits and vegetables to accelerate the moisture removal process, thereby resulted in shortening drying time (Nowacka et al., 2016). However, in our case, there was not drying time reduction in ultrasound treated pears dried at 55 and 60°C in comparison with control samples dried at the same respective temperatures.Under these drying conditions, the drying time of control and ultrasound pre-treated samples were quite similar. Probably, this could be caused by the fact that ultrasound pre-treatment were applied through the vacuum packaging and not only through the pear samples immersed in the water medium or by structural changes of pear tissue that occurred during ultrasound pre-treatment. The structural properties of the food material are decisive for the action of the ultrasound waves (Corrêa et al., 2017); from this point of view, the changes in pear composition and structure may be modified by ultrasound application and can affect the ability of pear to be dried.Another possible explanation may be related to insufficient sonication time and thus ultrasound application with a sonication time of 10 min did not influence on the rapid moisture removal and enhancement of the drying process. More intense ultrasonic field or longer sonication time could be necessary to cause significant effects on the drying process. The similar behaviour was observed in carrot slices by (Nowacka and Wedzik, 2016), where it was demonstrated that ultrasound pre-treatment did not significantly impact on the drying process and shorter drying time. However, carrot slices were vacuum packed and then treated with ultrasound application before the drying. These authors stated that the vacuum packed carrots were used to avoid or reduce adverse effects of rinsing out substances contained in the raw material.

Moreover, the ability to intensify the water transfer due to ultrasonic waves with different frequencies based on the food material (Nowacka and

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Wedzik, 2016). By taking into consideration the combined pre-treatment of ultrasound application with 10 min and the drying temperature of 50°C, the drying time was 750 min and 900 min for ultrasound pre-treated and control samples, respectively. At a drying temperature of 50°C, ultrasound pre-treated samples had shorter drying time than control ones.This behaviour may be explained by the lower drying temperature and longer exposure time produced the higher damage in control samples.

In our case, considering the effect of ultrasound application on the drying process of 'Rocha' pears, these obtained findings could be associated with the isolation of the pear samples by vacuum-packaging from the liquid medium (water) during the ultrasound application. This type of pre-treatment procedure, probably caused the limited effect of cavitation and only sponge effect occurred. Therefore, it can be notified that the occurrence of this sponge effect was insufficient to accelerate the drying process and moisture movement.

However, in the cases, ultrasound pre-treatment was applied prior to drying, samples were directly immersed in a liquid medium and which hada significant effect on the both drying time (a reduction of drying time) and drying kinetics for melon (Dias da Silva et al., 2016),pineapple (Fernandes et al., 2008), banana (Nowacka et al., 2012), apple (Fijalkowska et al., 2015), mulberry (Tao et al., 2016), garlic (Tao et al., 2018). These authors have stated that, the influence of ultrasonic pre-treatment on the enhancement of drying kinetics and less drying time can be attributed to the mechanical effect of ultrasound (alternating compressions and expansions) by the creation of micro-channels, which can facilitate the detachment of water molecules from fruit or vegetables' cellular structure. This phenomena is called 'sponge effect', thus this can contribute to decrease in both internal and external mass transport resistance (García-Pérez et al., 2012, Tao and Sun, 2015; Tao et al., 2018).At the same time, the cavitation phenomena of ultrasound and the accompanied physical and chemical changes, involving microstreaming, turbulence, locally extreme high pressure and temperature may destroy the tissue structure (as intracellular and extracellular), thus reducing the diffusion boundary layer and promoting the moisture transfer (Tao and Sun, 2015; Tao et al., 2018).

Consequently, above mentioned studies, the obtained results were positive, indicating that the ultrasound application can positively influence on the drying kinetics of foodstuffs. In this work, ultrasound pre-treatment did not highlight the positive effects on the drying kinetics in comparison to similar works in the literature.

a)

b)
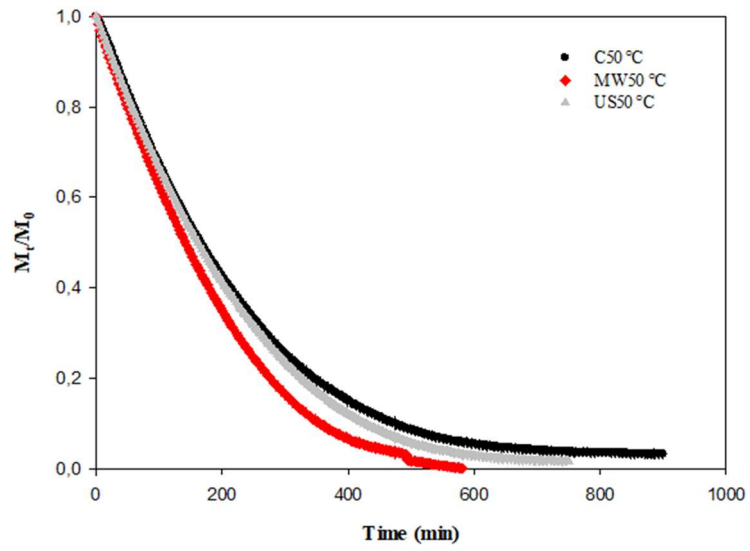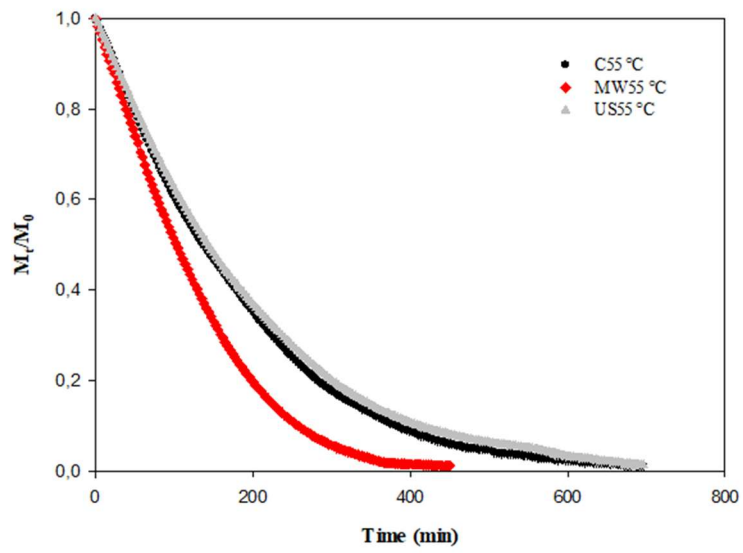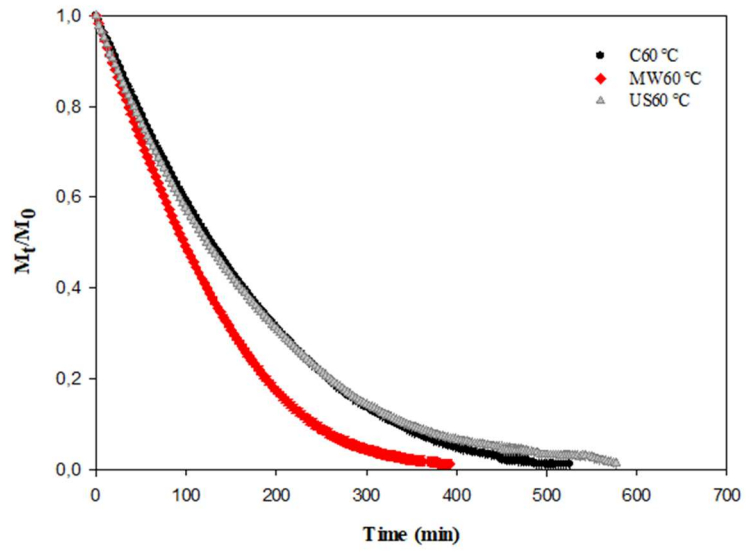
c)

Figure IV.30 *Experimental drying curves of control (C), microwave (MW) and ultrasound (US) pre-treated pears 50°C (a), 55°C (b), 60°C (c)*

IV.3.2 Mathematical modelling: Empirical models

In order to estimate the moisture content (X) as a function of drying time (min), the empirical models were demonstrated in Table III.1, were fitted and the statistical parameters of the models were summarized in Table IV.13. The values of parameters for each model were reported in Table IV.14.

The average equilibrium water content of control, microwave and ultrasound treated pear samples was found as 0.148 ± 0.004, 0.156 ± 0.003, 0.148 ± 0.005 kg water kg dry matter^{-1}, respectively using the GAB equation (eq. 19) and these values were used for all drying modelling.

The coefficient of determination (R^2) and the standard deviation of the experimental error (s) were one of the primary criteria to select the best model to account for variation in the drying curves of dried pear samples. A good fitting between the experimental data and the correlations were a combination of the highest R^2 value and the lowest value of s (Ramos et al., 2014).

Table IV.13 *Model parameters of control, microwave and ultrasound pre-treated 'Rocha' pears dried at 50, 55 and 60°C*

Model Name	Temperature	Parameters	Samples		
			Control	Microwave	Ultrasound
Henderson	50°C	a	1.061	1.074	1.068
and Pabis		k	0.481	0.598	0.508
	55°C	a	1.068	1.098	1.058
		k	0.615	0.881	0.577
	60°C	a	1.074	1.090	1.041
		k	0.646	0.925	0.632
Page	50°C	k	0.169	0.154	0.165
		N	1.184	1.240	1.191
	55°C	k	0.213	0.170	0.282
		N	1.186	1.310	1.124
	60°C	k	0.184	0.177	0.312
		N	1.224	1.319	1.126
Modified	50°C	k	0.228	0.538	0.462
Page		N	1.184	1.240	1.191
	55°C	k	0.558	0.769	0.531
		N	1.186	1.310	1.157
	60°C	k	0.584	0.820	0.594
		N	1.220	1.310	1.124

According to models'parameters in Table IV.13, for the Henderson &Pabis, Page and Modified Page models, the drying constant k had a value of 0.154 to 0.925 and these values generally increased with an increase 'n drying temperatures in control, microwave and ultrasound treated samples.

Table IV.14 *Correlation coefficients (R^2 and s) of drying models*

Model Name	Temperature	Correlation Coefficients	Samples		
			Control	Microwave	Ultrasound
Henderson & Pabis	50°C	R^2	0.997	0.989	0.995
		s	0.0835	0.1432	0.1263
	55°C	R^2	0.994	0.986	0.996
		s	0.1202	0.1530	0.1001
	60°C	R^2	0.991	0.983	0.996
		s	0.1612	0.1733	0.1071
	50°C	R^2	1.000	0.997	0.999
Page		s	0.0255	0.0595	0.0481
	55°C	R^2	0.999	0.998	0.998
		s	0.0541	0.0534	0.0776
	60°C	R^2	0.999	0.997	0.999
		s	0.0650	0.0737	0.0499
Modified Page	50°C	R^2	1.000	0.998	0.999
		s	0.0255	0.0597	0.0482
	55°C	R^2	0.999	0.998	0.999
		s	0.0542	0.0536	0.0380
	60°C	R^2	0.999	0.997	0.999
		s	0.0644	0.0741	0.0508

The statistical parameter estimations showed that $R^2$and s values were ranged from 0.983 to 1.000 and from 0.0255 to 0.1733, respectively for all evaluated samples. Between the all tested empirical models, the Page model gave the best results and demonstrated good agreement with experimental data obtained fromcontrol samples dried at 50°C, ultrasound treated samples dried at 50°C, control samples dried at 55°C, microwave treated samples dried at 55°C, both microwave and ultrasound treated samples dried at 60°C. Furthermore, Modified Page model was found to be the best appropriate model to describe 'Rocha' pear drying curves for microwave treated samples dried 50°C, ultrasound treated samples driedat 55°C and control samples dried at 60°C with the highest R^2 values and the lowest s values.

The residuals, referring to experiments carried out at 50, 55 and 60°C, were reported in Figure IV.31(a-c) for control, microwave and ultrasound samples dried at 50°C, in Figure IV.32(a-c) for control, microwave and

ultrasound samples dried at 55°C, and in Figure IV.33(a-c) for control, microwave and ultrasound samples dried at 60°C. Predict values were reported on the graphs Figure IV.31(a-c),Figure IV.32(a-c) and Figure IV.33(a-c) which refer to the moisture content (X) of control, microwave and ultrasound treated samples on dry basis during the drying process.

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a)
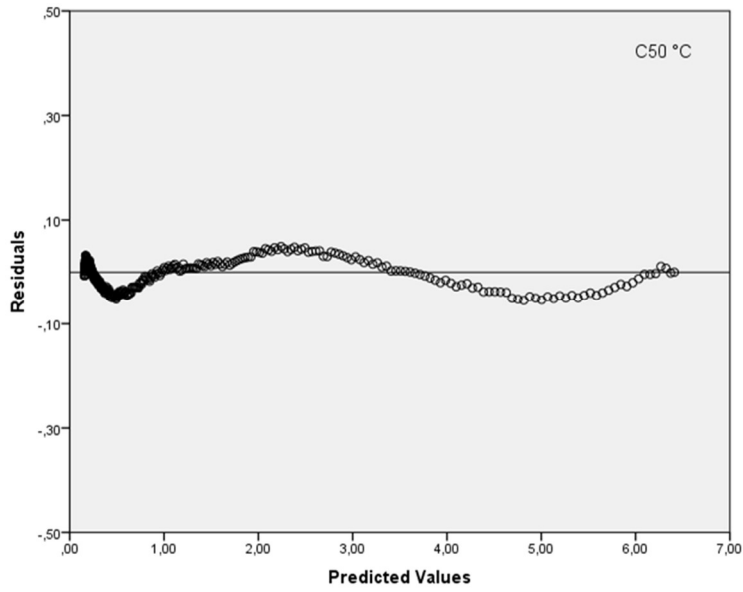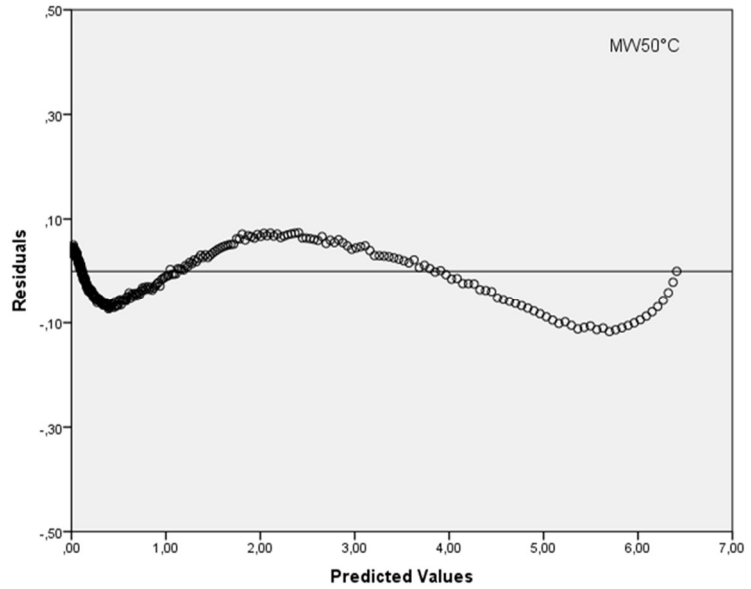
b)
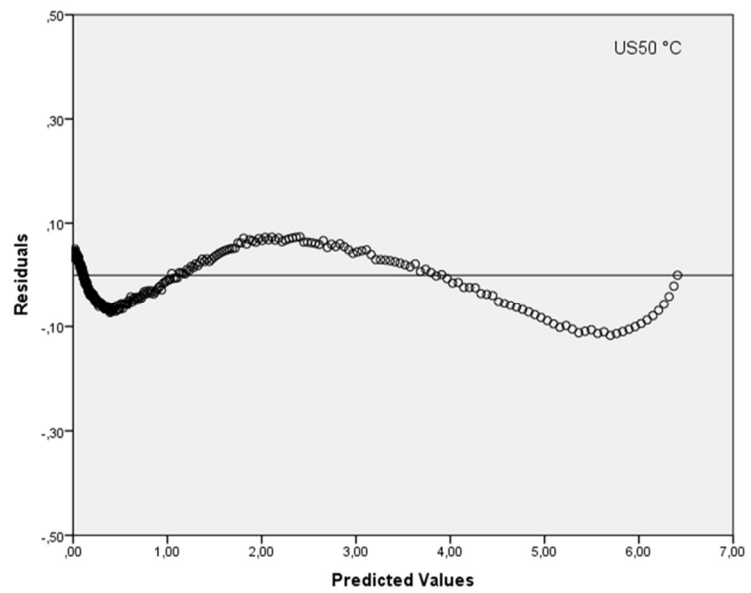
c)

Figure IV.31 *Residual analysis for control (C)(a), microwave (MW)(b) and ultrasound (US)(c) pre-treated pears dried at 50°C*

a)

b)
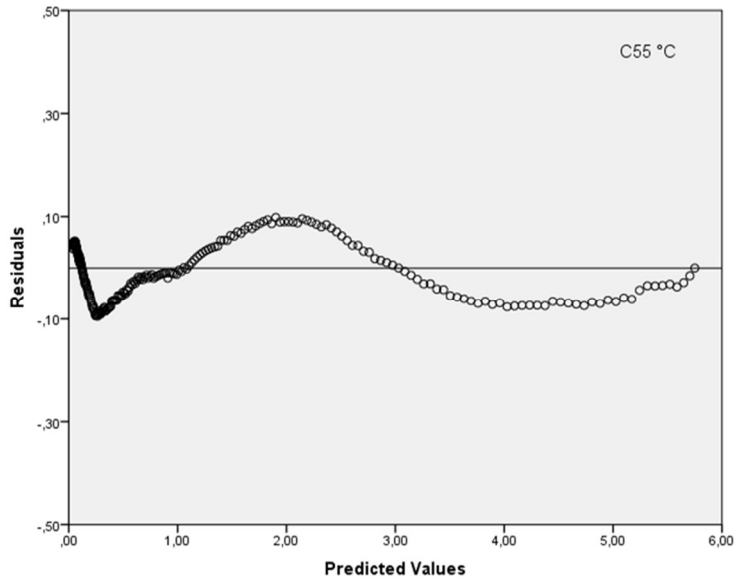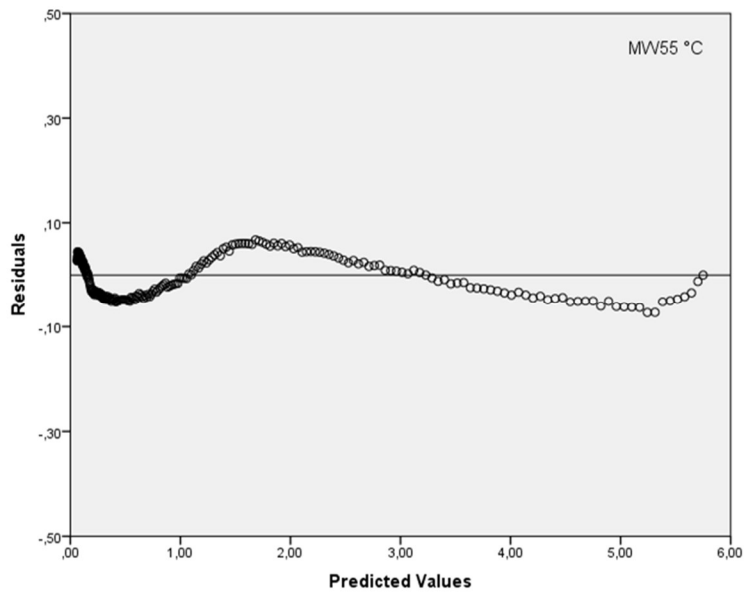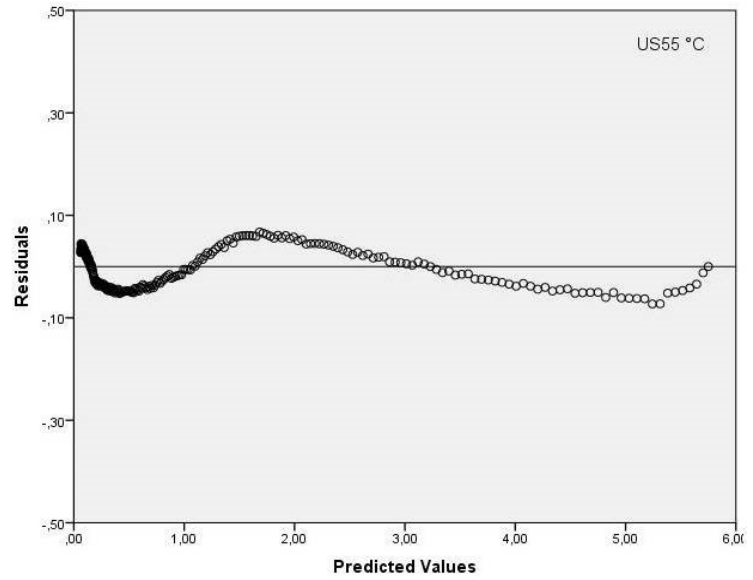
c)

Figure IV.32 *Residual analysis for control (C)(a), microwave (MW)(b) and ultrasound (US)(c) pre-treated pears dried at 55°C*
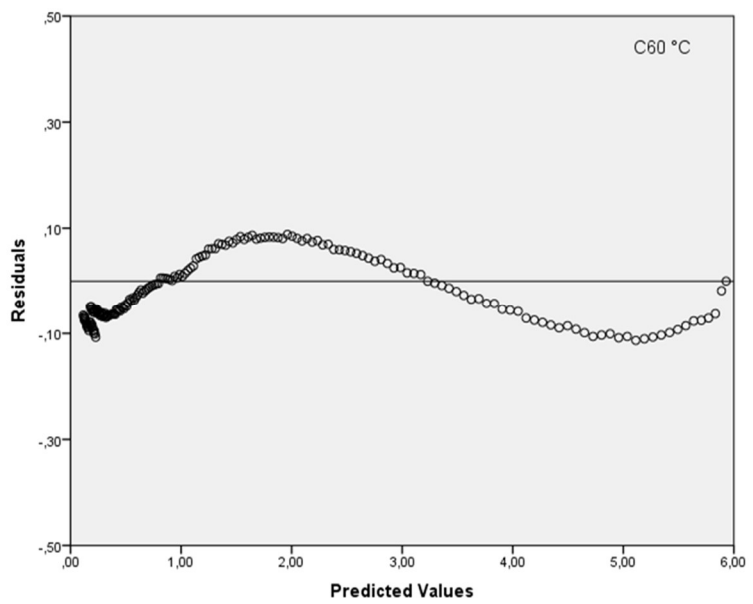
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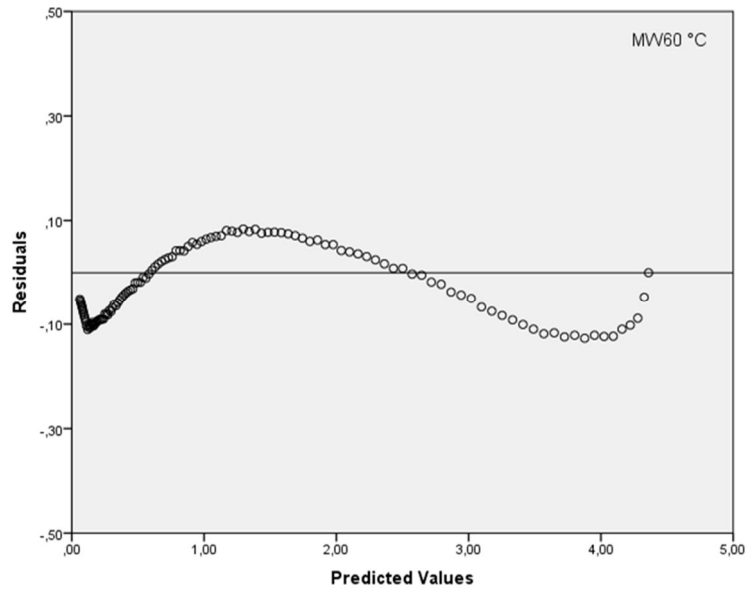
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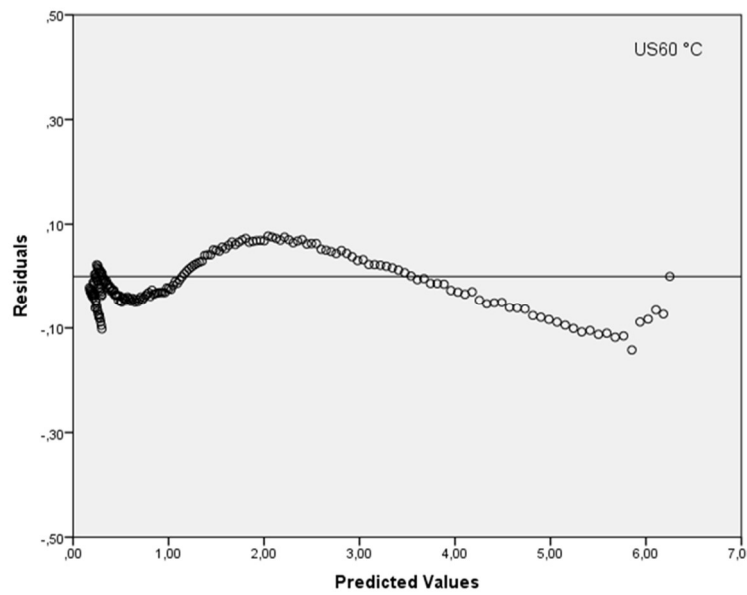
c)

Figure IV.33 *Residual analysis for control (C)(a), microwave (MW)(b) and ultrasound (US)(c) pre-treated pears driedat 60°C*

Chapter IV

The experimental data and the best fitting model results (Page and Modified Page models)were demonstrated in Figure IV.34(a-c),Figure IV.35(a-c),Figure IV.36(a-c) for pear samples dried at 50, 55 and 60°C, respectively. The Page models and Modified Page models were able to predict with sufficient accuracy the evolution of the moisture content of 'Rocha' pear slabs.
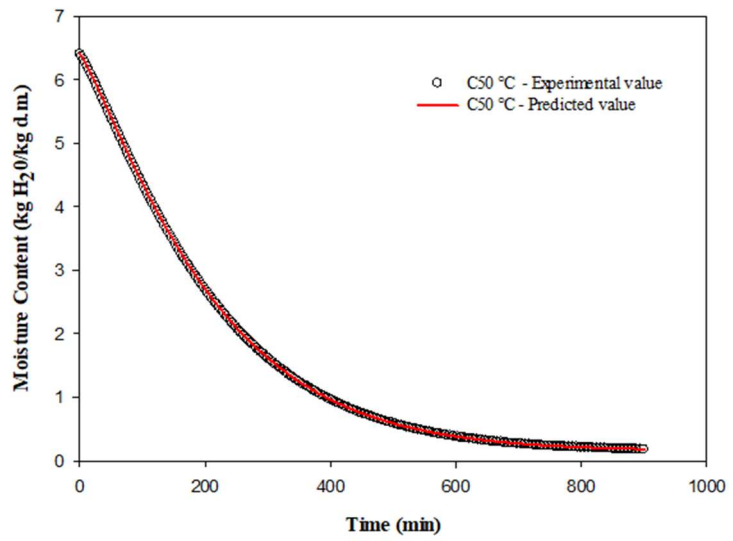
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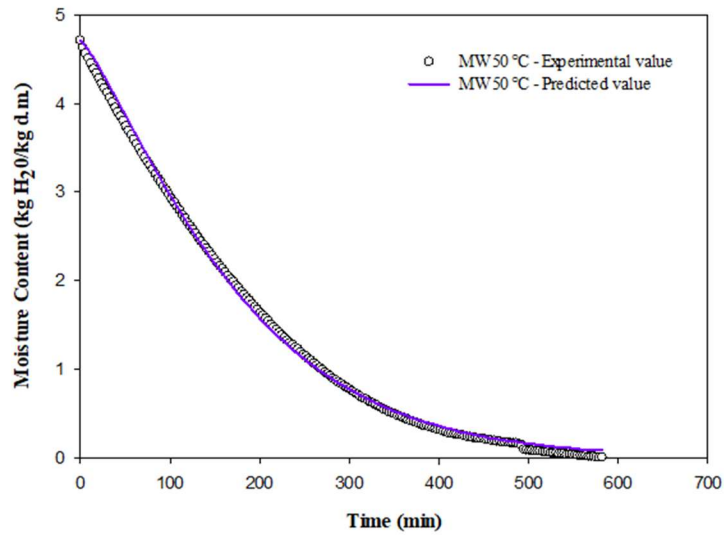
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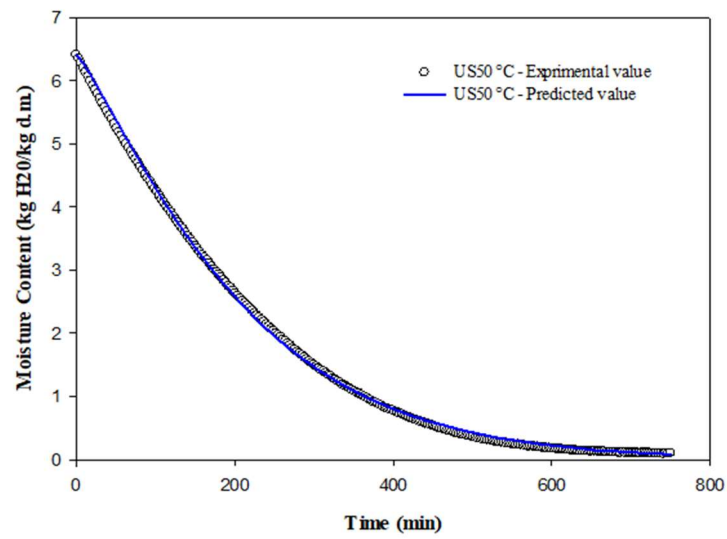
c)

Figure IV.34 *Experimental (symbols) and predicted (lines) drying curves of control (C)(a), microwave (MW)(b) and ultrasound (US)(c)pre-treated pears dried 50°C*
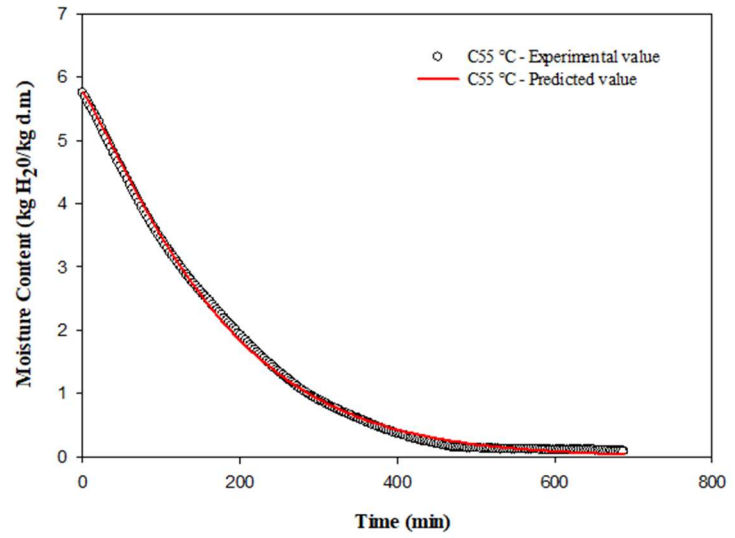
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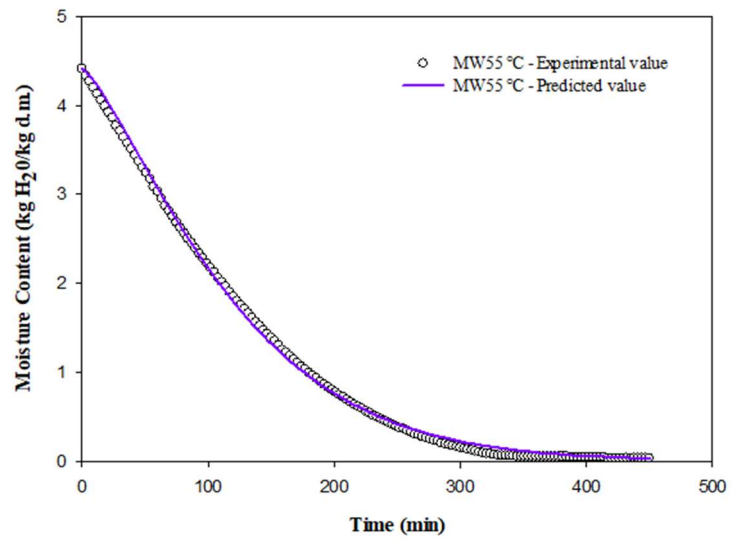
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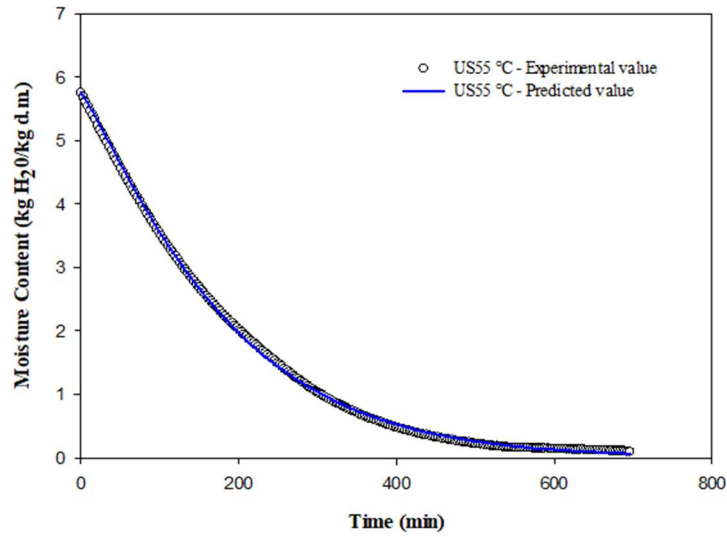
c)

Figure IV.35 *Experimental (symbols) and predicted (lines) drying curves of control (C)(a), microwave (MW)(b) and ultrasound (US)(c) pre-treated p ears dried at 55°C*

a)

133
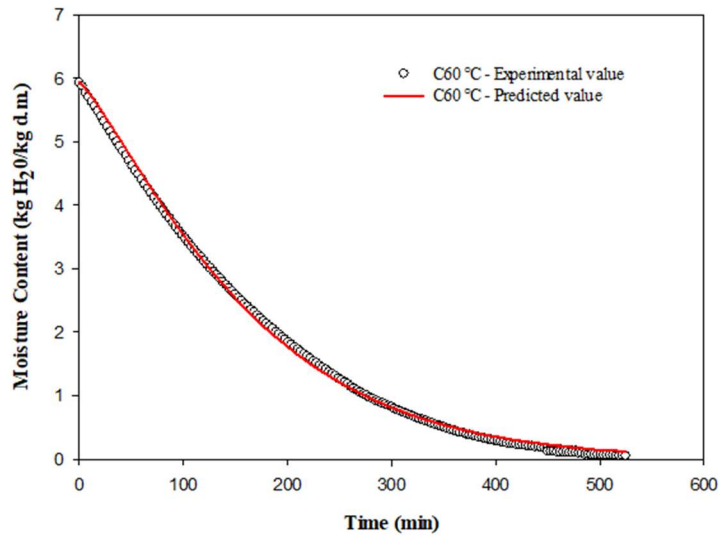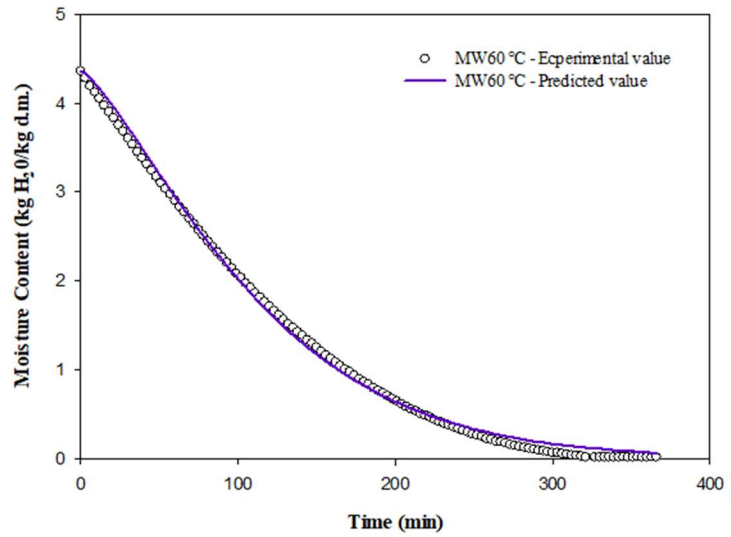
b)
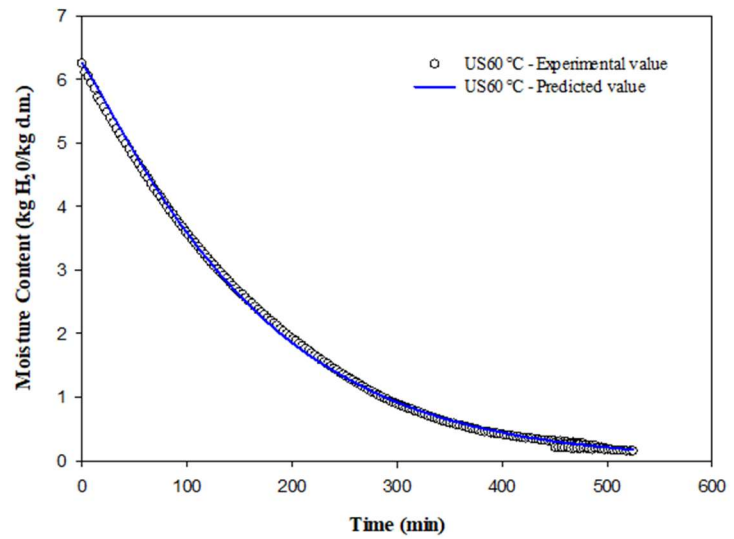
c)

Figure IV.36 *Experimental (symbols) and predicted (lines) drying curves of control (C)(a), microwave (MW)(b) and ultrasound (US)(c) pre-treated pears dried at 60°C*

IV.3.3 Colour evaluation

Colour parameters of fresh pears and control, microwave and ultrasound pre-treated pears dried at 50, 55 and 60°C were presented in Table IV.15. According to the results, colour parameters were affected by both applied pre-treatments and drying temperatures. After drying process, the L* value decreased in all microwave treated samples at each drying temperature and the lowest L* value was in microwave treated dried samples at 50°C. Thus, the colour of all microwave treated pears got browner after hot air drying process in comparison to fresh, control and ultrasound treated dried ones. Similar observations were reported by Krokida et al. (2000), microwave had a negative effect on the lightness parameter (L*) for apple, potato, banana and carrot samples conventionally dried at 70°C. There was a similarity of L* values between the control and ultrasound pre-treated samples dried at 50, 55 and 60°C (except for ultrasound treated pears dried 60°C). The highest L* value was observed in ultrasound pre-treated pears dried at 60°C as 79.05 ± 0.25, while the L* values of fresh samples were 78.60 ± 0.25.The highest L* values of ultrasound pre-treated pears dried at 60°C indicated that the ultrasound application with higher drying temperature of 60°C was sufficient to preserve the lightness as those of the fresh fruit. Thus, the original colour of pear can be better protected when the samples received the combination of ultrasound pre-treatment and higher drying temperatures of 60°C.

In concern with the white index, drying process resulted in a reduction of white index for all dried samples. In particular, a significant decrease was observed in all microwave treated dried pears. However, ultrasound pre-treated dried pears at 60°C demonstrated that similar white index values to those of fresh ones (p > 0.05).

The total colour difference is a crucial parameter for the dried fruit and vegetables, which indicates the human eye's ability to differentiate between colours of of products (Horuz and Maskan, 2017). The drying temperature influenced also the values of total colour changes (ΔE), revealing a decreasing tendency with the increase of temperature from 50 to 60°C. These results showed that the occurrence of colour deterioration during the drying process, being more pronounced when the lowest drying temperature was employed. This tendency may explained by the browning reactions or the formation of browning products occurring at lower temperature and long-time exposure to drying process at low temperature (50°C).The highest value of ΔE was in all microwave pre-treated dried pears with respect to control and ultrasound pre-treated dried ones. These results showed that all microwave treated dried samples were darker than the others. This trend may be attributed to non-uniform temperature distribution during microwave treatment, and few regions of pear samples could get heated very rapidly, while the remaining region could get heated to a lesser extent. Controlling

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heating uniformity is an important parameter to obtain high quality of microwave-dried fruits. During the microwave applications, constant microwave power can cause an increase of the average product temperature and overheating of the food material can be prevented by controlling the microwave power (Horuz et al., 2017). The large number of factors may influence the temperature distribution during the microwave applications such as the thickness, geometry, dielectric properties of foods and microwave energy (Vadivambad and Jayas, 2007). The highest colour alterations of microwave treated dried samples may be dependent on the thickness of pear, microwave time or loss of homogeneity during the microwave treatment.

The lowest value of ΔE was observed in ultrasound pears dried at 60°C, indicating that the combined pre-treatment of ultrasound application and higher drying temperature helped better preserve the original colour of pear in dried snacks.

Table IV.15 *Colour parameters for fresh, control (C), microwave (MW) and ultrasound (US) pre-treated 'Rocha' pears dried at 50, 55 and 60°C. Different superscript letters (a,b,c etc.) in the same column mean significant differences (p < 0.05)*

Sample	L*	WI	ΔE
Fresh	78.60 ± 0.90^e	72.11 ± 1.00^f	-
C50°C	71.35 ± 1.98^c	61.21 ± 1.21^c	11.57 ± 0.71^d
MW50°C	55.77 ± 2.99^a	48.35 ± 1.17^a	23.41 ± 2.18^f
US50°C	73.77 ± 0.89^{cd}	63.23 ± 0.88^{cd}	9.80 ± 0.71^{cd}
C55°C	72.46 ± 1.66^c	63.03 ± 0.60^{cd}	10.23 ± 0.78^{cd}
MW55°C	58.35 ± 0.84^{ab}	51.38 ± 1.15^b	21.99 ± 0.92^f
US55°C	77.15 ± 0.42^{de}	68.00 ± 0.48^e	6.11 ± 0.32^{ab}
C60°C	75.06 ± 0.80^{cde}	64.96 ± 0.67^d	8.13 ± 0.71^{bc}
MW60°C	60.53 ± 0.68^b	52.88 ± 0.95^b	14.91 ± 0.81^e
US60°C	79.05 ± 0.25^e	70.96 ± 0.21^f	3.86 ± 0.23^a

(a)	(b)	(c)	(d)
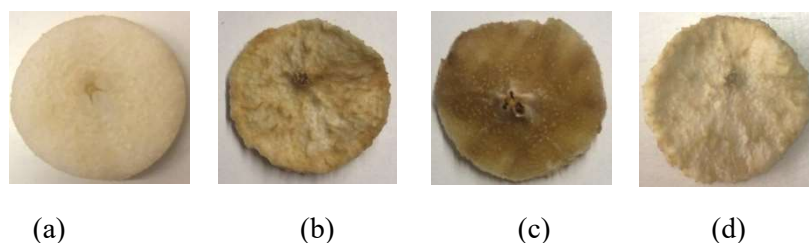
Figure IV.37*Rocha pear slabs: fresh (a), control (b), microwave treated (c) and ultrasound treated (d) dried at 60°C*

IV.3.4 Total phenolic content (TPC)

The total phenolic content of fresh pears, control, microwave and ultrasound pre-treated pears dried at 50, 55 and 60°C was summarized in Figure IV.38. The present experiments demonstrated that drying temperature and microwave and ultrasound pre-treatments had a remarkable effect on the total phenolic content of pear samples. The total phenolic content of fresh pear slabs was found as 336.82 mg GAE/100 g db. Total phenolic content levels were generally decreased after the drying process. A significant reduction of total phenolic content was observed in all microwave treated pears after drying and there were no statistical differences ($p > 0.05$) between all microwave treated dried samples; these results indicated that microwave pre-treatment had a negative impact on the total phenolic content and damaged the nutritional composition of all dried pears. Moreover, total phenolic content of microwave treated dried pears was slightly lower than control and ultrasound treated dried pears ($p < 0.05$).Related to the effect of microwave pre-treatment on the phenolic compounds of pear which are agreement with the findings of other researchers (Ghanem et al., 2012; Hayat et al., 2010), who reported that microwave heat treatment can liberate the bound phenolic compounds and increase the amount of free phenolic compounds in the foodstuffs. The reduction in the bound fractions with microwave power and time demonstrated that bound phenolic compounds (ester- and glycoside- bound) could be cleaved by microwave application and this behaviour may be due to the heating effect caused by the electromagnetic radiations during the microwave treatment. In addition, the greatest changes in total phenolic composition of microwave treated dried pears might be attributed to the structural changes in fruits due to the combined drying method (MW application and hot air drying). During the microwave treatments, observed shrinkage phenomenon can cause an internal stresses (non-uniform temperature) and surface tension which

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resulted in surface microcrakcs and leakage of exudate from the pears. Probably, the leakage of pigmented exudates decreased the total phenolic content during this combined drying (Zielinska and Michalska, 2016).

Ultrasound treated dried pears had higher total phenolic content than than control dried ones at drying temperatures of 50, 55 and 60°C, however, no significant differences ($p > 0.05$) were observed between all dried control pears and ultrasound pre-treated pears dried at 50 and 55°C. Among the all dried pears, the highest total phenolic content was 345.60 mg GEA/100 g db in ultrasound pre-treated pears dried at 60°C, but only this value of untreated pre-treated ones at 60°C was not statistically different from the fresh pears. Based on these results, the combined application of ultrasound pre-treatment and higher drying temperature of 60°C affected positively on the total phenolic content of pears. This behaviour could be explained by the higher processing temperature and less exposure time which contributed to a protective effect against oxidative and heat damage to the phenolic composition of pears. In addition, such situation may be attributed to the better availability and extractability of antioxidant compounds by an ultrasound application which can enhance to obtain larger pores in the pear tissue and thereby improving the extraction of polyphenols during the sample preparation (Amami et al., 2017; Wiktor et al., 2016).

The decrease of total phenolic compounds due to drying process may be associated with the modifications in the chemical structure of phenolic compounds or the binding of polyphenols with other compounds, such as proteins (Di Scala et al., 2011; Ek et al., 2017).

In this case, the ultrasound application with higher drying temperature (60°C) exhibited the better retain of antioxidant activity of pear samples.
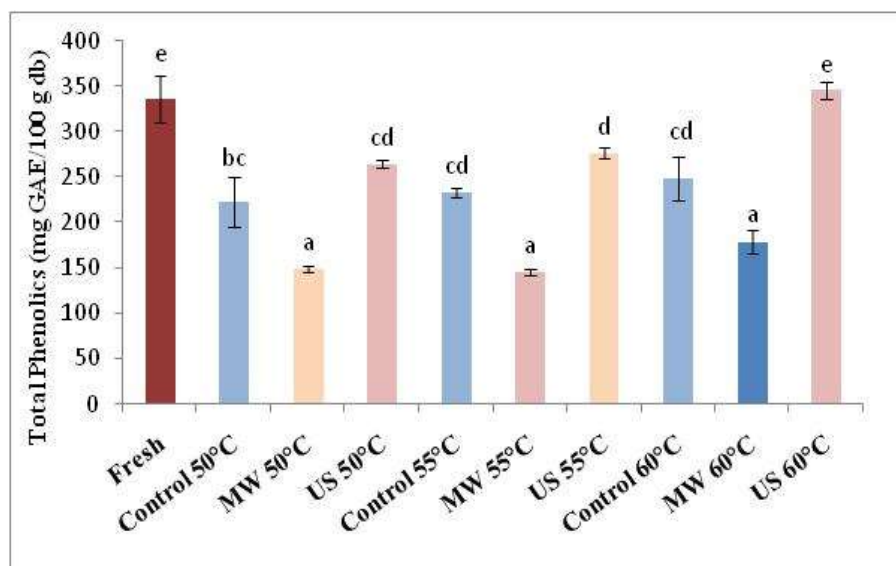
Figure IV.38 *Total phenolic content of fresh pears and control microwave and ultrasound pre-treated pears dried at 50°C, 55°C, 60°C*

IV.3.5 DPPH radical scavenging activity

The antioxidant activity of fresh pears and dried pears with different pre-treatments was evaluated by DPPH radical scavenging activity assay, which was given in Figure IV.39. The lowest EC_{50} corresponds to the highest antioxidant activity of pears. The fresh samples provided a radical scavenging activity of 9.39 mg/mL. After drying process, the dried pear samples had less antioxidant activity than fresh pears. (except for ultrasound treated pears dried at 60°C). No significant differences ($p > 0.05$) were found between the fresh samples and the ultrasound treated ones dried at 60°C. According to these results, the higher antioxidant activity value of dried pears was positively correlated with the higher drying temperature.In this case, the lower drying temperature produced more degradation of antioxidant activity in pear samples. Previous studies stated that the lowest antioxidant activity of fruits or vegetables of air dried samples was linked to longer drying temperature to low process temperature, and thus exposure to raise the temperature and oxygen (Garau et al., 2007; Nowacka et al., 2019; Vega-Gálvez et al., 2009).

The microwave treatment resulted in significantly less antioxidant activity in comparison with control and ultrasound pre-treated dried pears, demonstrating that some phenolic acids probably were degraded by microwave application. The antioxidant activity values of control and

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ultrasound pre-treated dried pears were significantly different ($p < 0.05$) than microwave treated dried ones. All pear samples were obtained from ultrasound pre-treatment exhibited better antioxidant properties, however there were no statistical differences ($p > 0.05$) between ultrasound pears dried at 55°C and control ones dried at 60°C. The ultrasound treated pears at higher drying temperature of 60°C showed that the better retention of antioxidant activity. Such situation was probably attributed to the protection of higher degradation of pear cellular structure by combined application (ultrasound treatment and higher drying temperature of 60°C) which may improve antioxidant activity of samples.

According to those obtained different results in antioxidant activity of dried pears, the observed DPPH profile may be associated with the modifications of chemical structure of the main antioxidant compounds in pear (i.e. arbutin, gallic acid, catechin, chlorogenic acid, epitachin acid,syringic acid) having a varying degree of antioxidant activity or interaction between not only phenolic compounds, and other pear constituents such as vitamin C, proteins by the combination of pre-treatments and drying temperature.

In our case, this proper combined drying method (ultrasound treatment and higher drying temperature of 60°C) may be efficient, which can preserve the antioxidant activity and phenolic compounds in 'Rocha' pear samples.

The total phenolic content and antioxidant activity influenced the white index (WI) of 'Rocha' pear. Total phenolic content showed a high positive correlation with white index ($R^2=0.928$), while the antioxidant activity (EC_{50}) was a high negative correlation with white index ($R^2= - 0.936$), because EC_{50} indicates the lower the EC_{50} value the higher the antioxidant activity of pear. If we evaluate this R^2 in terms of absolute value, it is possible to see high correlation.

Moreover, a high negative correlation was found among these parameters (TPC and EC_{50}) with a good agreement for the pear samples ($R^2= - 0.845$). If we evaluate this R^2 in terms of absolute value, it is possible to see high correlation.
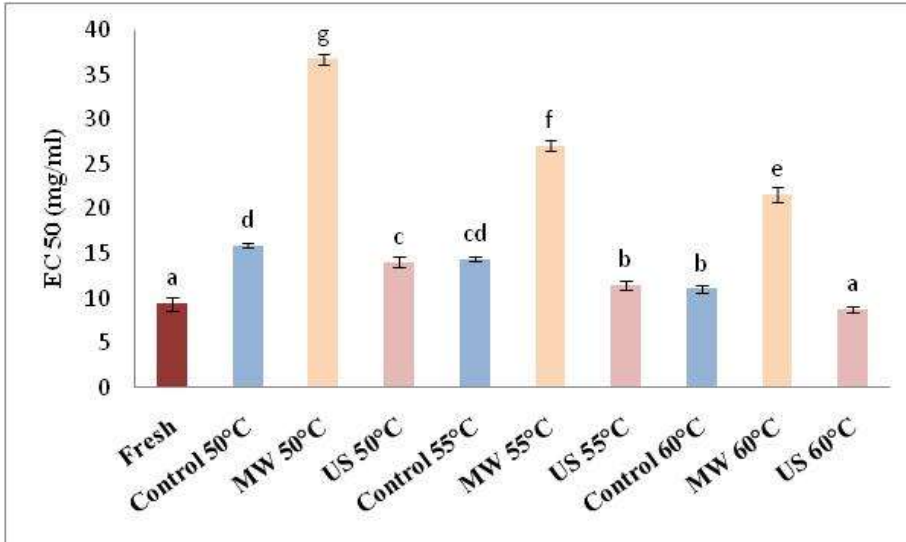
Figure IV.39 *Antioxidant activity of fresh pears and control, microwave and ultrasound pre-treated pears dried at 50, 55 and 60 °C*

IV.3.6 Shrinkage evolution

Shrinkage has a negative consequence on the quality attributes of dehydrated food products. Such physical phenomenon occurs during the drying process of foods when the viscoelactic matrix contracts into the space before occupied by moisture removed from the cells (Aguilera, 2003; Hafezi et al., 2013).

The effects of pre-treatments (microwave and ultrasound) and drying temperatures (50, 55 and 60°C) on shrinkage of pear slabs were demonstrated in Figure IV.40. It was seen that from Figure IV.40, the pre-treatments and drying temperature had significant ($p < 0.05$) effect on the shrinkage of pears. The lower drying temperature resulted in increasing of shrinkage in samples. The shrinkage of air dried of pear slabs was as83.90 %, 84.42 %, 81.74 % at air drying temperature of 50 °C for the control, microwave, ultrasound pre-treated samples, respectively. However, the shrinkage of pears dried at 60°C was found as 83.54 %, 82.96 %, 79.62 % for the control, microwave, ultrasound pre-treated samples, respectively. This case indicated that thelower drying temperature of 50°C reduced internal stress and promoted more shrinkage, which may be attributed to longer drying process time gives more time for the pear samples to shrink. These results are in agreement with previous works by Aral and Beşe (2016), Horuz and Maskan (2015), Yemmireddy et al. (2013). The microwave

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treated dried pears had lower shrinkage values than the control dried ones, but no statistical differences ($p > 0.05$) were observed between the microwave and control dried pears at each drying temperature. Ultrasound pre-treated samples significantly were less shrunk ($p < 0.05$) in comparison to control and microwave treated dried ones at drying temperatures of 50, 55 and 60°C. However, the ultrasond treated dried pears at drying temperature of 60°C produced less shrunk (79.62 %) pears, showing that this combined drying method (US 60°C) may be more effective on the shrinkage phenomenon due to protection of dried pear cell wall and tissue structure.Nowacka et al. (2016) reported that untreated carrot tissue had smaller cavities and irregular shape after drying. Ultrasound treatment (ultrasound frequency and sonication time) had remarkable influence on structural characteristics of carrot due to the sponge effect and the phenomenon of micro-channels. Another possible explanation may be related to reduced effect of the cavitation phenomenon in the isolation of pear samples from liquid medium by the sample vacuum-packaging. In this way, cavitation did not take place in the pear material and the cell integrity of pear tissue could not be more damaged. The combination between ultrasound pre-treatment (35 kHz-10 min) and the higher drying temperature of 60°C may provide the formation of micropores with more porous, uniform and larger pores which resulted in the mechanical stabilization of pear surface and limited degree of shrinkage.
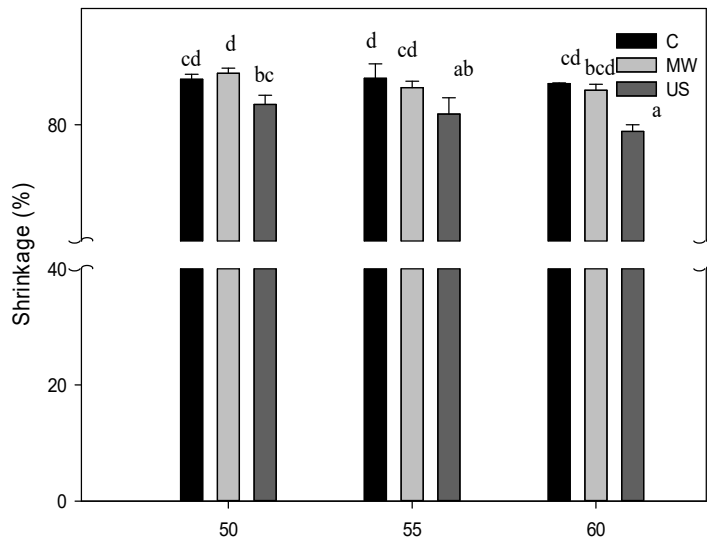
Figure IV.40 *Shrinkage of control (C), microwave (MW) and ultrasound (US) pre-treated pears dried at 50, 55 and 60°C*

IV.3.7 Texture

Texture evaluation is an importantconcept, determining the sensorial quality of dried fruit and vegetables (Chong et al., 2008; Dias da Silva et al., 2016). Textural attributes (hardness, springiness, cohesiveness, chewiness) of control, microwave and ultrasound pre-treated pears dried at 50, 55 and 60°C were presented in Table IV.16.The hardness has great importance as textural attribute for mouth feel of dried pear. According to Table IV.16, the drying temperatures and pre-treatments had significant impact on the hardness of dried samples. After drying, the highest hardness values were observed in microwave treated samples dried at 50°C, indicating this value was significantly different (p < 0.05) respect to control and ultrasound treated dried ones. Moreover, this situation resulted in an undesirable quality of dried pears for consumers. In general, control and ultrasound had a similar trend at drying temperatures of 50 and 55°C. Concerning of hardness results, the higher drying temperatures of 55 and 60°C had a more pronounced effect on the softening of samples, particularly, control and ultrasound pre-treated pears which is an attribute for obtaining high quality dried fruits. Springiness

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is based on the gelling agent in the fruits, evaluating the elastic behaviour of the samples. In concern with springiness, there were not significantly ($p > 0.05$) differences between all dried pears. It was seen that the drying process (i.e.temperature) had not remarkable effect on springiness. Similar observations were found by Guiné and Barroca (2012) for pumpkin and by Seerangurayan et al. (2019) for date. There were no significant differences between the fresh and dried fruits at different drying temperatures. These authors reported that drying process did not alter significantly the capacity of fruits (pumpkin and date) to return to their original shape after deformation. Chong et al. (2009) reported that the springiness of the commercially dried food products, for example, apricot, fig, kiwi, pear, persimmon, mango, cherry tomato was in the range of 0.754-1.007. The obtained springiness values of 'Rocha' pears were in this range, which is in good agreement.

Cohesiveness measures the rate at which the material dis-integrates under mechanical action. After drying, the analogous cohesiveness values were found inall dried pears (except for C55°C and C60°C), indicating these dried pears have similar strengths of internal bonding, however,control pear samples dried at 60°C ($p < 0.05$) were significantly higher than the other dried ones. This case may explained by the higher drying temperature which could damage the cell membrane structure during the drying process. In relation to chewiness, the higher drying temperatures (55 and 60°C) decreased significantly this parameter in all pear samples. The higher chewesiness values were found in microwave treated pears dried at 50°C, showing similar behaviour as hardness in this case. The lowest chewiness was observed in ultrasound treated pears dried at 55°C, however, no statistical differences ($p > 0.05$) were found between these samples: C55°C, US55°C, C60°C and US60°C. In addition ultrasound pre-treatment may contribute to less damage topear texture andprevent the collapse of tissue at higher drying temperatures (55 and 60°C).

Table IV.16 *Textural attributes of control (C), microwave (MW)and ultrasound (US) pre-treated 'Rocha' pears dried at 50,55, and 60°C*
Different lower letters (a, b, c…) in the same column mean significant diffrences (p < 0.05)

Sample	Hardness (g)	Springiness	Cohesiveness	Chewiness (g)
C50°C	2163.35 ± 148.8[bc]	0.751 ± 0.01[a]	0.655 ± 0.04[bc]	1037.33 ± 60.96[abc]
MW50°C	3222.27 ± 113.3[d]	0.800 ± 0.01[a]	0.615 ± 0.03[abc]	1571.30 ± 71.09[d]
US50°C	2354.18 ± 73.13[c]	0.796 ± 0.01[a]	0.666 ± 0.06[c]	1279.54 ± 61.18[cd]
C55°C	1788.72 ± 221.9[ab]	0.753 ± 0.05[a]	0.557 ± 0.02[a]	724.52 ± 122.90[ab]
MW55°C	2110.39 ± 254.9[bc]	0.807 ± 0.03[a]	0.567 ± 0.04[ab]	951.79 ± 177.65[abc]
US55°C	1599.80 ± 110.43[a]	0.723 ± 0.01[a]	0.598 ± 0.04[abc]	681.55 ± 114.31[a]
C60°C	1437.14 ± 113.60[a]	0.760 ± 0.01[a]	0.670 ± 0.00[c]	736.91 ± 9.01[ab]
MW60°C	2217.86 ± 223.77[bc]	0.790 ± 0.00[a]	0.610 ± 0.01[abc]	1098.64 ± 167.61[bcd]
US60°C	1859.20 ± 145.03[ab]	0.750 ± 0.00[a]	0.630. ± 0.02[abc]	885.69 ± 105.20[ab]

IV.3.8 Rehydration capacity

Rehydration is a process that specifies one of the most important quality attributes of dehydrated products. Such process depends on the used pre-treatments prior to drying, food material properties and drying/rehydration conditions (Ricche at al., 2016).

Figure IV.41(a-c) reported that the influence of pre-treatments (microwave and ultrasound) and drying temperatures (50, 55 and 60°C) on the rehydration behaviour of the pear samples. For all rehydration experiments showed that the amount of water uptake increased at the initial stage of process, however, with increasing rehydration time at the last stage of the process the rate of water uptake decreased when reached the saturation levels.

The rehydration tests demonstrated that the rehydration capacity of dried pears increased with increasing drying temperature due to the effect of

temperature on the cell wall and tissue of pear. Moreover, the ultrasound and microwave pre-treatment improved the rehydration behaviour (i.e. weight gain %) of pear slabs in comparison with the control ones at all investigated drying temperatures, however, the ultrasound pre-treated pears exhibited a higher rehydration capacity than microwave treated dried ones.

Related to ultrasound pre-treatments, several researchers have been stated an improved of rehydration properties of dried fruits and vegetables: Ricce et al. (2016) reported that carrot samples subjected to ultrasonic pre-treatment at both 30 and 60 min were greater rehydration capacity than respect to untreated ones, which may be associated with the higher porosity and micro-channel formation. Thus, this higher porosity allowed the better entrance of water during the rehydration process. Fijalkowska et al.(2016) Szadzińska et al.(2018) indicated that the rehydration capacity and the level of rehydration improvement are based on the degree of cellular and structural disruption by the ultrasound application. The combination of ultrasound and ethanol pre-treatment contributed to the improvement of both the rehydraton rate and water retention of pumpkin samples (Rojas et al., 2019). In this present study, the rehydration capacity of pears with ultrasound pre-treatment and dried at 60°C were higher than those of dried ones at other temperatures. This combined effect of ultrasound and higher drying temperature of 60°C may be explained by the rapid drying process which provided the more porous pear structure and thus, resulted in the higher water penetration. The obtained results indicated that the rehydration process efficiency (i.e. rehydration rate and rehydration capacity) was influenced by the structural changes produced by ultrasound application (ultrasound temperature and time, type of liquid medium) and drying temperature.

In concern with microwave pre-treatment, the pear samples with microwave application had the greater rehydration capacity than control ones in all cases. This behaviour may be attributed to the formation of pear structure during the microwave application, and became more porous due to faster heating and better moisture removal. This porous structure could increase the rehydration capacity and improve the reconstruction of the dried pear structure. Dehghannya et al. (2019) reported that the microwave application increases the internal vapor pressure, especially at higher microwave power, which creatives the non-compact structure because of the vapor travelling through the food product, shorter microwave exposure time. These observed structure modifications in food materials contribute to less shrinkage and better rehydration capacity. Starting from this point, microwave power and time, as well as air drying temperature have a considerable effect on the rehydration behaviour of food materials.

In conclusion, these results indicated that ultrasound and microwave pre-treated pears dried at higher drying temperature of 60°C, presented the higher rehydration capacity, particularly, ultrasound pre-treatment ones. This combined method for both pre-treatments at 60°C may have the lower cell

disruption and tissue damages in pear samples and thus improving rehydration behaviour. In this case, this combined method may be useful to preserve the 'Rocha' pear structure during the rehydration process.
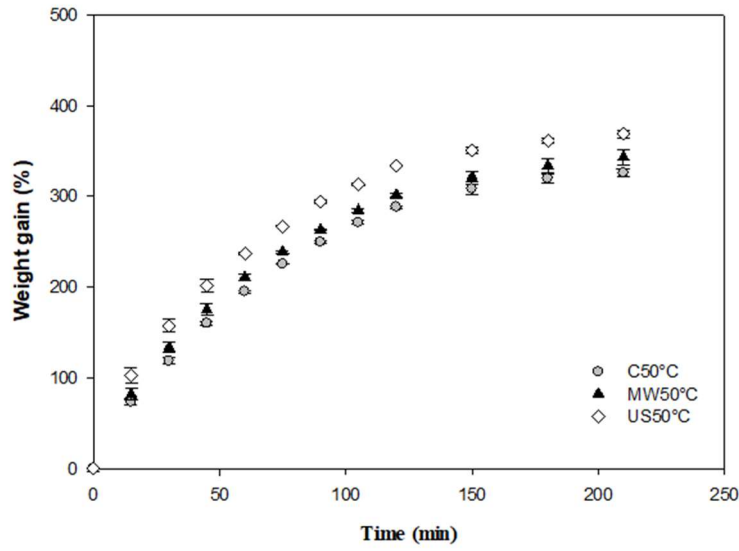
a)
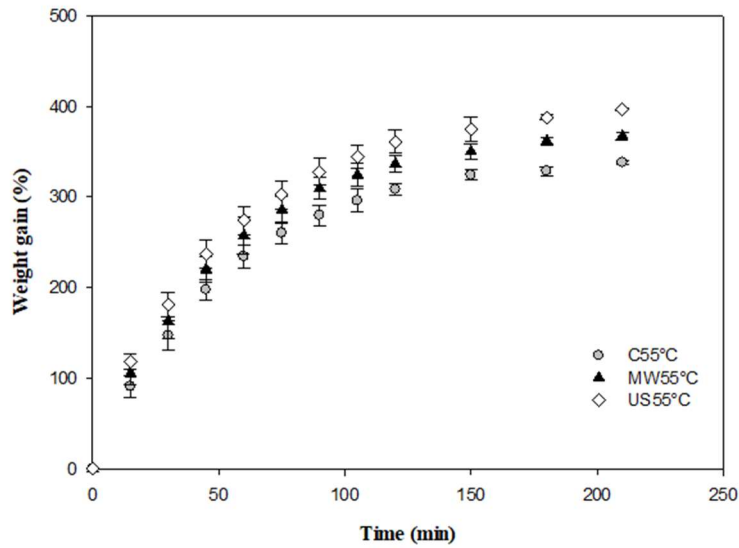
b)
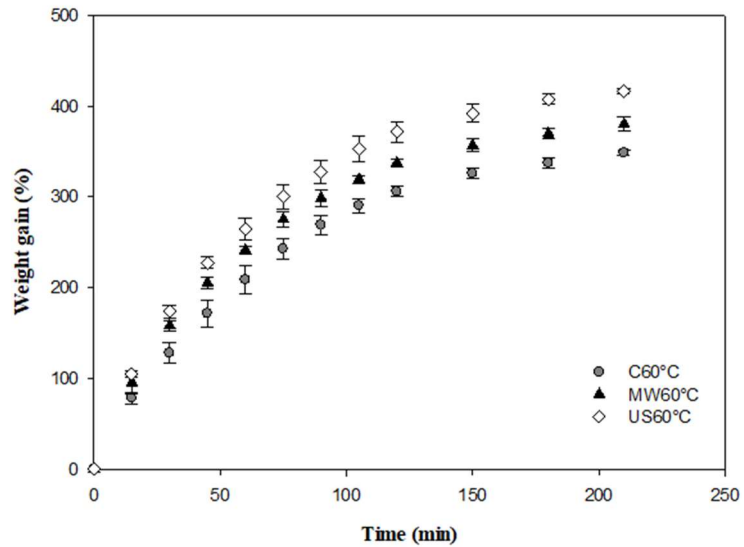
c)

Figure IV.41 *Rehydration kinetics of control (C), microwave (MW) and ultrasound (US) pre-treated pears dried at 50°C (a), 55°C (b) and 60°C (c)*

IV.3.9 Conclusions

The influence of air drying temperature (50, 55 and 60°C) combined with different pre-treatments as microwave and ultrasound on drying characteristics were evaluated and some quality parameters of both fresh and dried 'Rocha' pears were also studied. It was observed that the drying time decreased with increasing drying temperature from 50 to 65°C. In addition, microwave pre-treatment had shorter drying time than control and ultrasound pre-treatment. However, ultrasound pre-treatment did not accelerate the drying process. It seems that the sponge effect is the main physical phenomena affecting the pear samples. Because, pear slabs were isolated by vacuum-packaging from the liquid medium during the ultrasonic treatment to reduce the effects of cavitation phenomena. Due to this type of treatment procedure before ultrasound application, probably cavitation phenomenon did not exist and only the sponge effect occurred. Therefore, the occurrence of such phenomenon only was insufficient to change the drying curves. The combined application of ultrasound pre-treatment and higher drying temperature (60°C) showed lower total colour changes, less shrinkage, better retention of total phenolic and antioxidant activity and higher rehydration capacity. On the other hand, microwave treated dried pears exhibited less

quality attributes. The total phenolics and antioxidant activity of samples were better retained at higher drying temperature (60°C). This case was mainly attributed to shorter drying time and therefore less exposure of the phenolics to thermal effect. Concerning with texture evaluation, the highest hardness and chewiness values were observed in microwave pears dried at 50°C. The higher drying temperatures (55 and 60°C) may be better for textural properties of control and ultrasound pre-treated dried samples.

Consequently, this preliminary study may be important to understand the effects of different pre-treatments (microwave and ultrasound) on the dried 'Rocha' pear quality. Additionally, the combined application of ultrasound pre-treatment and higher drying temperature (60°C) seems to promising technique to obtain high quality dried pear snacks.

Chapter V
Conclusions

The choice of appropriate pre-treatments and drying methods are one of the most important key factors for obtaining the high quality dried snacks in thedehydrated food industry. The selection of each pre-treatment and drying system are depend on the food product and its market value. In addition, the nutritional compounds' degradation and their bioactivity are related to the pre-treatment's and drying processing's conditions, particularly, time and temperature, and their control. From this viewpoint, the optimization of pre-treatment and drying conditions contribute to improve the overall quality attributes of dried fruits and vegetables.

In this work, the influence of different innovative and natural dipping pre-treatments, as well as physical pre-treatments (microwave and ultrasound) and hot air drying conditions (i.e. temperature and time) were investigated on the quality attributes of selected fruits ('Annurca'apple, 'Terzarola gialla'peach, and 'Rocha'pear) in terms of drying characteristics, colour, shrinkage, total phenolic content, antioxidant activity, volatile aroma compounds(VOCs), texture, microstructure, preliminary sensory evaluation, rehydration behaviour. The optimal process conditions were determined for each investigated fruit to obtain high quality dried snacks. The most important key point is that these proposed innovative and natural pre-treatments do not contain any chemical substances (i.e. sulphates) which may cause health problems such as asthmatic and allergic reactions in sensitive people. Moreover, some parts of this thesis were carried out in collaboration with the Centre for the Biotechnology and Fine Chemistry (CBQF) of the Catholic University, Porto, Portugal,in there the effects of combined application of microwave and ultrasound pre-treatments and air drying temperatures were studied on the evaluation of the dried 'Rocha' pear's quality. This study allowed to understand the efficiency and availability of microwave and ultrasound applications as a pre-treatment for the improvement of the dried product quality.

In this context, this work may be useful to provide information related to the development of innovative and natural pre-treatments' conditions prior to

the drying process of fruits. Moreover, the use of ultrasound as a pre-treatment could bebeneficial to conventional hot air drying process for improving the efficiency of fruits' drying.

Therefore, the combined drying pre-treatments and optimal drying conditions may seem to be a suitable process to produce high quality and health dried snacks for both consumers anddehydrated food industry. In addition, this combined drying techniques can be accepted as an alternative cost- effective method to reduce the drying times for industrial applications.

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Abbreviations and Symbols

Abbreviations

AD	Air drying
B.C.	Boundary conditions
C	Control samples
Ca^{+2}	Calcium ion
$CaCl_2$	Calcium chloride
C.I.	Confidence interval
CO_2	Carbon dioxide
D	Dried product
DHC	Dry matter holding capacity
DPPH	1,1-diphenyl-2-picrylhydrazyl solution
EC_{50}	Sample concentration (mg ml^{-1}) required to inhibit 50% of the DPPH radical scavenging activity (mg mL^{-1} sample)
I.C.	Initial conditions
KOH	Potassium hydroxide
MW	Microwave pre-treatment
NaCl	Sodium chloride
Na_2CO_3	Sodium carbonate
POD	Peroxidase
PPO	Polyphenol oxidase
R	Rehydrated product
RA	Rehydration ability
RMSE	Root mean square error
SE	Standart error of the parameter
Sh	Sherwood number
TPA	Texture profile analysis curve
TPC	Total phenolic content (mg GAE/100 g db)
TR	Treated samples

UOD-AD	Combination of ultrasound assisted osmotic dehydration pre-treatment and convective air drying
US	Ultrasound pre-treatment
USCS	Combination of ultrasound and vacuum
UTR	Untreated samples
VI	Vacuum impregnation
VOCs	Volatile organic compounds
WAC	Water absorption capacity
WHC	Water holding capacity
WI	White index

Symbols

a	Drying coefficient
a*	Colour parameter (greeness/redness)
a_w	Water activity
$abs_{control}$	Absorbance of control
abs_{sample}	Absorbance of sample
b*	Colour parameter (blueness/yellowness)
C_0	Initial moisture content of the samples
C constant	Gugghenheim constant
c	Dimensionless water content in the differential volume
D_0	Effective moisture diffusivitiy
D	Effective diffusion coefficient(cm^2/s)
D_{eff}	Diffusion coefficient (m^2/s)
E_a	Active energy (kJ/mol)
H°	Hue angle
h_m	Moisture transfer coefficient (m/s)
K	A factor correcting properties of the multilayer molecules with respect to the bulk liquid
k	Drying coefficient
k_m	Overall transport coefficient
M	Total mass (g)
M_{CF}	Amount of liquid
M_0	Initial moisture content (kg water/kg dry matter)
M_e	Equilibrium moisture content (kg/kg$_{d.b.}$)
M_t	Moisture content of a given drying time (kg water/kg dry matter)
M_{sur}	Moisture at the surface
m_i	Initial mass (g)

m_f	Final mass (g)
N	Drying coefficient
R	Universal gas constant (J/mol.K)
R^2	Coefficient of determination
R_0	Radius of the sample (m)
S (%)	Shrinkage
S_h	The ratio of the convective mass transfer to the ratio of diffusive mass transport
s	Standart deviation of the experimental error
T	Temperature
t	Temporal time (s)
t	Time (min)
u	Shrinkage velocity
u_0	Shrinkage velocity coefficient (cm/s)
V_0	Initial volume of samples
V_t	Final volume of samples
x	Spatial coordinate along which material transport takes place (cm)
x_i	Mass fraction component(g/g)
X_e	Average equilibrium moisture content
X_f	Final moisture content(kg water/kg dry matter)
X_i	Initial moisture content(kg water/kg dry matter)
X_m	Water content on a dry basis corresponding to the monolayer value
χ^2	Reduced chi square
z_R^{ss}	Liquid phase
ΔE	Total colour difference
τ	Dimensionless time
ρ_s	Solid density (kg/m^3)