University of São Paulo
"Luiz de Queiroz" College of Agriculture

Enhancing carrot convective drying by combining ethanol and ultrasound as pre-treatments: effect on product structure, quality, energy consumption, drying and rehydration kinetics

Karoline Costa dos Santos

Dissertation presented to obtain the degree of Master in Science. Area: Food and Science Technology

Piracicaba
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Food Engineer

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versão revisada de acordo com a resolução CoPGr 6018 de 2011

Advisor:
Prof. Dr. **PEDRO ESTEVES DUARTE AUGUSTO**

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DEDICATION

I dedicate this work to all those who believe and support my dreams and allowed me to take on this new challenge. All those who directly or indirectly accompanied me on this academic journey of great personal and professional growth, transmitting positive thoughts and prayers. And to all the family and friends who always offered me pleasant company.

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EPIGRAPH

“When life gets you down, do you wanna know what you've gotta do?
Just keep swimming!”

(Dory, Finding Nemo, 2003)

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RESUMO

Melhorando a secagem convectiva de cenoura combinando etanol e ultrassom como pré-tratamentos: efeito na estrutura, qualidade do produto, energia comsumida, cinética de secagem e reidratação

Este trabalho avaliou pré-tratamentos com etanol e/ou ultrassom para a secagem convectiva de cenouras, avaliando também mudanças estruturais, parâmetros de qualidade tecnológica e nutricional e descrevendo os mecanismos envolvidos. Os pré-tratamentos com etanol e ethanol+ultrassom modificaram tanto a microestrutura da cenoura (modificações da parede celular do tecido parenquimático) quanto a macroestrutura (contração e resistência à perfuração). Diferentemente, do ultrassom em água que resultou em um inchaço celular e comportamento de textura semelhante ao controle. Após a secagem, não foram observadas diferenças na taxa de retração em todos os tratamentos. Os pré-tratamentos com ethanol e ethanol+ultrassom melhoraram a cinética de secagem, reduzindo o tempo de processamento (~ 50%) e o consumo de energia (42- 62%). Esses pré-tratamentos também aumentaram a reidratação, cuja taxa inicial e retenção de água foram maiores que o controle. Além disso, o conteúdo de carotenóide foi preservado após a secagem, para todos os tratamentos. Portanto, este estudo aponta uma alternativa para aumentar a produção de alimentos estáveis com melhores processos de secagem e reidratação, sem comprometer a qualidade.

Palavras-chave: Secagem convectiva, Reidratação, Etanol, Ultrassom

ABSTRACT

Enhancing carrot convective drying combining ethanol and ultrasound as pre-treatments: effect on product structure, quality, energy consumption, drying and rehydration kinetics

This work evaluated pre-treatments with ethanol and/or ultrasound to the convective drying of carrots, also evaluating structural changes, technological and nutritional quality parameters and describing the involved mechanisms. The pre-treatments with ethanol and ethanol+ultrasound modified both carrot microstructure (cell wall modifications of parenchymatic tissue) and macrostructure (shrinkage and resistance to perforation). Differently, ultrasound in water resulted in cellular swelling and texture behaviour similar to the control. After drying, no differences in shrinkage ratio were observed in all treatments. However, pre-treatments with ethanol and ethanol+ultrasound improved the drying kinetics, reducing the processing time (~50%) and the energy consumption (42- 62%). These pre-treatments also enhanced rehydration, whose initial rate and water retention were higher than the control. In addition, the carotenoid content was preserved after drying, for all the treatments. Therefore, this study describe an alternative to increase the production of stable foods with improved drying and rehydration processes, without compromising quality.

Keywords: Convective drying, Rehydration, Ethanol, Ultrasound

RESUMEN

Mejora del secado convectivo de zanahoria combinando etanol y ultrasonido como pretratamientos: efecto en la estructura del producto, calidad, consumo de energía, cinética de secado y rehidratación

Este trabajo evaluó pretratamientos con etanol y / o ultrasonido para el secado convectivo de zanahorias, evaluando también cambios estructurales, parámetros tecnológicos y de calidad nutricional y describiendo los mecanismos involucrados. Los pretratamientos con etanol y etanol + ultrasonido modificaron tanto la microestructura de la zanahoria (modificaciones de la pared celular del tejido parenquimático) como la macroestructura (encogimiento y resistencia a la perforación). De manera diferente, los ultrasonidos en agua dieron como resultado una hinchazón celular y un comportamiento de textura similar al control. Después del secado, no se observaron diferencias en la relación de contracción en todos los tratamientos. Sin embargo, los pretratamientos con etanol y etanol + ultrasonidos mejoraron la cinética de secado, reduciendo el tiempo de procesamiento (~ 50%) y el consumo de energía (42-62%). Estos pretratamientos también mejoraron la rehidratación, cuya tasa inicial y retención de agua fueron más altas que el control. Además, el contenido de carotenoides se conservó después del secado, para todos los tratamientos. Por tanto, este estudio describe una alternativa para incrementar la producción de alimentos estables con procesos mejorados de secado y rehidratación, sin comprometer la calidad.

Palabras clave: Secado por convección, Rehidratación, Etanol, Ultrasonido

FIGURE LIST

LIST EQUATIONS

Eq. (1); (5)	Page model	$MR(t) = \exp(-kt^n)$
Eq. (2)	Absolute power	$P = mCp(\frac{dT}{dt})$
Eq. (3)	Volumetric power	$P_V = (\frac{P}{v})$
Eq. (4)	Experimental dimensional moisture	$MR(t) = \frac{M_t - M_e}{M_p - M_e}$
Eq. (6)	Estimative of the total energy consumption	$TEC = \frac{E_{PT} + E_D}{m}$
Eq. (7)	Energy consumption during pre-treatments	$E_{PT} = W.V.t_P$
Eq. (8)	Energy consumption during convective drying	$E_D = A.v.\rho_a.Cp_a.\Delta T.t_D$
Eq. (9)	Peleg model	$M(t) = M_0 + \frac{t}{k_1 + k_2.t}$
Eq. (10)	Carotenoid content	$\left[cc\left(\frac{\mu g}{g}\right)\right] = \frac{A_{450} x\ 536{,}85\ x\ V}{m\ x\ 137{,}4}$
Eq. (11)	Sum of squared errors	$SSE = \sum_{I=1}^{X}((PRETICTED) - (EXPERIMENTAL)_I^2$

LIST ABBREVIATIONS

Ethanol+US	Ethanol+Ultrasound
Water+US	Water+Ultrasound
In natura	Slices of carrot without any processing
MR(t)	Dimensionless moisture (Eq.(1);(5))
d.b	Dry basis
w.b	Wet basis
M_t	Moisture (%d.b) the drying process time (t) (Eq.(4))
M_e	Moisture equilibrium (d.b) (Eq. (4))
M_p	Weight the sample *in natura* (d.b) (Eq.(4))
k	Diffusion coefficient (h^{-n}) (Eq. (1))
n	Type diffusion (Eq. (1))
TEC	Estimative of the total energy consumption (Eq. (6))
M	Mass *in natura* sample (kg) (Eq.(6))
W	Volumetric power (W/L) (Eq. (6))
V	Volume of Water or Ethanol (L) (Eq.(6))
t_p	Time pre-treatment (Eq.(7))
E_{PT}	Energy consumption during pre-treatments (Eq.(7))
E_D	Energy consumption during convective drying (Eq.(8))
A	Cross-sectional area of drying (m^2) (Eq.(8))
P	Ambient air density (Eq.(8))
Cp	Specific heat capacity (J/kg.k) of ambient air (Eq.(8))
ΔT	Difference between the ambient air and drying air (Eq.(8))
V	Air velocity (m/s) (Eq.(8))
t_D	Time need to the samples reach a moisture (20% w.b) (Eq.(8))
M(t)	Moisture content (d.b) in instant (t) (Eq.(9))
M_0	Initial moisture content (d.b) (Eq.(9))
K_1	Maximum water absorption rate (min $d.b^{-1}$) (Eq.(9))
K_2	Water retention capacity ($d.b^{-1}$) (Eq.(9))
CC	Carotenoid content (µg/g) (Eq.(10))
V	Volume of the hexanoic phase (mL) (Eq.(10))
M	Mass the sample used (g) (Eq.(10))

1. INTRODUCTION

Drying is an important operation in the food industry, producing safe and stable products, and also reducing post-harvest losses. This operation allows obtaining various products such as snacks, soups and dried fruits (MALEKI et al., 2019), which can be consumed directly or after rehydration.

Drying has numerous advantages in food preservation. However, the conventional convective drying is a long process, which also presents a high energy consumption (MOTEVALI; MINAEI; KHOSHTAGAZA, 2011). Moreover, the long processing time, associated to high temperatures, can result in undesirable changes, such as nutrient degradation or poor rehydration capacity.

Therefore, different strategies are being studied to enhance food drying, including the application of pre-treatments (LLAVATA et al., 2020). In this context, both ethanol and ultrasound can be used as a promising alternative in food processing.

The pre-treatment with ultrasound has been studied in different products, while the studies using pre-treatments with ethanol are now increasing. However, the combination of both approaches (conducting ultrasound processing with ethanol) was only recently proposed. Rojas et al. (2020) conducted the only work combining ethanol and ultrasound (Ethanol+US) pre-treatments to convective drying, with pumpkin pulp. The combined treatment reduced the drying time and the energy consumption during processing, improved the rehydration and avoid carotenoid degradation. The same combination (Ethanol+US) was also proposed prior to apple convective drying (ZUBERNIK et al., 2019), with smaller processing time (3 min). They obtained significative reduction of drying time, although the combination did not minimize the degradation the phenolic compounds. The combination Ethanol+US was also evaluated on melon, with two ethanol concentrations (50 and 100%) and convective drying at 60 ° C. The treatment with a higher concentration of ethanol obtained shorting drying time, but there was degradation of phenolic compounds, ascorbic acid and carotenoids, when compared to dry samples without treatment and fresh melons (DA CUNHA et al., 2020). However, in all woks, the authors did not evaluate the product structure nor performed the pre-treatment using water in the ultrasonic bath. Moreover, the three works evaluated homogeneous vegetables, while it would be interesting to study a matrix with different structures.

Three other works employed the combination Ethanol+US, but as pre-treatment to infrared drying techniques whose mechanisms are different from convective drying. Thesse pre-treatments were evaluated prior to the infrared drying of potatoes (ROJAS; AUGUSTO, 2018c) and garlic (FENG et al., 2019) reducing the drying time. However, the rehydration properties of potatoes were impaired due to the structure modification associated its composition, while allicin losses were registered in the garlic samples. The Ethanol+US combination was also investigated in pulsed vacuum drying of apple, reducing the drying time and liberating free amino acids (AMANOR-ATIEMOH et al., 2020). These different results reinforce the need to evaluate other structures and quality parameters by using these pre-treatments.

Therefore, although being promising, the combination of ethanol and ultrasound (Ethanol+US) technologies as pre-treatments to convective drying still needs to be better understood. Particularly, its effect on product structure, and the subsequent impact on processing, and how different tissues are affected by those pre-treatments still must be better described. This work is based in this context.

Carrot was selected as food matrix for drying, not only due to its commercial importance, but also as this vegetable can be consumed both directly dried (as a health-claim snack or composing other products, such as cereal bars, breakfast cereals, granola, cookies, etc) or after rehydration (as in soups, puree, creams or cakes, among other possibilities), and also due to its differences in structure and firmness of the pulp - which allow a better understanding of microstructure effects. In addition, carrot has an appreciated nutritional composition, due to its high carotenoid content, which is interesting in this study as a quality parameter. Moreover, carrots exhibit two distinct structural regions (cortex and core) with parenchymatic tissue and two vascular tissues (xylem and phloem), representing a typical anisotropy of food matrix. Their complex structure can provide an important opportunity to describe the mechanisms of mass transfer during drying under a more realistic perspective.

Consequently, the present work studied the effect of ethanol pre-treatment along with ultrasound on carrot structure and convective drying, also evaluating the energy consumption, and quality parameters (evaluated through the rehydration kinetics and carotenoid retention).

This work was already published on Ultrasonics Sonochemistry (APPENDIX B; *DOI: 10.1016/j.ultsonch.2020.105304*).

2. OBJECTIVES

Study the effect of ethanol pre-treatment along with ultrasound on carrot convective drying and to evaluate the effect on drying kinetics and product quality (rehydration kinetics and carotenoids content).

2.1 Specific objectives

- To investigate the influence of using ultrasound together with ethanol on drying kinetics, rehydration kinetics and microstructure of carrot slices;
- to apply the mathematical model and describe the drying and rehydration kinetics;
- to identify structural changes in the parenchyma and xylem tissues with the use of pre-treatments;
- to evaluate sample shrinkage after pre-treatment and drying, in the different steps of processing and using different approaches;
- to evaluate the effect of pre-treatments on carrot nutritional quality, evaluated by the total carotenoids content;
- to identify the best treatment for the convective drying of carrot slices.

3. LITERATURE REVIEW

3.1. Food Drying

Drying is an ancient and effective preservation technology for food products. This unit operation comprises both heat and mass transfer mechanisms, being carried out to remove water from the product. Consequently, drying assure stable products, also improving logistics.

When a solid is subjected to drying, two processes occur simultaneously (MUJUMDAR; OSMAN, 2006): 1st - Energy transfer from the environment in which the solid is located, which is used to remove moisture from the product surface to the ambient (mass transfer); 2nd - Transfer of internal moisture to the surface of the solid, which is then removed by the 1st process. In the 1st process, the removal of water vapor from the surface will depend on the external conditions of temperature, air velocity and relative moisture, the food exposure area and the total pressure, among others. In the 2nd process, the internal movement of the water in the food will depend on the constitution of its nature, the temperature and the moisture content. In fact, different mechanisms of mass transfer can take place in this process, such as diffusion and capillarity, among others.

The mechanism for removing moisture through the above-mentioned processes can be obtained by different methods. Figure 1 describes a possible classification of drying processing types (MCMINN; MAGEE, 1999). Although convective drying is the most used method, different improvements have been developed (LLAVATA et al., 2020; RODRÍGUEZ et al., 2018).

The most common drying method for food products is the convective drying. In this method, heat is supplied by the hot air that travels over the surface of the solid. The hot air also solubilizes the water removed from the product. As the air circulates, the water is removed and transported, making the air moist. There are several types of direct convective dryers, among them convective tray dryers.

Figure 2 shows a convective tray dryer, similar to that used in the present work. For drying, ambient cold air (represented by the blue arrow) is collected from the external environment of the system and passes through a sensor that measures the air inlet velocity (a). Then, the air is heated (b, represented by the red arrows), passes through a temperature and humidity sensor (c) and through the food product. During the contact of the hot air with the food, the exchange of heat occurs through convection,

and the air gets moist and cold (represented by the green arrows). The temperature and humidity of the outlet air is monitored by the sensor (e). To make hot air renewal more efficient, a fan is used to suction the exhaust air (f). Moisture loss is monitored continuously through three load cells, which measure the sample weight during drying in real time (d).

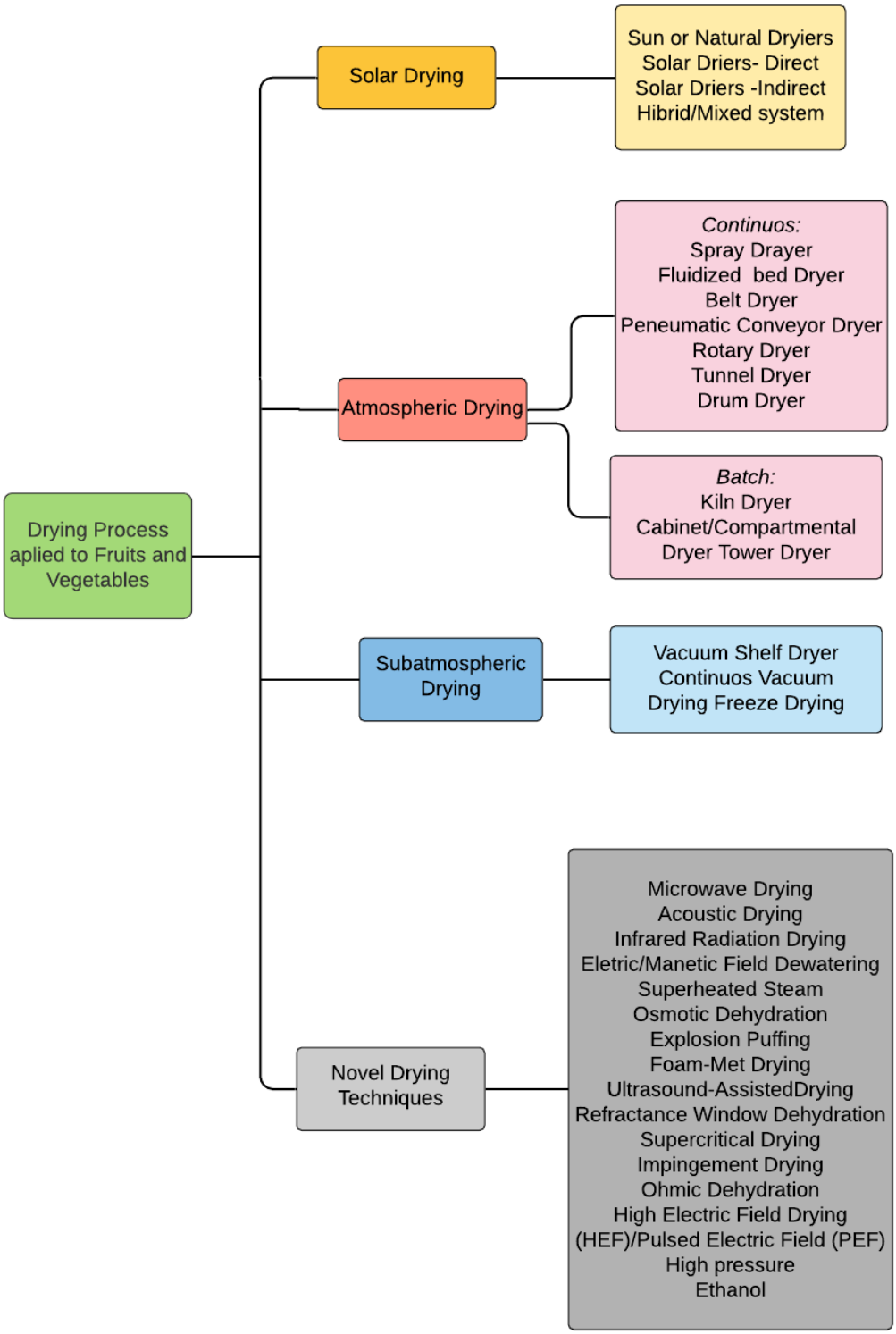

Figure 1: Drying type classifications adapted of Mcminn; Mage (1999).

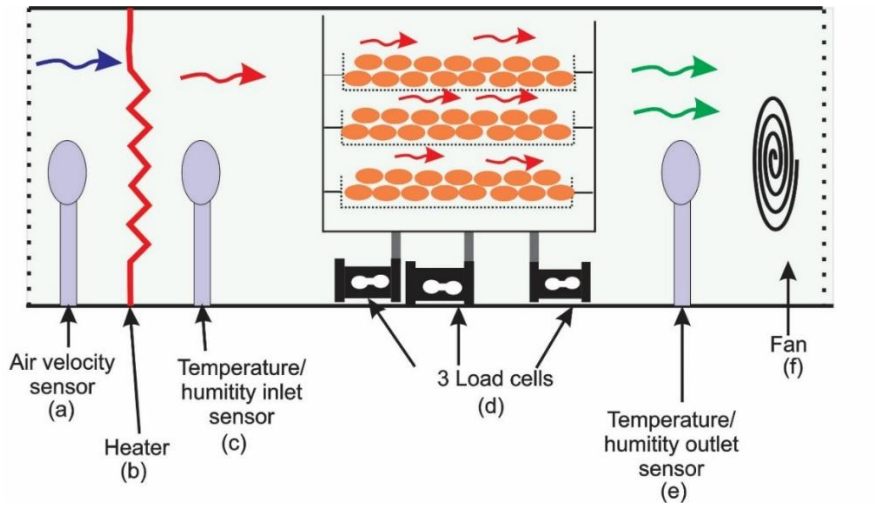

Figure 2: Schematic of the tray drier used in this work.

Figure 3 expresses the behaviour of a typical drying curve, which can be classified into three distinct zones: the induction period, constant rate period and falling rate period (GRAU; ANDRES; BARAT, 2014; MUJUMDAR; OSMAN, 2006). The induction period is the first stage in drying process. In this initial period, there is the removal of free water from the solid surface by means of vaporization, which causes an increase in the temperature of the food surface to a wet bulb temperature. Upon reaching the wet bulb temperature, the period of constant rate begins. In the second stage, the absorbed heat is used to remove water that flows from the centre to the surface of the solid. As the water is removed and the product dries, the rate of water transport to the surface decreases. The significant reduction in moisture removal characterizes the end of the period of constant rate, which is finalized reaching the critical moisture content (xe) in a given time (tc, Figure 3). As the product dries, it becomes even more difficult to remove the internal moisture from the solid, as it is strongly protected by a layer of already dry product. Therefore, the last stage is characterized by the falling rate period or "diffusion period". In this stage the water is diffused from the centre through the surface and then from the surface into the air. In this period, the drying rate decreases even more rapidly, and the product's moisture decreases until it reaches the equilibrium moisture (xe, Figure 3). The equilibrium moisture characterizes the end of the drying processing, where no more water flows out and moisture loss becomes constant.

Convective drying in a try dryer is considered a thin layer drying. Thin-layer drying is defined when a thin, uniform layer of material is fully exposed to air flow during drying (ONWUDE et al., 2016). Figure 3 illustrates the thin layer drying behaviour in a dryer exposed to the air stream, such as try dryers. The diagram depicted describes the drying of a single uniform layer of material with the same thickness, which allows the mathematical estimates of drying kinetics to be applied.

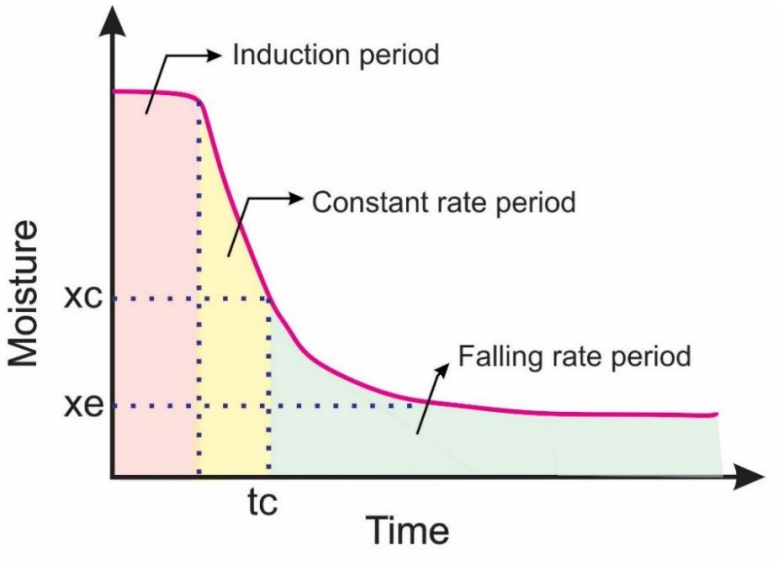

Figure 3: Typical drying curve of food products (without scale).

Several models of drying thin-layer fruits and vegetables are present in the literature. The models can be classified into theoretical, semi-theoretical and empirical (ONWUDE et al., 2016). These models mainly involve drying kinetics and describe the influence of certain variables on the mass transfer mechanism (KROKIDA; MARINOS-KOURIS, 2003).The application and choice of model will depend on factors such as drying temperature, air velocity, material thickness, relative humidity, initial moisture content and the objective of evaluation.

Kucuk et al. (2014) reviewed the models used in the literature from 2003 to 2013 for drying agricultural materials in a thin layer system, identifying 67 models. The authors concluded that the Page Model was the second-best model for fitting data of several products.

The Page Model (Eq. (1)) was obtained by (PAGE, 1949) after modifying the first order model (LEWIS, 1921). The modification made by Page resulted in the

addition of an empirical parameter with no dimension (*n)* in the first-order kinetics. Moreover, the parameter *k* (s^{-n}) represents the kinetics parameter.

$$MR = \mathrm{Exp}(-kt^n) \qquad \text{Eq. (1)}$$

The Page Model has been widely used to describe food drying, in general as an empirical interpretation. However, recent studies started to interpret Page Model and describe it phenomenologically (Simpson et al. (2017)). According to the authors, the dimensionless parameter *n*, can interpret the mechanism of water flow inside the product: when *n* = 1, the process is pure diffusional, whereas *n* > 1 represents a super diffusive behaviour and *n* < 1 a sub-diffusive behaviour. Moreover, the parameter *k* is associated with the “diffusion” rate and the sample format.

Based on that, further interpretation started to be conducted, such as by correlating the mass transfer with the microstructure of the food matrix. For instance, Rojas, Augusto (2018b) interpreted that when the *n* > 1, other mechanisms of mass transfer occur beyond diffusion, such as capillarity, which was demonstrated through xylem vessels of pumpkin.

To advance this discussion, it is necessary to study matrices with different structures, with natural channels (such as xylem and phloem) that can assist the water flow during drying. Carrots are an interesting alternative to conduct this study. Carrots exhibit two distinct structural regions (cortex and core) and both vascular tissues (xylem and phloem) - which is more detailed in the section 3.5. Therefore, carrot was used in this work.

In addition to the detailed study of the work matrix, it is necessary to obtain the best advantages in terms of preservation, quality and cost. For this, new drying improvement techniques can be employed, such as the use of ethanol and ultrasound as a pre-treatment. These pre-treatments induce structural changes in the matrix, improving the mass transfer mechanisms, reducing the drying time and presenting potential of reducing operational costs.

Therefore, the use of ethanol / ultrasound can be a promising alternative in food processing, with a simple application approach and with excellent results in improving drying and dry food quality.

3.2 Use of ethanol as drying accelerator

Drying accelerators are compounds that foster drying, by different mechanisms.

Among different approaches to enhance food drying, the use of drying accelerators, in special ethanol, is recently increasing (LLAVATA et al., 2020).

Some works have demonstrated the effectiveness of using ethanol to improve drying processes of food products, considering pre-treatments of immersion, aspersion and atmosphere modification, as well as different methods of drying.

Apples were immersed in ethanol for longs period and subsequently dried by microwave (FUNEBO et al., 2002). The results showed that most part of moisture was removed during pre-treatment. Moreover, the authors observed that ethanol pre-treatment increased rehydration capacity by 4.5 $kg.kg^{-1}$ (control), to 6.5 $kg.kg^{-1}$ (ethanol).

However, ethanol improved drying even shorter treatment periods.

For instance, the immersion of pumpkin cylinders for 15-30 min was studied by Rojas; Augusto (2018b) and Rojas; Silveira; Augusto (2020). Ethanol pre-treatment reduced the convective drying time in ~52%, while the combination of ethanol with ultrasound (for 30 min) reduced 59% the drying time. Moreover, it impacted in reduction of the energy consumption by 44% and promoted the carotenoid preservation in ~100% during drying.

Infrared drying enhancement was also obtained.

Scallion slices were immersed in ethanol at atmospheric pressure and also at vacuum conditions before infrared radiation. Both treatments promoted higher drying and rehydration rates. In addition, the use of ethanol provided greater maintenance of ascorbic acid, the flavor and cell structure of scallion than the control treatment. Different results were obtained by conducting the pre-treatment of ethanol by iimmersion of perforated potatoes before infrared drying (ROJAS; SILVEIRA; AUGUSTO, 2019). Although the pre-treatments increased the drying rate, the use of ethanol slightly decrease the rehydration properties – which was associated with the characteristics of infrared drying.

Different forms of ethanol pre-treatment were also studied.

Green and ripe bananas had their surface treated with ethanol prior to convective drying (CORRÊA et al., 2012), resulting in greater diffusion, smaller drying time and less energy consumption. Mixed rice balls and soy protein powder were

injected with ethanol and dried in a fluidized bed (TATEMOTO et al., 2015), obtaining higher drying rate. Ethanol was also used to modify the atmosphere during pineapple drying (BRAGA et al., 2009). The atmosphere was added of 0.5% v/v ethanol, promoting a rapid vaporization of water and better retaining the volatile compounds after drying.

Initially, the studies associated the efficiency of ethanol in improving drying, with early evaporation during processing. However, the study of Silva; Braga; Santos (2012) introduced the concept of Marangoni Effect in food drying. The Marangoni Effect is the mass transfer mechanism between two fluids with different surface tensions. Therefore, in the water and ethanol mixture, the difference in surface tension can improve the water transport.

Figure 4 shows a schematic diagram of the Marangoni Effect during pre-treatment and drying of vegetable products.

During the pre-treatment by emersion in ethanol before drying, the samples have a surface tension gradient promoted by the low superficial tension of ethanol in relation to water. Therefore, due to both Marangoni Effect and differences on osmotic pressure, both ethanol is incorporated into the food, while part of the product moisture is removed to the ethanol. Ethanol entry into to food, induces water outflow of the intracellular space through cell membranes and cell walls for the intercellular space. Then, the water migrates from the intercellular space to the surface of the sample, when part of the food moisture is removed and transferred to the environment of higher alcohol concentration. Simultaneously, the cell wall compounds (which are solubilized in ethanol) dissolve resulting in the reduction of intercellular spaces, in addition to the intracellular air outlet. Therefore, the cells undergo modification with wilting and shrinking, but maintain their structure.

At the end of the pre-treatment, the intercellular spaces are filled with residual ethanol and water.

Then, during drying, ethanol vaporizes firstly, due to its high vapor pressure, leaving water on the sample surface. Due to water higher surface tension, strongly pulls ethanol+water from inside the sample (that contains more ethanol). This process repeats generating a constant flow until it finds a surface tension equilibrium.

In the next stages of drying, the Marangoni Effect is no longer prevalent; however, the structural changes caused by the use of ethanol can contribute to improve the water transfer and improving the drying process of vegetables.

Therefore, due effective action of ethanol in reducing the drying time, increasing the drying rate and also improving the rehydration capacity, this solvent was used in the present study to provide improvement in the carrot drying and also contribute to the improvement of quality such as rehydration and nutritional content.

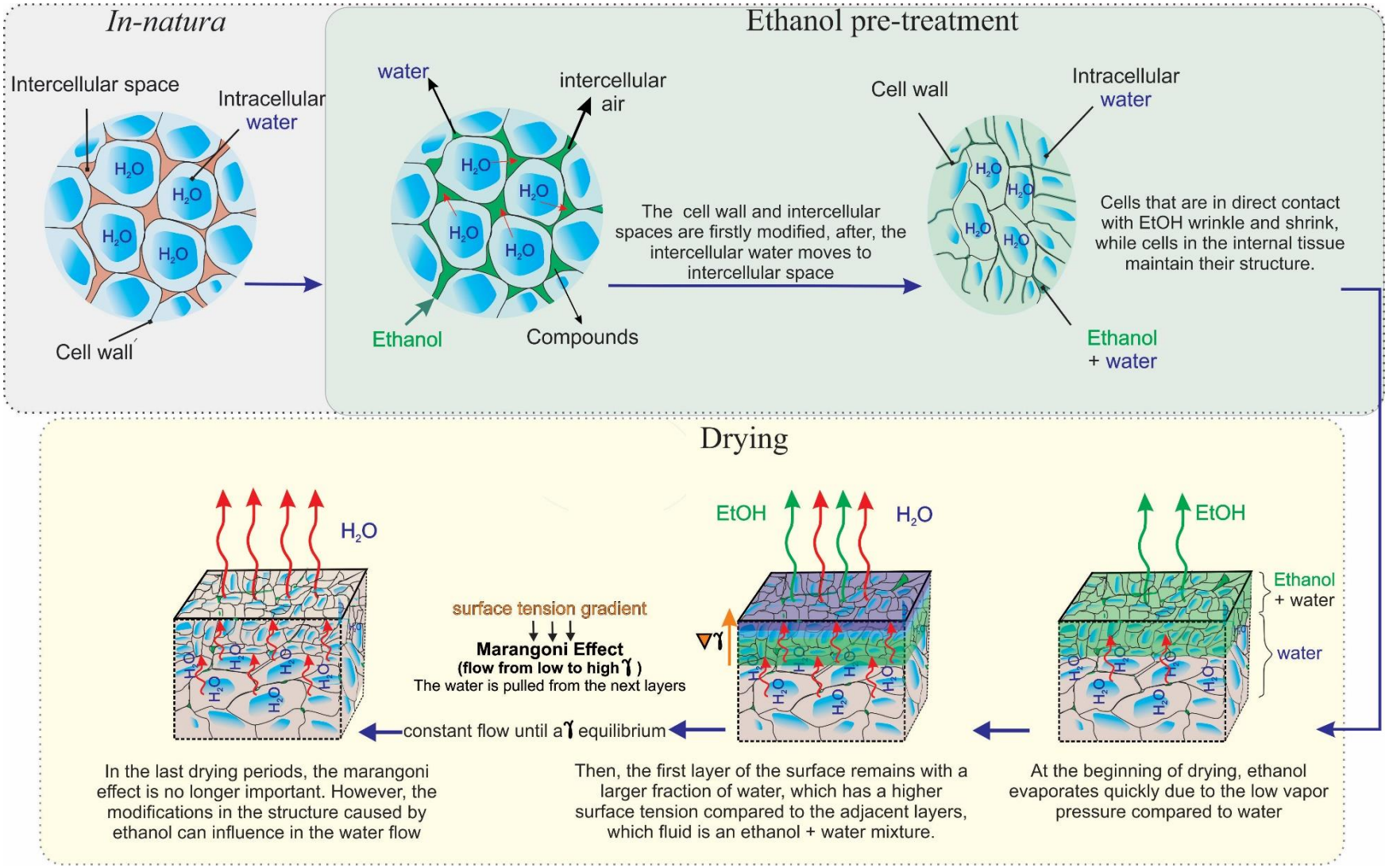

Figure 4: Schematic diagram of the Marangoni Effect during pre-treatment and drying of vegetable products

3.3 Ultrasound and food drying

Ultrasound is an emerging technology based on the propagation of acoustics waves that used frequency above of human hearing (>16 kHz), being divided in two categories: high and low power. The low power ultrasound has intensity of 1 W/cm² and frequency higher that 100 kHz, while high power ultrasound has frequency below 100 kHz with intensities higher that 1W/cm². While low power ultrasound is utilized to get information about physio-chemical properties of food, the high power ultrasound is used to change physical and chemical characteristics of food (SORIA; VILLAMIEL, 2010).

High power ultrasound can be used to promote structural changes in food products, affecting their properties and processes.

All ultrasound systems have three part: the generator, transducer, and delivery system. The generator transforms the electricity in current at the desired ultrasonic frequency and trigger the transducer. The transducer transforms the current in mechanical vibrations. Then the delivery system conveys the vibration to the ultrasonic reactor (MASON, 1998; TAO; SUN, 2015). The acoustic waves need a means of propagation, which can be solid, liquids or gases (MICHAEL; LU; KATHRYN, 2005). Solid foods are commonly processed into a liquid (typically water) in one of the two types devices commonly used: ultrasonic baths and ultrasonic probes. In ultrasonic baths the samples are immersed in liquids that conduct the acoustic waves, and the transducers are placed below the bath vat. In probe systems, the transducer is placed above the probe, which is immersed in the liquid medium and the acoustic energy is transmitted. Although probe systems can concentrate better the energy, their wearing and scale up are negative aspects, limiting their application.

The ultrasound effects can be directly related to the field of ultrasonic application and the device/system, so it is of fundamental importance to calculate the real energy applied in the system (CÁRCEL et al., 2014). When using liquid systems, the estimative of the absolute power (P) delivered to the system can be calculated by the colorimetric methods (RASO et al., 1999), which is described in Eq. (2):

$$P = mCp(\frac{dT}{dt}) \qquad \text{Eq. (2)}$$

Where m is the sample mass, Cp is the specific heat capacity, and $(\frac{dT}{dt})$ is the rate of change of temperature during sonification.

The volumetric power can be obtained (P_V) Eq. (3) by the ratio between absolute power and the volume (v) of liquid used as the wave propagation medium (cm^3 or mL or L) expressed for example in W/cm^3.

$$P_V = \left(\frac{P}{v}\right) \quad \text{Eq. (3)}$$

Therefore, the calculation of the real volumetric power was used as a methodological part of the present work.

Ultrasound can be used for several activities of food industry, among them, the drying operation (CHEMAT; ZILL-E-HUMA; KHAN, 2011).

The improvement of mass transfer by ultrasound can be achieved through two types of mechanisms: directs and indirects.

The direct mechanisms are mass transfer mechanisms, such as the so called inertial flow and sponge effect (MIANO; IBARZ; AUGUSTO, 2016), when sample absorbs the fluid it is immersed. Consequently, the ultrasound direct mechanisms induced structural and compositional changes, which can impact the further drying processing.

The indirect mechanisms are the structural changes induced by ultrasound, which are associated with the rupture of tissues and cells due to acoustic cavitation, resulting in the formation of microchannels (HUANG et al., 2019; MAGALHÃES et al., 2017). The opening of microchannels can improve mass transfer (MIANO; ROJAS; AUGUSTO, 2019), such as the following drying after the pre-treatments with ultrasound. However, acoustic cavitation can also result in the formation of isolated channels without connection with each other and with an external medium, as well as channels with different tortuosity and permeability, which can affect the improvement in mass transfer.

Some works reported the use ultrasound as pre-treatment of food drying (RODRÍGUEZ et al., 2018). Studies with different vegetable products report decreasing of drying time, with different evaluations in relation to other aspects.

Ultrasound was applied prior to drying of potato cylinders with an ultrasonic bath of 91 W/L and 25 kHz up to 120 min (MIANO; ROJAS; AUGUSTO, 2019). The results showed the mass transfer was improved by the ultrasonic pre-treatment and drying was accelerated. Different mechanisms for the observed results were discussed.

Pineapples were treated with ultrasonic bath of 23.2 W/L and 25 kHz up to 30 min before drying (RODRÍGUEZ et al., 2017). In all the sonification times studied, a reduction in the drying time was observed. In addition, this pre-treatment better retained vitamins B_1, B_2, B_3 and B_5, flavonoids and malic acid.

Melons were pre-treated with ultrasound with a power of 4870 W / m^2 and a frequency of 25 kHz up to 30 min (DIAS DA SILVA et al., 2016). Compared to Control the treatment with ultrasound promoted faster drying rates. However, ultrasound was not able to minimize the degradation of carotenoids caused by drying at 60 °C.

Mushrooms were treated with 480 W ultrasound and frequency of 35 kHz for 30 min (ÇAKMAK et al., 2016). The application of ultrasound prior to drying provided a ~ 32% reduction in drying time also preserving the content of phenolic compounds.

Apples were submitted to ultrasound with power of 3 and 4 W / cm^2 with a frequency of 21 and 35 kHz respectively, for 30 min in an ultrasound bath (FIJALKOWSKA et al., 2016). The results showed that sonication reduced the drying time by 13–17% compared to the untreated sample.

Moreover, the ultrasound technology can be used to maintain or improve the nutritional properties of dry foods.

Several studies demonstrated that ultrasound supposedly promote an increase the nutrition content after the they application, such as ascorbic acid in lemon, tomato and strawberry juices (BHAT et al., 2011; GAO et al., 2019; WANG et al., 2019a); carotenoids in tomato juice (GAO et al., 2019b); total flavonoids, catechin, gallic acid in kiwi juice (WANG; VANGA; RAGHAVAN, 2019); chlorogenic acid, catechin, epicatechin and caffeic acid in apple juice (ABID et al., 2014). However, the bioactive molecules cannot be synthesized during sonification. The ultrasonic process can be cause disruption of juice cells by cavitation (ROJAS et al., 2016), releasing components and increasing their extraction (AGUILAR et al., 2017). For this reason, the cited results can be related to a better extraction and/or reactivity of substances during the assay.

Even so, it is interesting to observe ultrasound can be useful in increase the nutritional properties of dry foods, such as promoting the incorporation of microencapsulated nutrients (ROJAS; ALVIM; AUGUSTO, 2019).

Recently, the combination of technologies started to be studied.

Ultrasound associated with a drying accelerator, such as ethanol can provide even more benefits during processing. In fact, the total carotenoid content in pumpkins was preserved by combining ethanol and ultrasound, while the control treatment presented degradation (ROJAS; SILVEIRA; AUGUSTO, 2020). Moreover, this combination also resulted in reduction of 44% the energy consumption.

Therefore, ultrasound technology as a pre-treatment prior to drying has been investigated and has proven to be a good source of optimization of the drying operation. However, many aspects are still unknown, in special the combination with other technology, the associated structural modifications and how different tissues are affected by that pre-treatments.

Therefore, the application of ethanol with ultrasound was proposed in the present study prior to convective drying of carrot slices, evaluating different parameters of processing and product quality.

3.4 Microstructure of carrot

Carrots were chosen as the raw material for this study because this vegetable is widely consumed worldwide, is a good source of bioactive compounds and it can be present in several preparations and commercial forms, including raw and processed. Further, dried carrots can be both consumed directly, as a healthy snack, or it can be rehydrated in different dishes. In addition, it is a raw material that can provide an important study and differentiating it from the mechanisms of mass transfer due to the existence of natural channels that can contribute to improving the output and removing moisture during drying.

Therefore, it is important to develop studies to better describe the action of physical and chemical agents as a pre-treatment of carrot drying.

Figure 5 presents an illustrative scheme of the organization of the cellular tissues of the root of dicot plant, such carrot.

In Figure 5 its possible observed that the dicot plant has roots composed of cortex that serve as deposits. The next layer is the endodermis that can control the movement of water and dissolved mineral ions. In the endodermis it is possible to find the vascular cylinder, which has a pericycle (which contributes to the entry of nutrients in the xylem), phloem and xylem. The xylem is at the centre of the root of a dicot and has the shape of a star with a variable number of points (seen through the cross section). Among the points are the phloem bundles (DAVID SADAVA et al., 2012).

The carrot microstructure is composed by three main tissues: xylem, phloem and parenchyma.

Parenchyma cells are present both in the cortex and core. In the cortex, the parenchyma is made up of smaller, elongated cells while the core is made up of larger parenchyma cells. The vascular tissues (xylem and phloem) are located in the core and help to transport water and nutrients (Figure 6).

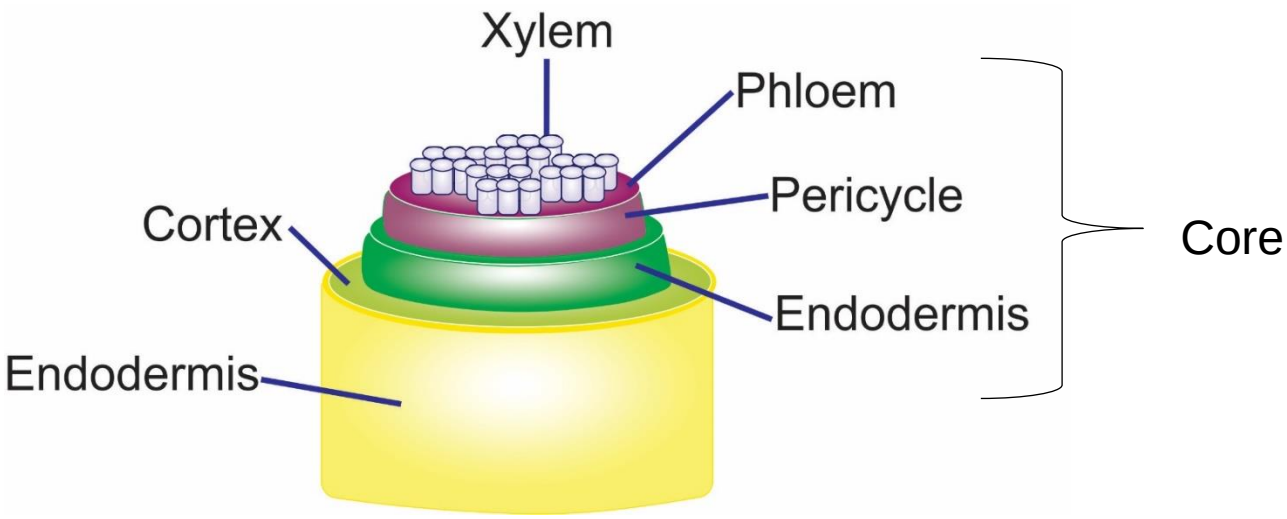

Figure 5: Diagram illustrating the tissue layers and their organization in dicot roots (DAVID SADAVA et al., 2012).

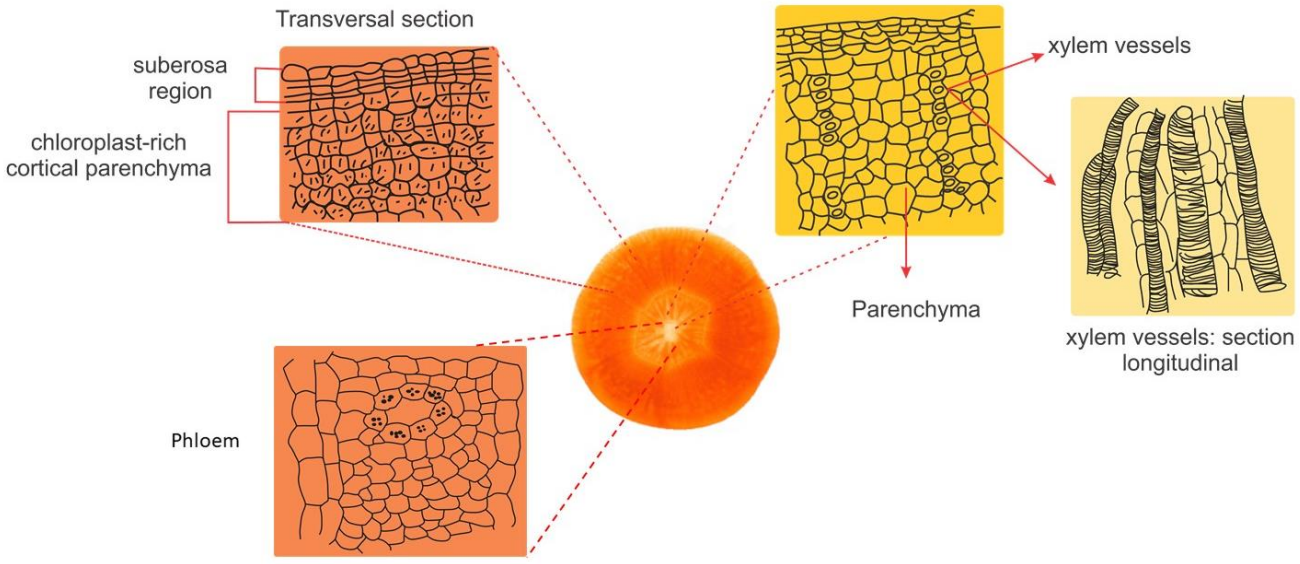

Figure 6: Location of the main structures of carrot cell tissues

The matrix constitution of the carrot root demonstrates the histological differences of this vegetable. Therefore, each region can respond differently during the pre-treatment, and for this reason, in this work it is proposed and investigated the influence of pre-treatments with ethanol and ultrasound in the different regions of the carrot (Cortex and Core), observing the microscopically structural changes in the parenchyma, xylem and phloem. In addition, investigation of the impact of treatments on texture also in each region.

4. MATERIAL and METHODS

The experimental flowchart is described in Figure 7. This work described ethanol and/or ultrasound pre-treatments to carrot convective drying and their influence on drying and rehydration kinetics, microstructure, texture, shrinkage and carotenoid content. Carrot was chosen by being a widely consumed vegetable, with good nutritional value (in special related with its carotenoid content), and the product can be consumed directly dried or rehydrated (such as in soups). Moreover, it is constituted by two different fibrous parts (demonstrated on section 3.4), which make its study interesting from a structural point of view: the core, which is the internal and softer pulp; and the cortex, which is the external and harder pulp.

4.1 Sample preparation

Carrots (*Daucus carota*) free of injuries and homogeneous in colour were obtained in a local market of Piracicaba-SP, being stored at ~6 °C before processing. The carrots were cut in 5 mm slices (cutter Britânia, Brazil), totalling 32 slices per treatment. The slices were then submitted to different treatments.

4.2 Pre-treatments

In order to better understand the effect of ethanol and ultrasound on carrots, the following pre-treatments were applied: Ethanol (E); Ethanol+Ultrasound (Ethanol+US); Water+Ultrasound (Water+US); and Control. Pre-treatment Ethanol evaluate only the effect of ethanol. It is impossible to isolate the pure ultrasound effect since a fluid must transmit the waves through a liquid until the solid product in the ultrasonic bath. Therefore, water was used in the treatment Water+US to closely evaluate the ultrasound effect. The effect of the two technologies combination was observed in the Ethanol+US pre-treatment. The Control is a sample that did not undergo any pre-treatment before drying, being only cut to standard size and dried under the same conditions as the other treatments.

The carrot slices were submerged in ethanol (99.8 % v/v) for 30 min (Ethanol). The ethanol and ultrasound action time was chosen based on the work Rojas et al. (2020) that obtained highest drying time reduction with combination Ethanol+US for 30 min. After the immersion time, the samples were removed from ethanol, which was drained, and the samples were superficially dried with absorbent paper.

Ultrasound was applied in an ultrasonic bath (Q13/25, Ultronique, Brazil; frequency of 25 kHz) at 20 ºC, using ethanol (99.8% v/v) or water, for 30 min. To certify the maintenance of the temperature during the ultrasonic pre-treatment (20 °C), an auxiliary thermostatic water bath (ColdLab CL 16–40 - Brazil) and a heat exchanger recirculating a cold solution of ethanol/water were used. Moreover, the good practices described by Vinatoru (VINATORU, 2015) were applied. The actual delivered volumetric power was 23.9 and 25.7 W/L (calculated through the calorimetric method according to (RASO et al., 1999)) for water and ethanol, respectively. After ultrasound processing, the samples were removed from ethanol or water, and their surfaces were drained and superficially dried with absorbent paper.

4.3 Structural analysis

Microstructural evaluation was carried out using the optical L1000 microscopy model (Bioval, Curitiba, Brazil) with 20 W halogen lamp and a portable camera of 5 megapixels. The carrots slices were cut into 20 µm dishes using a handheld microtome (Ancap, São Paulo, Brazil) and observed with 10-fold magnification lens. For better observing, a blue dye of 0.1% toluidine was used. From then, the microstructure was verified on the *in natura* sample and pre-treated samples. The images were captured after securing a representative field. The imagens were captured in core and cortex of carrot.

4.4 Texture Analysis

Ethanol and ultrasound may act differently on the external and core regions of carrot due to different composition and structure of tissues and cells, which are distributed along carrot length. Therefore, this work analysed the effects of pre-treatments at macrostructural level through texture analysis. For this, it was considered both the core and external regions in order to understand and describe the mechanism in materials with different structures such as carrots.

Texture analysis was performed in the *in natura* carrot slices and those after the pre-treatments. Texture was assessed by a penetration test using a texture analyser (TA.XT Plus, Stable Micro Systems Ltd., Surrey, UK) with a 50 kgf load cell (490.3 N). A cylindrical probe of 2 mm diameter was used to penetrate the samples thickness at a constant rate of 1 mm/s until the distance of 3 mm. The curve force (N) versus

penetration (mm), was used to describe the texture. The analysis was performed with five carrot slices for each treatment, considering both the core and external regions. Each slice was perforated 4 times in the core and 4 times in the cortex. Illustrative scheme of the analysis is described in Figure 7.

4.5 Drying

Convective drying was conducted in a tray dryer (UOP8MKII, Armfield®, England) at 40 °C with air velocity of 1 m/s. This temperature was selected in order to minimize carotenoid degradation, once a decrease of 52.5 and 57.8% was observed in the carotenoid content of dried carrots at 50 and 70 °C respectively (MD SALEH et al., 2020). The carrot slices were placed on perforated metal trays, allowing the hot air flows through all their surfaces. Samples remained in the dryer until constant weight. The sample mass was recorded continuously through the UOP8-MKII-306 software (Armfield®), without the need to withdrawal of samples.

The moisture at each time was obtained by the mass balance, considering the initial (of *in natura s*amples) and final (after drying) moistures, which were obtained after completely drying crushed carrots at 105ºC with the aid of moisture analyser (MX-50, A & D Company, Tokyo, Japan). It is important to highlight the sample “moisture” is a lumped parameter that includes both volatile liquids: the remaining water and the absorbed ethanol (ROJAS; AUGUSTO, 2018b).

The drying curves were elaborated according to the dimensionless moisture (MR) during the processing time, calculated according to Eq. 4 where M_t is the moisture (% d.b.) content during the drying process time (t), M_e (% d.b.) is the equilibrium moisture and M_p (% d.b.) is the carrot moisture after pre-treatment. In the case of the Control sample, M_p (% d.b.) is equal to the *in natura* carrots moisture.

$$MR(t) = \frac{M_t - M_e}{M_p - M_e} \qquad \text{Eq. (4)}$$

Drying kinetics was evaluated using the Page Model (Eq. 5), where k (h^{-1}) is the drying rate parameter and n is the dimensionless shape parameter. According to Simpson et al., (2017) interpretation of Page Model, the parameter k is associated with the “diffusion coefficient” and sample geometry, while the parameter n describes the “diffusion type”: $n>1$ is related with super diffusion, while $n<1$ is related with sub diffusion. When $n \neq 1$, mechanisms other than diffusion are important; for example, the

“super diffusion process” (*n*> 1) may indicate the importance of capillarity (ROJAS; AUGUSTO, 2018b).

$$MR(t) = \exp(-kt^n) \quad \text{Eq. (5)}$$

4.6 Estimative of the total energy consumptions

An estimative of the total energy consumption (TEC) (Eq. 6) during processing (including pre-treatment and drying) was calculated according to Motevali et al.(2011); Onwude et al. (2019) and Rojas et al. (2020). It is important to mention this approach does not consider the strict energy consumption during processing, being an estimative of the energy consumption by considering the performed pre-treatments. Even so, it is useful for comparison purposes. The total energy consumption (TEC, Eq. 6) was calculated considering 1 kg of *in natura* sample (m) and the two terms: E_{PT} represents the energy consumption during pre-treatments (Eq. 7), and E_D represents the energy consumption during convective drying (Eq. 8).

$$TEC = \frac{E_{PT}+E_D}{m} \quad \text{Eq. (6)}$$

$$E_{PT} = W.V.t_P \quad \text{Eq. (7)}$$

Where, W is the US actual volumetric power (W/L - determined by the calorimetric method depending on whether water or ethanol was used), V is the volume of water or ethanol (L) used in the US bath, t_P is the time of pre-treatment.

$$E_D = A.v.\rho_a.Cp_a.\Delta T.t_D \quad \text{Eq. (8)}$$

Where A is the cross-sectional area of drying (m^2), ρ is the ambient air density (25 °C), Cp is the specific heat capacity (J/kg·K) of ambient air, ΔT is the temperature difference between the ambient air and drying air, v is the air velocity (m/s), and t_D is the drying time needed to the samples reach a moisture 20%$_{w.b.}$ For the calculations, the initial sample mass, the histories of temperature and air velocity were considered for each treatment and drying process replicate.

4.7 Shrinkage

The shrinkage of carrot slices was measured after pre-treatments and also after drying. Carrot slices radial area was used as the shrinkage evaluation parameter. For

this, carrot slices with standard dimensions (5 mm thick and 3.5 cm diameter) were used.

The *in natura* samples and those after pre-treatments were circular in shape; so, the area formula of a circle was used to calculate their area. The diameter was verified with the aid of a digital calliper.

After drying, the samples lose their circular shape, so it is not possible anymore to calculate the area by considering a circular shape. Therefore, the projected areas were measured by image analysis. The dried samples were placed on a dark (black) surface near to a scale reference. Their images were captured at the same distance and then processed in ImageJ version 1.52a (National Institutes of Health, USA) software. The accurate photos have been converted to grayscale (8 bits). With the help of the command "set scale" and a ruler close to the samples, the scale was defined. Then the images were converted to binary scale using the "threshold" command. For the results, we used the command "Analyse", which provides a response window with the area of the calculated samples. For the purpose of visualization and better understanding the results, the deformation was expressed as shrinkage ratio (%).

Shrinkage assessment was divided into three ratios, in order to evaluate the deformation during pre-treatment, during drying and also the whole process treatments: (1) Ratio between the area after pre-treatments (before drying) and the initial area (of *in natura* sample); (2) Ratio between final area (after drying) and the area after pre-treatments (before drying); and (3) Ratio between final area (after drying) and the initial area (of *in natura* sample), considering each treatment.

4.8 Rehydration kinetics

The rehydration process was conducted at 25 ± 1 °C (water bath MA 095 / CFRE, Marconi). The dried carrot slices were submerged in distilled water (4 g of dried product with 20%$_{w.b}$ moisture was used with 1 L of distilled water). The sample moisture over the rehydration time was calculated by mass balance, considering the carrot initial moisture obtained with the moisture analyser, as described before.

For this, the samples were taken from the water, drained and dried superficially, weighed, and then returned to the water again. This step was performed every 5 min for the first 25 min, then every 10 min until 130 minutes, and then every 30 min until reach constant mass.

The rehydration data was fitted using the Peleg Model (Eq. 9) (Peleg, 1979), where *M(t)* is moisture content in dry basis (d.b., g water/g dry matter) at time *t* (min), M_0 is initial moisture content (d.b.), and k_1 (min·d.b^{-1}) and k_2 (d.b^{-1}) are parameters related with the water absorption rate and quantity: the reciprocal of k_1 represents the maximum water absorption rate (at the beginning of rehydration), and the reciprocal of k_2 is associated with the water retention capacity.

$$M(t) = M_0 + \frac{t}{k_1 + k_2.t} \qquad \text{Eq. (9)}$$

4.9 Total carotenoid content

Prolonged exposure to hot air may cause nutrient degradation during drying. Therefore, the present work evaluated the carotenoid content over the proposed treatments in comparison with the *in natura* carrot in order to evaluate the nutritional quality. To avoid errors due to lack of homogeneity between samples and also to avoid over-drying effects, all treatments were dried to the final moisture of 20% (in wet basis). This value has been selected as a reference as the maximum moisture value recommended for dehydrated fruits and vegetables (XIAO DONG CHEN; KAMLESH C. PATEL, 2008).

After drying with the different pre-treatments, the samples were rehydrated for 8 hours at 25 °C and the total carotenoid content was measured according to the methodology described by Potosí-Calvache et al. (2017) with modifications.

About 0.25 g of samples was weighed and transferred to aluminium-coated glass tubes in order to protect the samples from light and oxygen. Then, 21 mL of ethanol:hexane (4:3) solution was added (ethanol 99.8% from Scientific Exodus, São Paulo, Brazil and hexane 98.5% from Labsynth, São Paulo, Brazil). Samples were triturated with the solution for 1 min using a rotor-stator homogenizer (Superohm, São Paulo, Brazil) and the probe was washed with an additional 21 mL of ethanol:hexane solution, which was reserved. The tube with sample and solvent was then stirred in a thermal bath at 250 rpm and 25 °C for 30 minutes (DUBNOFF MA 095 / CFRE, Marconi, Brazil). Then, the solvent was separate from the sample and transferred to other vessel protected from light and oxygen. After this, the decanted residue was mixed with the solution used for washing the homogenizer probe in a tube that was stirred for 30 min at the same conditions. Subsequently, the solvent was removed from the residue and added to the vessel containing the solvent from the first extraction. 5

mL of distilled water was added to this vessel, manually stirred and allowed to stand for 5 minutes to separate the phases (aqueous phase and hexanoic phase). The volume of the hexanoic phase was noted for subsequent calculation of carotenoid content.

After obtaining the phase of interest (the hexanoic phase), its absorbance was read at 450 nm (FEMTO 600S, São Paulo, Brazil) using hexane for calibration. The carotenoid content of the extracts was calculated by Eq. 10 and expressed as *β*-carotene equivalents (µg)/ g of sample. Where 536.85 g/mol is the molecular weight of *β*-carotene, *V* is the volume (mL) of the hexanoic phase, *m* (g) is the mass of the used sample, and 137.4 mM^{-1} is the extinction coefficient for *β*-carotene in hexane.

$$\left[cc\left(\frac{\mu g}{g}\right)\right] = \frac{A_{450} x\ 536{,}85\ x\ V}{m\ x\ 137{,}4} \qquad \text{Eq. (10)}$$

4.10 Experimental Design, Regressions and Statistics

The experiments were performed with three replicates. Results were analysed by analysis of variance (ANOVA) and differences among treatments were determined using the Tukey test at a 5% significance level using Statistica 7® software (Statsoft, USA).

The parameters of each models were iteratively adjusted to the experimental data by minimizing the sum of squared errors (SSE in Eq. 11) between the experimental and the predicted values. A generalized reduced gradient algorithm was used, implemented in the 'Solver' tool of software Excel 2016 (Microsoft, USA). The different initial guesses of the parameters were evaluated to detect possible convergence to local optima.

$$SSE = \sum_{I=1}^{X}((PRETICTED) - (EXPERIMENTAL)_I^2 \qquad \text{Eq. (11)}$$

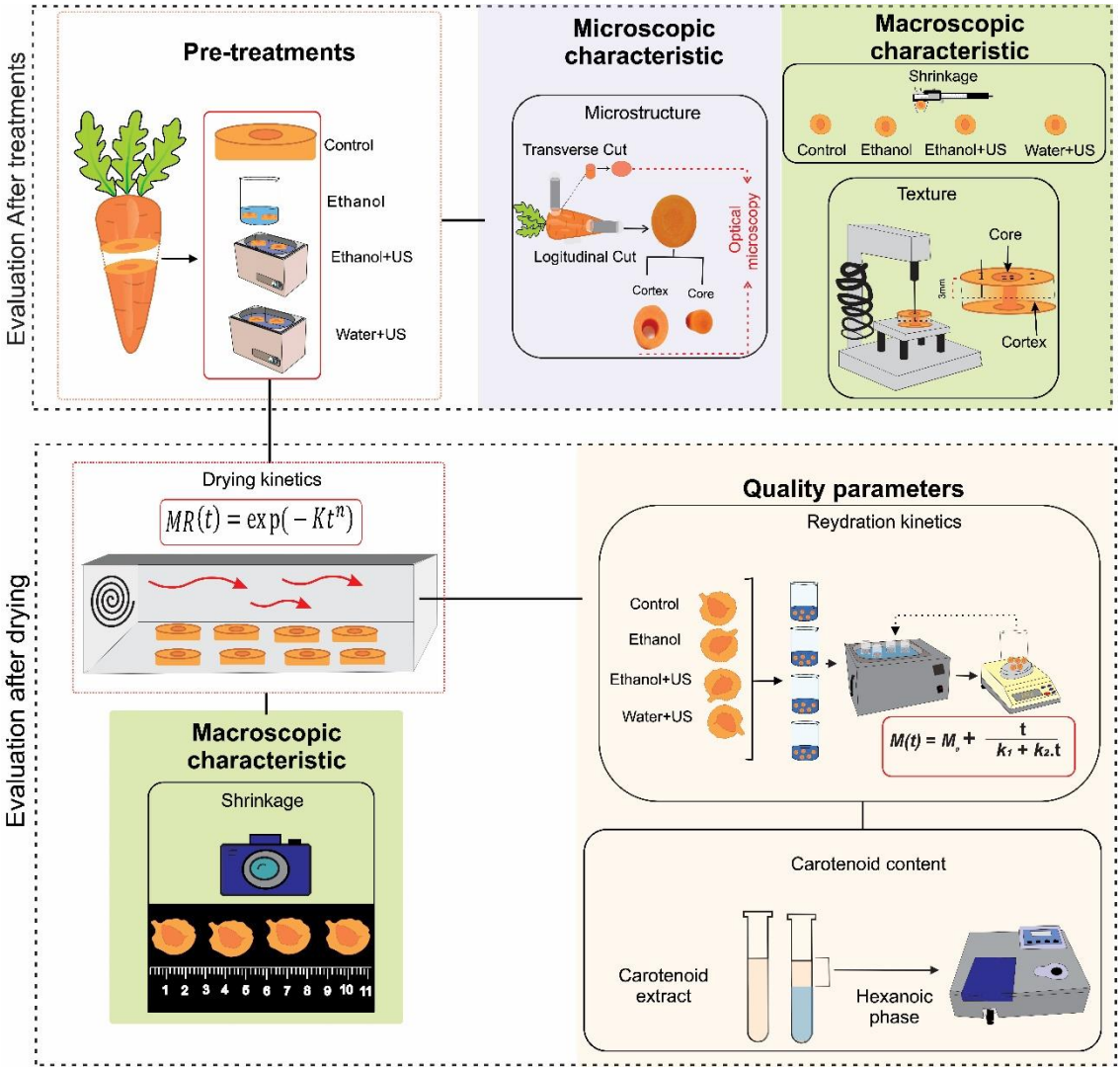

Figure 7: Illustrative diagram of sample preparation and analyses

5. RESULTS AND DISCUSSION

5.1 Effect of treatments on carrot microscopic and macroscopic structure

Figure 8 shows the carrot structure before and after the different pre-treatments. The carrot has three main structures: Parenchyma, Phloem and Xylem. These tissues are distributed in both the core and cortex regions of the vegetable (Section 3.4 shows the description for “core” and “cortex”).

The parenchyma is the predominant cellular tissue in the roots. It is composed by cells of polyhedral shape, being the fundamental filling tissue present throughout the root in which other tissues are found, such as the vascular (xylem, Figure 8, B, and phloem, Figure 8, C) tissue (EVERT RAY FRANKLIN, 2013). However, the structure of the parenchyma tissue can vary even in the same vegetable. In carrots, the parenchyma presented walls rich in elongated chromoplast at the cortex (Figure 8, A), and larger cells in the core close to the phloem (Figure 8, D) and xylem (Figure 8, E).

According to Evert, Ray and Franklin (2013), the xylem is responsible for nutrient transport and storage, and the phloem is the main nutrient conducting tissue. In carrots, Smith and Ho (2007) reported the core region in carrots is mainly composed by vascular tissue, while the cortex region is predominantly parenchymal tissue, which in fact can be seen in Figure 8. This confirms the difference of these two regions. Therefore, the objective of following discussions is to describe and explain the structural changes that occurred with the use of pre-treatments in the tissues presented in both the core and on the cortex of the carrot.

The pre-treatments induced structural changes evidenced in some carrot tissues.

No structural changes were evidenced in the xylem (Figure 8, B) and the phloem vessels (Figure 8, C), in any of the treatments, which can be associated to its more rigid and compact structure. Smith and Ho (2007) described that carrot xylems have a second lignified cell wall, which makes the xylem tracheal elements stronger than the parenchyma. Similar result was reported by Rojas; Augusto (2018a) in pumpkin.

After the treatments with Ethanol and Ethanol+US, the cortical parenchyma showed a slight shrinking of the cell wall when compared to the fresh sample (Control). Although cellular organization was maintained, this change on cell wall can be important to change its permeability – as discussed further. On the other hand, the parenchyma tissue next to the phloem (Figure 8, D) and xylem (Figure 8, E) at the core, obtained higher wrinkle levels of the cell wall, which became thinner and

disorganized, losing their polyhedral shape. The changes in parenchyma cells can be associated to the loss of water, air and other compounds during the treatments with ethanol, altering the cells structure. This effect was also observed by (ROJAS; AUGUSTO, 2018b) in pumpkin parenchyma.

Canteri et al. (2019) studied the composition of the cell wall of different vegetables, including carrots. They demonstrated that ethanol could extract polyphenols, some proteins and lipids from the cell wall and/or membrane. However, ethanol was not able to extract cellulose, lignin, pectin or hemicellulose from the cell wall. Consequently, this can help to explain the results here obtained. Once the main structural components of the cell wall are not extracted, but some components of cell membrane and cell wall can be, the general structure of the cells is maintained but reducing the thickness. It may have contributed to the improvement of mass transfer (which can be seen in Figure 12). However, although ethanol probably changes the cell wall and membrane composition, the measurable loss of solids in carrots were negligible.

Unlike the observed in the Ethanol and Ethanol+US treatments, the Water+US treatment resulted in parenchyma cells swelling, at both the core and cortex, due to water inlet (Figure 8, A). Xylem and phloem vessels can assist the transport and migration of water into the cells. These structures are responsible for transporting water and nutrients in the living carrot. However, Rojas; Augusto (2018a) demonstrated that water can be transported by capillarity in pumpkin xylem vessels during both drying and rehydration (https://youtu.be/o5vbxs1G81s). According to the authors, the water passes through the xylem vessels, from which begins to be distributed firstly through intercellular spaces, then diffuses through the walls and membranes into the cells.

Another factor associated with water migration into cells is the use of ultrasound, which improves mass transfer (WANG et al., 2018) due to direct and indirect effects (MIANO; IBARZ; AUGUSTO, 2016).

The "sponge effect" is produced by mechanical waves passing through the product, and it helps to keep the microchannels unobstructed and favours the migration of water into the solid (GAMBOA-SANTOS et al., 2014; RICCE et al., 2016; ROJAS; AUGUSTO, 2018b; TAO et al., 2019; WANG et al., 2018).

Moreover, it is widely described (but rarely demonstrated) in the literature that ultrasound treatments form new micro-channels in the product due to acoustic cavitation. However, this phenomenon could not be evidenced in carrots during pre-

treatments with Ethanol+US and Water+US (Figure 8), which can also explain the low influence of ultrasound treatments on drying time reduction (as it will be discussed on Section 5.2). The carrot has a stiffer or compact structure in comparison with other vegetables (such as potato (MIANO; ROJAS; AUGUSTO, 2019), and melon, (FERNANDES; GALLÃO; RODRIGUES, 2008) for example), which may have contributed to little or no visible microchannel formation in the evaluated conditions. Moreover, the pronounced formation of microchannels may be associated with the level of power employed. Wang et al. (2018) evaluated the use of ultrasound in a probe system as a pre-treatment of carrots and found that increasing the nominal power from 1800 to 3600 W/L resulted in greater effect on the carrot structure, with formation of microchannels. However, it is difficult to compare our results once only the nominal power was reported by Wang et al. (2018) (and the difference between the nominal and actual delivered power can be in the order of 90-95% - (CARVALHO et al., 2018). Moreover, although interesting and valuable approach, the ultrasonic probe system has important drawbacks from an industrial point of view, such as high cost and wearing, and it is not suitable for scale production. Therefore, further studies are still needed to understand the effect of ultrasound conditions on structure of different vegetables.

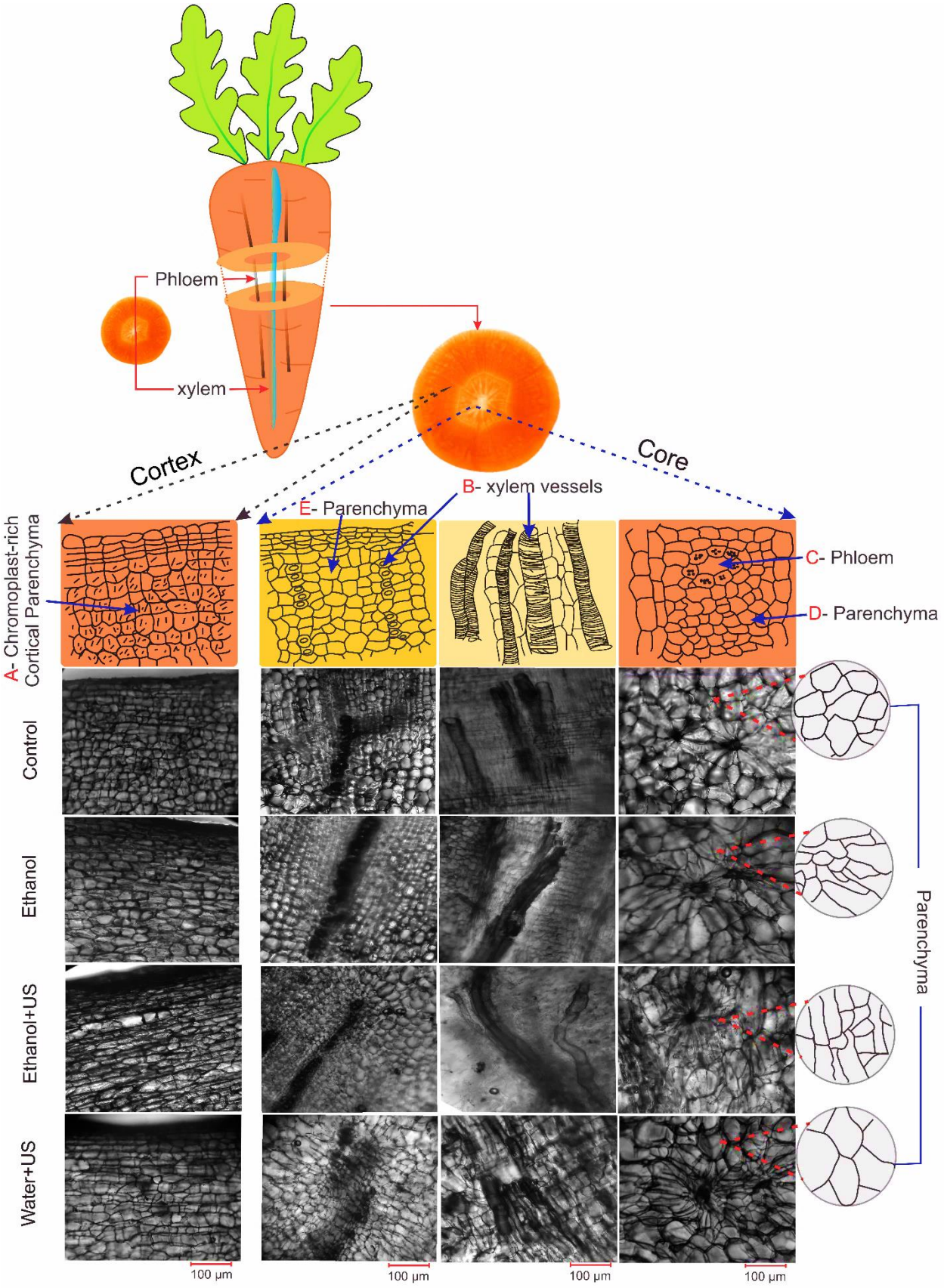

Figure 8: Carrot microscopic structures: core and cortex of the Control, Ethanol, Ethanol+US and Water+US pre-treatments.

Structural differences and modifications can be manifested on macroscopic changes. Consequently, texture was evaluated to complement the discussion of structural changes. Figure 9 shows the texture, through a puncture assay, of carrot core and cortex after the different pre-treatments. Despite the high standard deviation range in all conditions, a qualitative discussion can be carried out.

In general, the graphics demonstrates the differences between the core and cortex regions of carrot. When compared to the cortex, the core region is less resistant, evidenced by the lowest time and force necessary to penetration for all conditions, except for – the Water+US treatment. The core region contains larger parenchymal cells (see Figure 8, D) while the outer part of the cortex has smaller parenchymal cells (see Figure 8, A) with a large amount of plastids containing carotenoids, which can explain the observed differences. It confirms the description of Zdunek e Umeda (2005), which demonstrated that less energy is needed to fracture a tissue made up of larger cells.

In the texture curves, there was an increase in force as the probe exerts pressure on the cortex. At this stage, the cortex is deformed according to the applied force, but there is no perforation. This phase ends when the probe penetrates the first layers of tissue causing an irreversibly rupture. The rupture occurs after ~1.25 mm for the Control and Water+US treatments, and ~2.8 mm for the treatments with ethanol (Ethanol and Ethanol+US). After rupture, the force exerted to maintain penetration in the cortex is approximately constant, which demonstrates the same profile of the layers of tissue along its thickness. On the other hand, the behaviour in the pre-treatments with Ethanol and Ethanol+US indicates the first layers of tissue are more resistant than the adjacent layers, that is why the applied force increases until the rupture. The greater resistance of the first layers of tissue is caused by the superficial dehydration of the samples when using ethanol, forming a more resistant tissue external layer. This can be confirmed in the work of (ROJAS; AUGUSTO, 2018b) where they demonstrated that ethanol only reach the initial layers of tissue.

Therefore, in the cortex, the use of ultrasound had not changed the texture profile (since the treatment Water+US is similar to the Control, and the Ethanol+US is similar to the Ethanol), and the effect of ethanol on the carrot structure was higher than those of ultrasound.

Similar trend and behaviour were observed in the core. However, unlike the cortex, the Water+US treatment presented a more rigid structure than the Control. This may be associated to the migration of water to the cell during this pre-treatment, as demonstrated on Figure 8, D, the consequent increase in cell turgor contribute to a greater resistance of the tissue to perforation. The predominant absorption of water by the core can be associated with the presence of phloem and xylem vessels, which help the transport and migration of water into the cell. After the initial deformation, the core tissue broke at ~ 0.7 mm for Water+US treatment, ~0.9 mm for the Control and and ~ 2 mm for Ethanol and Ethanol+US. After the rupture, the force exerted to maintain penetration into the core is approximately constant for Water+US similar to the Control, being slightly decreased in Ethanol and Ethanol+US – indicating dehydration and compactness in the surface layer of the samples that include ethanol during the pre-treatment.

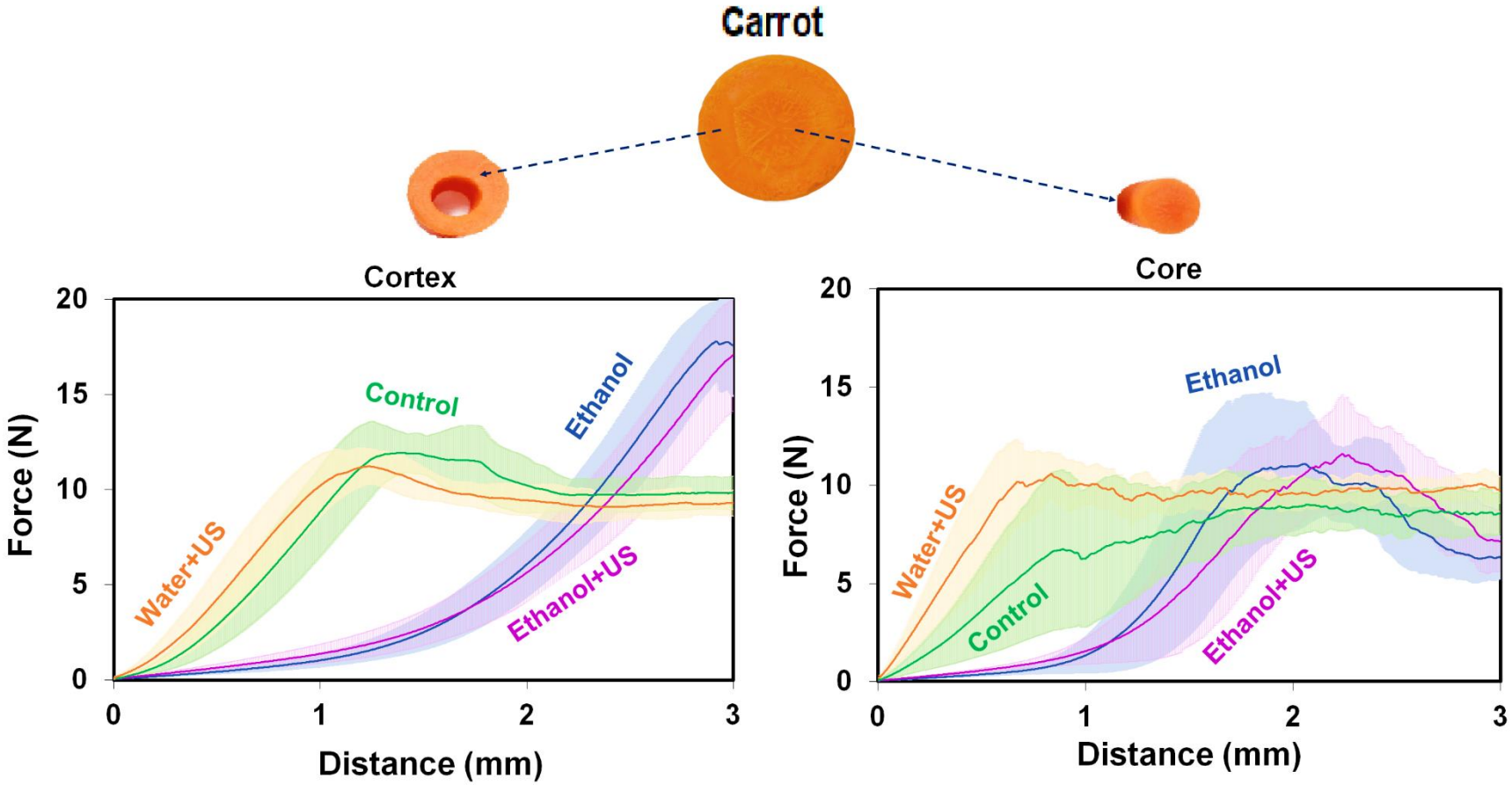

Figure 9: Resistance to puncture (Force (N) x Distance (mm)) of each carrot region (cortex and core) after pre-treatments. The shaded area in each treatment indicates the standard deviation.

Shrinkage was also investigated since it is another macroscopic characteristic which is correlated with microscopic structural modifications. Figure 10 shows the visual appearance of carrot slices under different conditions: fresh and pre-treated (with ethanol and/or ultrasound) carrots before and after drying.

The shrinkage percentage of carrot slices are shown in Figure 11, considering the changes due to only pre-treatment (Figure 11A), only drying (Figure 11B), and the whole process (Figure 11C).

Figure 11A corresponds to the ratio between the area after pre-treatments (before drying) and the initial area (from the *in natura* sample). This allows us to visualize the radial shrinkage behaviour of carrot slices during pre-treatments. All treatments showed different shrinkage rates when compared to each other ($p < 0.05$). At this stage, the Ethanol treatment had about 12 % shrinkage, already the Ethanol+US stands out with the greatest shrinkage, about 22%, this shrinkage levels could be explained because water and air has already been removed during the pre-treatment. On the other hand, the Water+US had a small (4%) increase in its area due to water absorption (which was also evidenced in microscopy and texture analyses).

Figure 11B corresponds to the ratio between the area after drying and the area after pre-treatments, which indicates the behaviour of the carrot slices during drying. It is possible to verify that the treatment with Ethanol+US presents a lower shrinkage (70%) during drying, when compared to the Control (78%) and other pre-treatments (75 and 77% were obtained for the treatment E and W + US, respectively). This is consistent because the Ethanol+US samples started drying already with a higher shrinkage level than the Control (described in Figure 11A). In the case of Water+US treatment, despite the water gain during treatment (Figure 8 and Figure 11A), it did not promote different shrinkage level compared to Control during drying.

Figure 11C corresponds to the ratio between the final drying area and the initial area (fresh sample), thus describing the shrinkage of the final product. Although the treatments have different drying rates (Figure 12, which will be explained later), this did not influence the shrinkage of the slices after drying. All presented equal shrinkage ratio ($p > 0.05$). This may have occurred due to the low temperature applied (40 °C), once at low temperatures, moisture is transported in a flat pattern with minimal stresses within the food.

After drying, the carrot's cortex has shrunk towards the core, for all treatments, with pronounced deformation in the form of undulations (similar to a flower – Figure

10). This behaviour was also observed in potato (Aprajeeta et al. (2015)).They observed that during drying, moisture leaves the potato pores filled with water on the surface of the material, and this disturbs the mechanical balance of the cell walls (this occurs linearly over time). According to the authors, this effect causes shrinkage towards the core of the sample due to the contraction effect, which is the stress caused by the presence of spaces on the outer wall of the cell, while the inner wall remains the same. Possibly this mechanism also occurs in carrots. Another option is that the major quantity of vascular tissue in the core region provided more resistance to deformation.

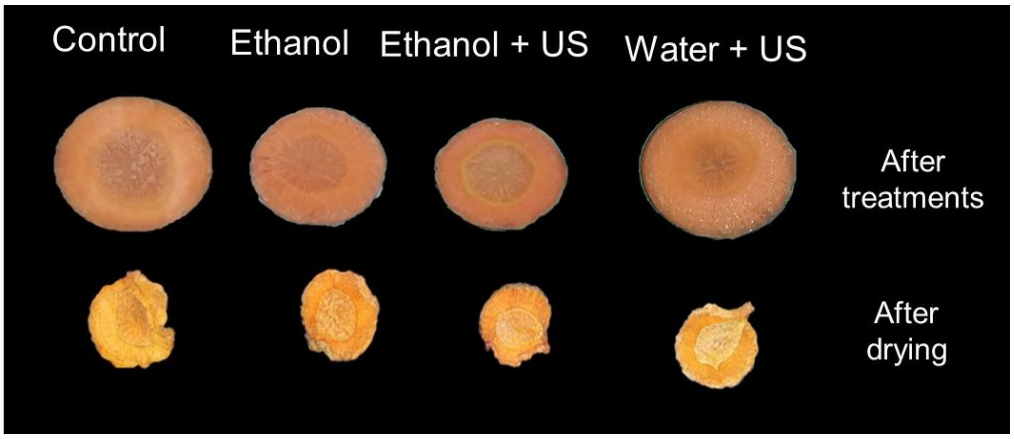

Figure 10: Shrinkage of pre-treated and Control carrot slices, after pre-treatment and drying.

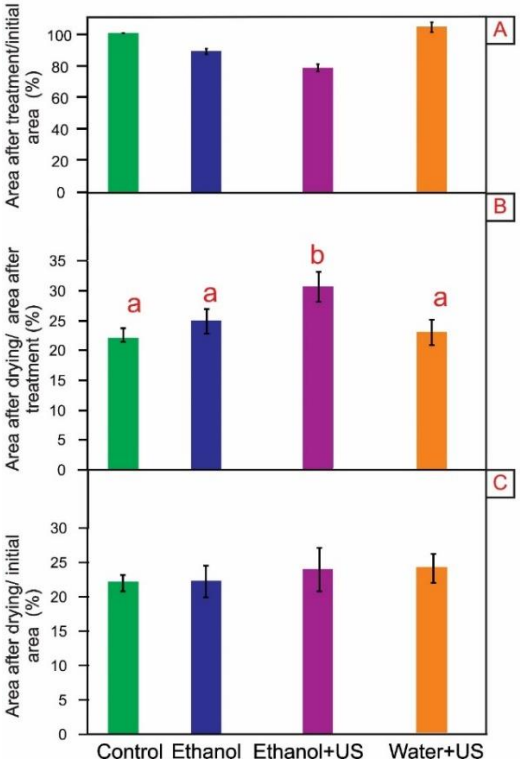

Figure 11: Shrinkage Ratio: A- Ratio of the final pre-treatment area to the initial Control area. All treatments were different from each other at 5% significance (Tukey). B- Ratio of final area of drying to initial area of treatments. Equal letters indicate similarity at the 5% significance level (Tukey). C- Ratio of final drying area to initial Control area. All treatments were equal at 5% significance level (Tukey).

5.2 Effect of ethanol and ultrasound pre-treatments on drying kinetics

The results of drying kinetics are presented in Figure 12, with the data adjusted to the Page Model (Equation (1); (5)). The inserted graphic in Figure 12 shows the "drying time", which was considered the time required to obtain different moisture levels (from 60 to 20%$_{w.b.}$): therefore, the graphic allows to visualize the behaviour of the carrot slices during different drying phases.

In general, the Ethanol and Ethanol+US pre-treatments resulted in faster drying when compared to the Water+US treatment and the Control. Note the similarity in drying behaviour between Ethanol and Ethanol+US treatments, and between Water+US and Control: therefore, ethanol had a higher contribution in decreasing the drying time than ultrasound.

The Water+US pre-treatment resulted in drying time statistically equal to the Control at 60% moisture ($p > 0.05$). However, in the moisture range of 50 to 20%, this

treatment dried faster than the Control. In fact, the time needed to finish drying (which we consider the final product moisture of 20% in wet basis - (XIAO DONG CHEN; KAMLESH C. PATEL, 2008)) in the Water+US treatment was 33% smaller than the Control. On the other hand, the treatment Ethanol and Ethanol+US required less drying time than Control and Water+US treatments. Considering the final moisture of 20% as target, the drying time required was 51 and 50% less for the Ethanol and Ethanol+US treatments, respectively, than the required by Control. This result is similar to the reported for pumpkin cylinders (ROJAS; SILVEIRA; AUGUSTO, 2020).

Although the Water+US treatment has a positive contribution to drying process (reduces 33% of the drying time and does not differ from the Ethanol and Ethanol+US treatments ($p > 0.05$)), it was not possible to highlight drastically improving the process with the use of ultrasound technology, due to the small contribution of ultrasound in decreasing the drying time in the Ethanol+US treatment (the greatest effect was the use of ethanol).

However, this does not definitively exclude the participation of ultrasound on the drying rate. The slope of the drying curve of the Water + US denotes shorter drying time for this treatment compared to the control. Another fact that does not exclude the influence of ultrasound is related to water gain during pre-treatment.

In fact, during the immersion of the samples in ethanol (with or without ultrasound), part of the water outlets and ethanol inlets – which occur simultaneously. Consequently, the “moisture” in these treatments includes both water and ethanol (as described on section 4.5). Therefore, after pre-treatments, the carrot moisture was reduced 6% in the Ethanol pre-treatment, and 21% in the Ethanol+US; in relation to the Control. Although it is still a challenge to know the exact proportion of water and ethanol after pre-treatment, microscopic evidence shows that ethanol enters only the superficial part of the samples (ROJAS; AUGUSTO, 2018b).

On the other hand, during pre-treatment using ultrasound and water, the sample absorbs water – the carrot moisture after pre-treatment Water+US was 38% higher than the control. In fact, ultrasound facilitates the entry of water into cells and intercellular spaces (KADAM et al., 2015; RICCE et al., 2016). This mechanism can help to unclog the natural pores of the samples, which can facilitate drying (RICCE et al., 2016). Consequently, it is interesting to observe although the pre-treatment Water+US increased the carrot moisture, there was no increase in drying time (Figure 12).

Therefore, depending on the pre-treatment, the sample's moisture in the beginning of drying is different. However, this was not the only factor affecting the process, once different slopes can be seen in the drying curves and the behaviour is consistent considering the time needed to reach different moisture levels (Figure 12).

This reinforces the need to investigate parameters to maximize the effect of ultrasound for each material.

The adjusted data allowed obtaining the parameters k and n of the Page Model (Figure 13).

The k parameter, which is associated with the drying rate, was lower for the Control ($0.2139 \pm 0.0162\ h^{-1}$) and Water+US ($0.2658 \pm 0.0373\ h^{-1}$) treatment, and they did not differ between them ($p = 0.62$). On the other hand, the pre-treatments with ethanol resulted in higher k values. Although no statistical differences were observed between them at $p<0.05$, this value was higher for Ethanol+US ($0.5467 \pm 0.0426\ h^{-1}$) than Ethanol ($0.4236 \pm 0.0841\ h^{-1}$) when considering $p<0.1$. This result reinforces the idea of ethanol efficacy and its bigger influence than the pre-treatment with ultrasound during the following drying. Even so, this does not exclude the participation of ultrasound in improving drying (Figure 12).

The n parameter can be interpreted as a behaviour index, once it indicates the mass transfer mechanism during processing. According to Simpson et al. (2017), $n < 1$ indicates a sub diffusion process, and $n > 1$ a super diffusion process. Therefore, when $n \neq 1$, mechanisms other than diffusion are important. For example, Rojas; Augusto (2018b) associated the "super diffusion process" ($n > 1$) with capillarity, while Rojas et al. (2020) associated a reduction on n value due to ultrasound processing of pumpkin as the formation of isolated cavities and channels larger enough to do not promote capillarity.

All the treatments presented a super diffusive behaviour ($n > 1$). However, the structural changes mentioned in section 5.1 did not affect the n value once any significant difference was observed among treatments ($p > 0.05$). Therefore, the described structural changes were able to change the rate of water outflux the samples, but they did not alter the mechanisms of mass transfer.

Studies with different vegetable products reported drying acceleration by using pre-treatments with ultrasound in water bath (RODRÍGUEZ et al., 2018). Some examples with convective drying are those with potato (MIANO; ROJAS; AUGUSTO, 2019), garlic (BOZKIR et al., 2019), unripe banana (LA FUENTE; TADINI, 2018),

pineapple (RODRÍGUEZ et al., 2017), mushrooms (ÇAKMAK et al., 2016) and apple (FIJALKOWSKA et al., 2016; NOWACKA et al., 2012). The improvement of mass transfer by ultrasound can be achieved through two types of mechanisms: directs and indirects.

The direct mechanisms are mass transfer mechanisms, such as the so called inertial flow and sponge effect (MIANO; IBARZ; AUGUSTO, 2016). In the present work, these direct mechanisms could happen during the pre-treatment, inducing the water influx into the sample (treatment Water+US) and the ethanol influx and water outflux to the sample (treatment Ethanol+US). Consequently, the ultrasound direct mechanisms induced structural and compositional changes in carrots, which impacted the further drying processing.

The indirect mechanisms are the structural changes induced by ultrasound, which are associated with the rupture of tissues and cells due to acoustic cavitation, resulting in the formation of microchannels (HUANG et al., 2019; MAGALHÃES et al., 2017). The opening of microchannels can improve mass transfer (MIANO; ROJAS; AUGUSTO, 2019), such as the following drying after the pre-treatments with ultrasound. However, acoustic cavitation can also result in the formation of isolated channels without connection with each other and with an external medium, as well as channels with different tortuosity and permeability, which can affect the improvement in mass transfer.

In the present study, the effect of ultrasound was little evident to drying when compared with ethanol, as well as no open channels were noticeable and only slightly structural modifications (Figure 8). It is worth mention the influence of ultrasound pre-treatment on drying rate will be influenced by different aspects associated with both the material and the ultrasonic processing (CÁRCEL et al., 2014; DE LA FUENTE-BLANCO et al., 2006; GAMBOA-SANTOS et al., 2014; MIANO; ROJAS; AUGUSTO, 2019; OZUNA et al., 2014). For instance, the energy and time of sonification applied in this work was insufficient for the formation of many channels in carrot, probably due to its stiffer structure. Similar results were found by Siucińska et al.(2016), who reported the ineffectiveness of ultrasound treatment in the mass transfer in cherries can be attributed to the specific morphological characteristics of the material, since that fruits are covered by a hard and waxy peel. Moreover, Ricce et al. (2016) studied pre-treatments of carrots with ultrasound in water (41 W/L, 25 kHz, up to 60 min). Although the authors did not evaluate structural changes, they reported small influence on drying

rate, with higher influence on the total amount of absorbed water. The authors suggested the ultrasonic direct effects (sponge effects) with water helps to extract the solids removed from the cells, avoiding the pores to clog during the following drying process. Nowacka; Wedzik (2016) applied pre-treatments of vacuum-packed carrots with ultrasound in water (3-4 W/cm^2; 21 and 35 kHz, up to 30 min). Although structural changes were observed, ultrasound did not change drying time.

The greatest effect in reducing drying time and increasing drying rate in carrots was due to the application of ethanol (Figure 12). In fact, the use of ethanol is a rising trend in drying research, once this pre-treatment is reported to greatly reduce processing time (LLAVATA et al., 2020). Considering convective drying, ethanol pre-treatment was proposed for pumpkin (ROJAS; AUGUSTO, 2018b), balls of mixed rice (TATEMOTO et al., 2015), bananas (CORRÊA et al., 2012), pineapple (BRAGA et al., 2009) and apple (FUNEBO et al., 2002). Moreover, the application of ethanol as pre-treatment to infrared drying was recently studied for potatoes, in combination with mechanical perforation (ROJAS; SILVEIRA; AUGUSTO, 2019) and scallion, with the combination with vacuum (WANG et al., 2019b). All works showed improved drying processes.

Different mechanisms are related with ethanol improvement.

Ethanol is an organic solvent that promotes early evaporation during the process (STREITWISER; HEATHCOCK, 1976). Moreover, Silva et al. (2012) proposed possible improvements due to Marangoni Effect: this effect promotes mass transfer in an interface between two fluids with different surface tensions. During the pre-treatment with ethanol, this gradient causes the input of ethanol in the sample, while differences on osmotic pressure causes a displacement of water from sample surface to the surrounding ethanol. Therefore, after pre-treatment, the sample outers layers contains both water and ethanol (as demonstrate by Rojas; Augusto (2018b)). During drying, ethanol vaporizes firstly, leaving water on the sample surface and resulting in a gradient of water/ethanol concentration. Therefore, during drying, the Marangoni Effect is also observed due to the existing gradient of surface tension across the sample: this process is repeated during processing, generating a constant flow until it finds a surface tension equilibrium. Moreover, the xylems vessels can improve the Marangoni effect, as demonstrated by Rojas; Augusto (2018b). Finally, the structural changes observed in parenchyma tissue with the use of ethanol (Figure 8) can improve drying by removing air and water from the tissues, as well as promoting

dissolution and disorganization of the cell wall compounds by ethanol (CANTERI et al., 2019) . All these structural changes may affect the cell wall and tissues permeability.

In addition, the effect of osmotic dehydration induced by ethanol may have contributed to the structural changes observed. In osmotic dehydration, water is removed due exposing the food to ethanol. This result in input of ethanol in the sample, while differences on osmotic pressure cause a displacement of water from the sample surface to the surrounding ethanol. In fact, Wang et al. (WANG et al., 2019b) described the pre-treatment using ethanol and ethanol using vacuum showed a good osmotic dehydration effect on scallion. In the present study, the combination of structural changes, combined with the osmotic process and the Marangoni Effect, can explain the success of ethanolic pre-treatment in reducing drying time.

Recently ethanol and ultrasound started to be combined. The ethanol and ultrasound were studied as pre-treatments for convective drying of pumpkin (ROJAS; SILVEIRA; AUGUSTO, 2020) and infrared drying of garlic (FENG et al., 2019) and potato (ROJAS; AUGUSTO, 2018c) slices and pulsed vacuum drying of apples (AMANOR-ATIEMOH et al., 2020). In addition, the combination of ethanol pre-treatment and ultrasound assisted drying was studied for apple slices (ROJAS; SILVEIRA; AUGUSTO, 2020). Therefore, the only three works in the literature combining ethanol and ultrasound as pre-treatments to convective drying were carried out with pumpkin (ROJAS; SILVEIRA; AUGUSTO, 2020); apple (ZUBERNIK et al., 2019) and melon (DA CUNHA et al., 2020).

Pumpkin cylinders were pre-treated using Ethanol+US (68 W/L, 25 kHz), up to 30 min (ROJAS; SILVEIRA; AUGUSTO, 2020). A similar reduction in drying time was obtained between treatments with Ethanol and Ethanol+US – results similar to those reported in the present work. Even so, treatment with Ethanol+US showed a higher drying rate and improved rehydration, also preserving the carotenoid content. Apples were pre-treated with Ethanol+US combination (300 W, 21 kHz), up to 3 min (ZUBERNIK et al., 2019). The drying time was reduced from 9.7% to 18.3% compared to the control. However, the use of this pre-treatment was not able to minimize the degradation of polyphenols in apple tissue during drying. Melons were immersed in ethanol with two different concentrations (50 and 100%) with and without ultrasound (154 W, 25 kHz) and vacuum up to 10 min (DA CUNHA et al., 2020). This treatment achieved a 56% reduction in drying time compared to the control. However, there was degradation of phenolic compounds, ascorbic acid and carotenoids. In all works.The

authors did not perform the treatment Water+US nor evaluated structure.

Considering infrared drying (ROJAS; AUGUSTO, 2018c), the combination of ethanol and ultrasound using a probe reactor (48 W/L, 20 kHz, up to 3 min) provided a significant reduction in drying time, which was attributed to the greatest structural changes in potato tissue. However, the high structural modifications had a negative impact on rehydration. In infrared drying of garlic slices (FENG et al., 2019), ethanol and ultrasound pre-treatments (50 W/L, three frequency, 20, 40 and 60 kHz up to 30 min) shortened the drying time for Ethanol+US treatment compared to treatments with ethanol and Water+US only. However, the allicin content has decreased, the main bioactive substance in garlic.

In pulsed vacuum drying, the combination of ethanol with ultrasound by using the ultrasonic bath (300 W/L, 20 kHz, up to 30 min in temperatures of 60, 70 and 80 °C) reduced drying time by 27% (60 °C), 31% (70 °C), and 22% at 80°C. Moreover, the total free amino acid was significantly increased with Ethanol+US for 30 min and 60 °C. This treatment also preserved the carbohydrates, phenolics, free total amino acids, as well as carboxylic acid (AMANOR-ATIEMOH et al., 2020).

In the present study, treatment with Ethanol+US ultrasound (25.7 W/L, 25 kHz, up to 30 min) contributed significantly to the improvement of drying in carrots, but the main effect was attributed to ethanol. Therefore, it is clear that the effect of ultrasound is different in each food matrix. For this reason, analysis combining reactor properties (dimensions, power, frequency) and process conditions (power, time, quantities, temperature), must be studied in order to determine which is the best condition to favours the improvement of drying without compromise quality parameters.

Moreover, it is important to highlight although the processing time reduction by itself is a very interesting result, this can also implies in reducing the energy consumption – which is relevant considering drying is a high-cost unit operation for the food industry, as this process consumes a lot of time and energy (NIETO CALVACHE et al., 2015). Therefore, the total energy consumed during pre-treatments and drying (until final moisture of 20%$_{w.b.}$) was estimated (Equation 6). Figure 14 shows the reduction (%) on the total energy consumption when pre-treatments were applied. Compared to Control, the energy was significantly reduced up to 53% ± 4% (Ethanol), 62% ± 5% (Ethanol+US) and 41% ± 8% (Water+US) for the applied pre-treatments. Non-significant differences were observed among pre-treatments ($p>0.05$), once there were non-significant differences on the drying time reductions (Figure 12). Even so,

the values represent a relevant reduction. In addition, the energy consumption during pre-treatments that include US application, represent only 0.3% ± 0.1% of the energy consumed during drying, reflecting that indeed the drying process itself is a process of intensive energy consumption. Even so, considering that there were no differences between pre-treatments, only the use of Ethanol pre-treatment would already provide a significant energy improvement compared to the Control process. Therefore, considering all the limitations and simplifications of this approach of calculi, it contributes to demonstrating that the energy consumption with the proposed pre-treatments application could be compensated during drying, in a greater proportion.

However, we emphasize that the reduction in total energy consumption does not necessarily imply in a reduction of the total cost of production. The estimated costs depend on the socioeconomic and geographical contexts of each region. For example, energy, raw material, equipment, and ethanol costs vary widely, depending on that context. Therefore, each specific micro-context must be evaluated. However, the reduction in energy consumption is a desired result by itself, as it demonstrated scientific, social and environmental contributions.

Summarizing, the pre-treatments with Ethanol and Ethanol+US was more efficient in drying carrot slices, which was associated to structural changes in the parenchyma, the ethanol vapour pressure and the flux promoted by the Marangoni Effect. In fact, partial dehydration of the parenchyma with ethanol (Ethanol and Ethanol+US) was seen in Figure 8, and greater shrinkage of the carrot slices was seen during these pre-treatment (Ethanol and Ethanol+US, Figure 11A). Considering a target final moisture of 20% (wet basis, (XIAO DONG CHEN; KAMLESH C. PATEL, 2008)), the evaluated pre-treatments reduced the drying time in 51 and 50% (Ethanol and Ethanol+US, respectively) while Water+US reduces 33%. In terms of energy consumption, all pre-treatments reduced the energy in 53% and 62% (Ethanol and Ethanol+US, respectively) while Water+US reduces 41%. Moreover, both pre-treatments and drying can affect quality parameters such as rehydration capacity and nutrient content. Therefore, it must also be evaluated, as described as follows.

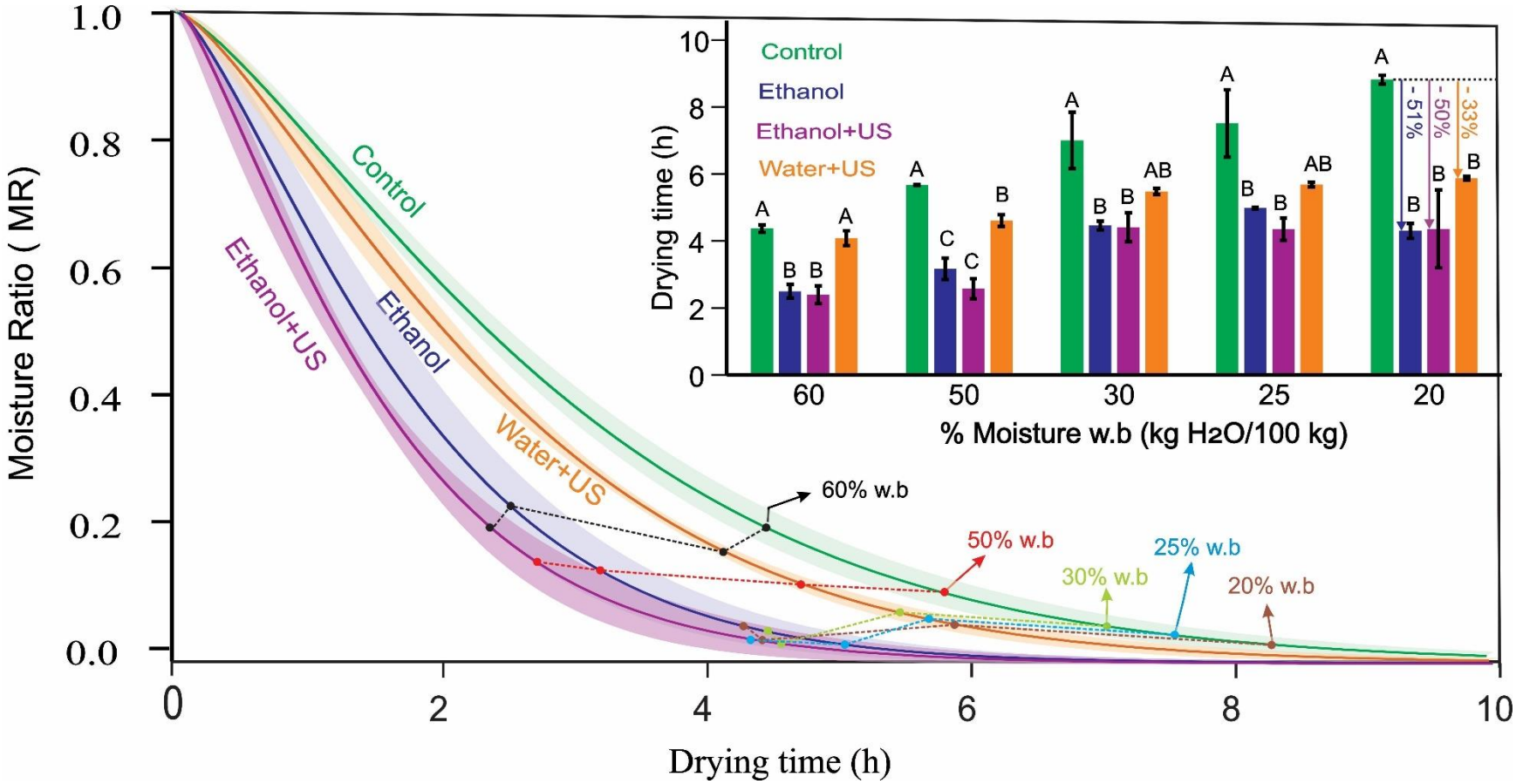

Figure 12: Convective drying kinetics of carrot slices with different pre-treatments (Control, Ethanol, Ethanol+US, Water+US; were MR is calculed in d.b according to Equation (1)). The curves are the data adjusted to the Page Model (Eq.1) and the shaded area indicates the standard deviation. Different moisture levels (in w.b) are highlighted. Insert: the bar graph corresponds to the drying time to obtain different moistures (in % w.b; vertical bars indicate standard deviation; different letters indicate statistically significant differences ($p < 0.05$) among treatments).

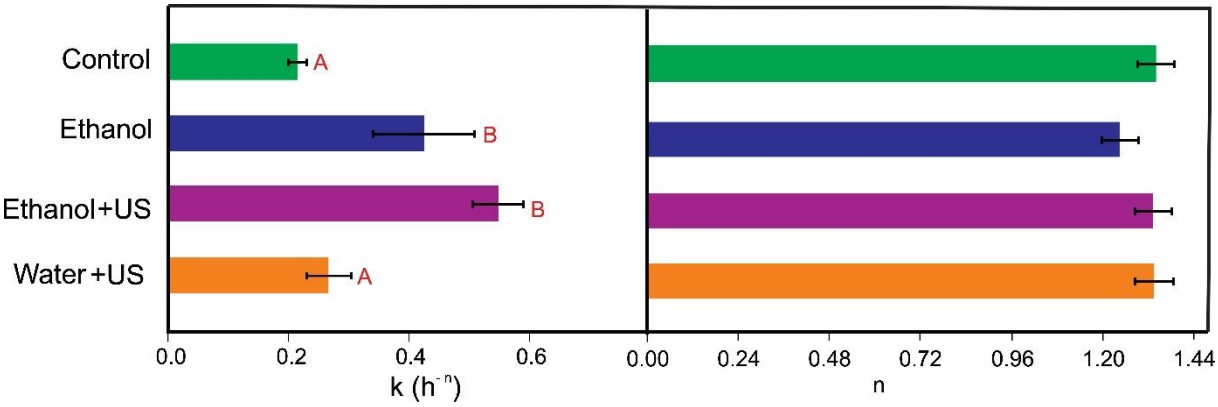

Figure 13: Parameters k and n of Page Model (Equation 1). Horizontal bars indicate the standard deviation. Different letters indicate statistically significant differences ($p < 0.05$) among treatments. For the parameter n all treatments were equal ($p > 0.05$).

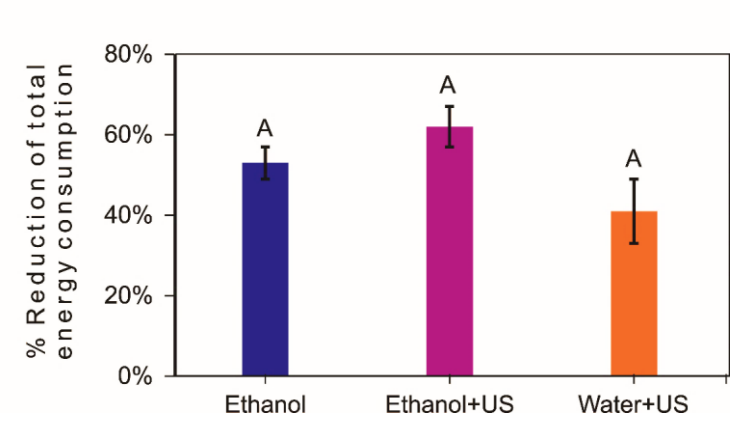

Figure 14: Reduction (%) of the total energy consumed until reach a final moisture of 20% w.b. for all pre-treatments regarding the Control treatment. Equal letters indicate non-significant differences ($p>0.05$) by Tukey test.

5.3 Influence of pre-treatments on carrot quality: rehydration kinetics and carotenoid content

Drying is an operation that promotes several changes in food, including structural modifications and possible undesirable changes in nutritional value. Consequently, this section will discuss the obtained product quality, through both its rehydration behaviour and carotenoid content, always comparing the Control and pre-treated dried carrots.

The results of rehydration kinetics are presented in Figure 15, with the data adjusted to the Peleg Model (Equation (9)). The adjusted model allowed obtaining the parameters k_1 and k_2, which helps to describe the rehydration behaviour: the lower the value of k_1, the higher the initial rate of rehydration, and the lower the value of k_2, the higher is the equilibrium moisture content.

For parameter k_1, no significant differences ($p > 0.05$) were found between Ethanol+US (0.0702 ± 0.2390 min $d.b^{-1}$) and Ethanol (0.9540 ± 0.0209 min $d.b^{-1}$) pre-treatments, while the Control (0.1327 ± 0.0239 min $d.b^{-1}$) behaved similarly to the treatment with Ethanol and Water+US (0.1476 ± 0.0141 min $d.b^{-1}$) ($p > 0.05$). For parameter k_2, there were no significant differences ($p > 0.05$) between Ethanol (0.1229 ± 0.0042 $d.b^{-1}$) and Ethanol+US (0.1124 ± 0.0074 $d.b^{-1}$) treated carrots, but difference ($p < 0.05$) was achieved from Water+US treatment and Control. These results evidence the improvement of rehydration with Ethanol and Ethanol+US treatment with higher rehydration rate and equilibrium moisture. In fact, the Ethanol+US treated sample had a water retention that exceeded the carrot original (*in natura*) moisture. The treatment with ethanol also presented a good capacity of incorporation of water reaching the same moisture content of the *in natura* carrot.

The improvement of rehydration with ethanol and/or ultrasound has been reported in other studies, such as Ethanol in pumpkin (ROJAS; AUGUSTO, 2018b) and apple (FUNEBO et al., 2002), ultrasound in carrot (WANG et al., 2018) and okra (TÜFEKÇI; ÖZKAL, 2017), and Ethanol+US in pumpkin (ROJAS; SILVEIRA; AUGUSTO, 2020). In these works, the improvement was attributed to the structural changes in tissues and cells induced by pre-treatments, which facilitates the water transfer and/or retention. However, drastic structural changes can affect the quality of rehydration, such as after infrared drying of potatoes (ROJAS; SILVEIRA; AUGUSTO, 2019): the Ethanol+US pre-treatment caused severe structural changes in the tissues and negatively impacted rehydration, which was slower than the Control treatment, and reaching a final moisture of only 74.6%w.b of the *in natura* vegetable. In that case, the main negative impact on rehydration was attributed to the application of high temperatures (80 ºC) during infrared drying, that gelatinized the starch present in potato, causing a water migration resistance provided by the surface resistance of the crust (starch gelatinized). This reflects that the effectiveness of a pre-treatment improving rehydration also depends on the composition and structure of the raw material, in addition to the effects of the drying process.

On the other hand, as observed in Figure 15, the Control and Water+US have lost their rehydration capabilities. It is probably explained by the structural changes due to longer drying time in the case of the Control. In the case of Water+US samples, since during pre-treatment the water filled inside the cells causing their swelling, during

drying that water had to leave the inside of the cells, then damaging the cell structure to a greater extent.

Summarizing, Control and Water+US treatments presented slower and smaller water absorption, which reinforces the negative structural modification due to drying. On the other hand, the pre-treatments with ethanol, with or without ultrasound, altered the carrot structure in a way that not only drying was improved, but they also avoid the negative aspects of drying in relation to rehydration. These results are important from both a perspective of application where the rehydration is necessary (such as in the formulation of soups, cakes and similar), or the description of structural modifications during pre-treatments and drying.

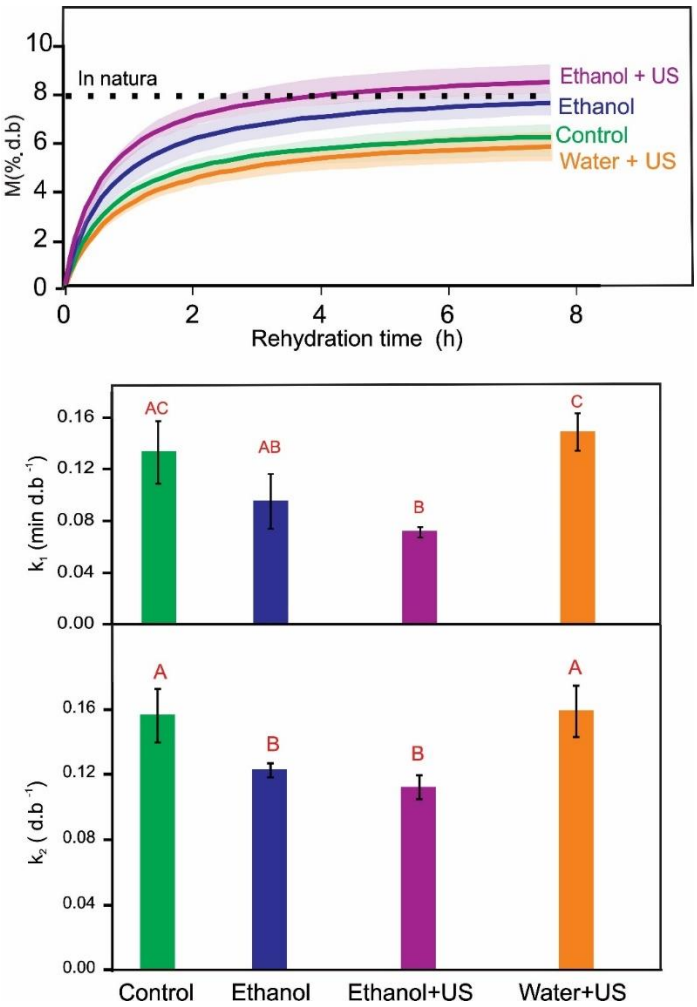

Figure 15: Rehydration kinetics of carrot slices with different pre-treatments (Control, Ethanol, Ethanol+US, Water+US). The curves are the data adjusted to the Peleg Model (Eq.9) and the shaded area indicates the standard deviation. Parameters k_1 and k_2 from Peleg Model. Vertical bars indicate the standard deviation. Different letters indicate statistically significant differences ($p < 0.05$) among treatments.

Carrots are rich in carotenoids, in special *β*-carotene (YARA-VARÓN et al., 2016), the natural pigment which give its intense orange colour. The carotenoid content varies according the carrot cultivar, and its origin. In this work, in the *in natura* sample, a carotenoid content of 15.7 ± 1 mg/100 g of *in natura* sample was obtained, which was similar to the reported by Haque et al., (2020) (10 mg/100 g of *in natura* sample), and Matějková; Petříková (2010) (8.4 – 14.1 mg/100 g of *in natura* samples). Carotenoids are important components associated with human nutrition (RODRIGUEZ-AMAYA, 2019) and health benefits (EGGERSDORFER; WYSS, 2018). However, carotenoids are compounds whose stability to oxidation is low and they can be degraded due to moisture loss over the drying time in contact with oxygen (CATHERINE NICOLLE et al., 2004). Therefore, drying process must maintain carotenoid concentration as high as possible.

In fact, different studies reported carotenoid degradation during drying of vegetables. Carotenoids degradation from 40% to 98.7% due to convective drying at 50-70 °C was reported in pumpkins (SONG et al., 2018), apricots (FRATIANNI et al., 2017), jackfruit bulb (SAXENA et al., 2012). Considering carrot drying, (ZIELINSKA; MARKOWSKI, 2012) reported 17% degradation at 60 °C and 36% at 90 °C, while (MD SALEH et al., 2020) reported degradation of 52.5% at 50 °C and 57.8% at 70 °C. However, convective drying conducted at 40 °C retained 92% of carrot carotenoids (FRIAS et al., 2010). Based on this context, we selected 40 ºC as processing temperature to avoid carotenoid degradation and maximizing the product quality.

Only one article reports the retention of carotenoid content with the application of pre-treatment with Ethanol and Ethanol+US: in the work of (ROJAS; SILVEIRA; AUGUSTO, 2020), pumpkin was convectively dried at 50 °C, with and without pre-treatments with Ethanol and Ethanol+US. The authors reported 27% reduction in the carotenoid content in the Control treatment, while the pre-treatments could maintain the original carotenoid content.

In the present study, there was no degradation of the total carotenoid content during drying for all treatments (Figure 16, $p > 0.05$), which can be related with the drying temperature (40 °C). Therefore, under the conditions evaluated in this study, the treatments with Ethanol and Ethanol+US can help improve drying and rehydration without compromising the nutritional quality of the final product. It is worth mention this is an interesting result from both academic and industrial perspectives.

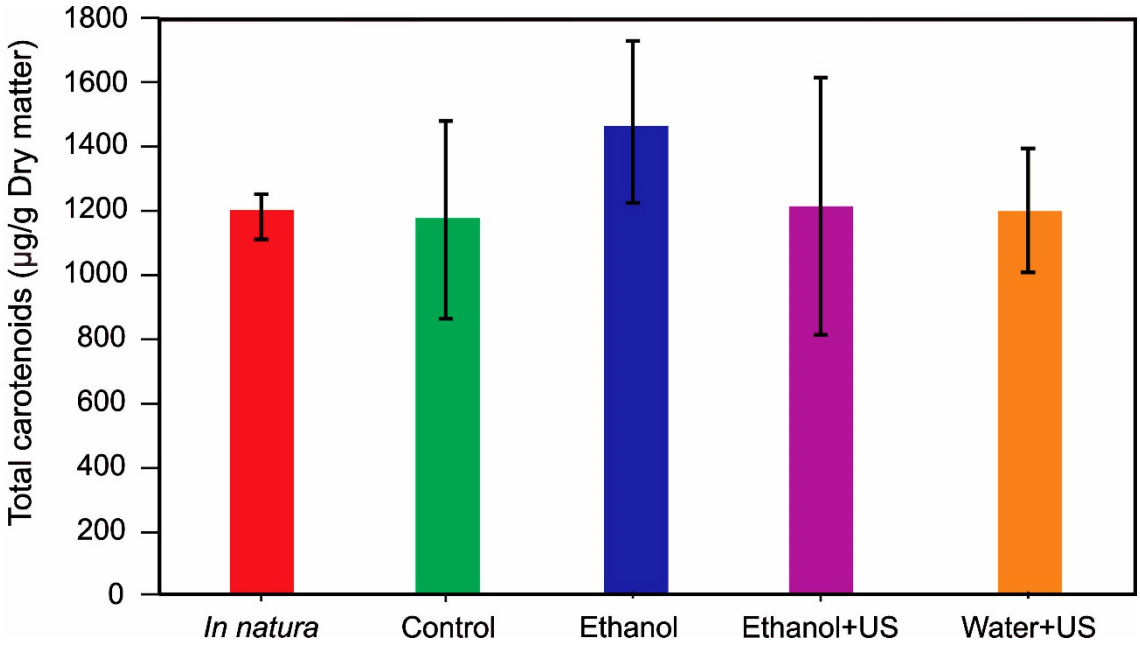

Figure 16: Total carotenoid content of the *in-natura*, and rehydrated samples (Control, Ethanol, Ethanol+US and Water+US). Vertical bars indicate the standard deviation. The carotenoids content of all treatments did not differ at p = 0.05.

5. 4 Final consideration

This work shows a possibility of obtaining dried carrots quickly, with the same carotenoid content of fresh carrots and spent less energy by using ethanol and ultrasound as pre-treatment to drying. However, it is worth mentioning that, although the objective of our work has been accomplished, further aspects must be evaluated in future studies.

Therefore, we highlight this work opens the opportunity for future evaluations, considering different food products and processing conditions, and including:

Quantify the exact amount of ethanol and water after each pre-treatment and during the drying processing;

- Quantify the residue of ethanol in different pre-treatments and drying conditions (we already started to develop this, demonstrating it is possible to achieve a negligible ethanol level in the dried product –(CARVALHO, G. R. ; SANTOS, K. C. ; MASSARIOLI, A. P. ; AUGUSTO, 2019));
- Evaluate the possibility of reusing ethanol, as well as different ethanolic solutions and also other compounds whose mechanisms and results can be similar or even better – what we call “drying accelerators”;

- Expand and improve the energy consumption evaluation, considering further environmental analysis and Life Cycle Assessment (for example, as developed by Merone et al. (MERONE et al., 2020));
- Applying ethanol at an industrial scale can be a challenge. Therefore, this theoretical basis serves as a support for future studies to evaluate the technical, operational, and industrial feasibility of applying ethanol, considering different aspects from costs, availability in each region and safety issues.

6. CONCLUSIONS

The use of ethanol and/or ultrasound was studied as pre-treatments to the convective drying of carrot slices. Both ethanol and ultrasound affected the parenchymatic cells, but the effect of ethanol was higher and mainly associated with the cell walls and membranes. The structural changes influenced the product texture (both cortex and core regions), shrinkage and improved mass transfer during both drying and rehydration. Ethanol and Ethanol+US pre-treatments reduced the drying time in ~50% when compared to the Control treatment, also increasing the water absorption and retention during rehydration. The energy consumption was reduced from 41-62% with the pre-treatments application, when compared to Control. Moreover, all the treatments could maintain the original carotenoid content. Therefore, treatments with Ethanol and Ethanol+US can be used to improve the drying process by convection of carrot slices without compromising the product's quality properties and also assist in reducing energy consumption.

7. SUGGESTIONS FOR FUTURE RESEARCH

From the results obtained in this study. Future research can be carried out in order to expand the knowledge in the area as well as to describe possibilities that have not yet been developed.

Therefore, some possibilities are here described.

Firstly, the evaluation of drying and shrinkage kinetics of each carrot region (cortex and core) can be studied separately, aiming to understand the contribution on each structure isolated on drying. This will allow to find the ideal conditions for drying carrots.

Then, different vegetables with non-homogeneous “pulp” can be evaluated and verified the contribution and response of each microstructure when using ethanol and ultrasound.

Next, other emerging technologies and their combinations can be used. For example, the use of the microwave heating (MWH) can promote a great reduction in drying time. Further, it can also be combined with vacuum, providing a quick drying at low pressures, retaining nutrients. The Infrared heating (IRH) can be used to assist the convective drying, providing high heat and mass transfer during the combined drying.

The use of other technologies can also be studied to promote structural changes. The pulsed electric field (PEF) can be used as a pre-treatment, promoting an increase in the drying rate. Combinations of these technologies can be conducted in order to obtain in-depth discussions about the mass transfer and information about better drying conditions.

Further, the evaluation of dry products can be studied considering specific applications. For example, soups with dried vegetables, studying the rehydration capacity and the nutritional and sensory qualities.

Finally, an in-depth discussion of mechanisms can be done considering different scales, transport phenomena, physical mechanisms, mathematical modelling and numerical simulation.

REFERENCES

ABID, M. et al. Sonication enhances polyphenolic compounds, sugars, carotenoids and mineral elements of apple juice. **Ultrasonics Sonochemistry**, v. 21, n. 1, p. 93–97, 2014.

AGUILAR, K. et al. Ascorbic acid stability in fruit juices during thermosonication. **Ultrasonics Sonochemistry**, v. 37, p. 375–381, 1 jul. 2017.

AMANOR- ATIEMOH, R. et al. Effect of ultrasound- ethanol pretreatment on drying kinetics, quality parameters, functional group, and amino acid profile of apple slices using pulsed vacuum drying. **Journal of Food Process Engineering**, v. 43, n. 2, 20 fev. 2020.

APRAJEETA, J.; GOPIRAJAH, R.; ANANDHARAMAKRISHNAN, C. Shrinkage and porosity effects on heat and mass transfer during potato drying. **Journal of Food Engineering**, v. 144, p. 119–128, 1 jan. 2015.

BHAT, R. et al. Sonication improves kasturi lime (Citrus microcarpa) juice quality. **Ultrasonics Sonochemistry**, v. 18, n. 6, p. 1295–1300, 2011.

BOZKIR, H. et al. Ultrasound as pretreatment for drying garlic slices in microwave and convective dryer. **Food Science and Biotechnology**, v. 28, n. 2, p. 347–354, 5 abr. 2019.

BRAGA, A. M. P. et al. Volatiles Identification in Pineapple Submitted to Drying in an Ethanolic Atmosphere. **Drying Technology**, v. 27, n. 2, p. 248–257, 30 jan. 2009.

ÇAKMAK, R. Ş. et al. Effects of electrical and sonication pretreatments on the drying rate and quality of mushrooms. **LWT - Food Science and Technology**, v. 69, p. 197–202, 1 jun. 2016.

CANTERI, M. H. G. et al. ATR-FTIR spectroscopy to determine cell wall composition: Application on a large diversity of fruits and vegetables. **Carbohydrate Polymers**, v. 212, p. 186–196, 15 maio 2019.

CÁRCEL, J. A. et al. Drying Assisted by Power Ultrasound. In: **Modern Drying Technology**. Weinheim, Germany: Wiley-VCH Verlag GmbH & Co. KGaA, 2014. v. 5p. 237–278.

CARVALHO, G. R.; SANTOS, K. C.; MASSARIOLI, A. P.; AUGUSTO, P. E. D. **Residue of ethanol in pineapple dried using ethanol and ultrasound as pre-treatments.** (Galoá, Ed.): 13º Latin American Symposium on Food Science (SLACA). **Anais**...Capinas: 2019Disponível em: <https://proceedings.science/slaca/slaca-2019/papers/residue-of-ethanol-in-pineapple-dried-using-ethanol-and-ultrasound-as-pre-treatments>

CARVALHO, G. R. DE et al. Effect of intermittent high-intensity sonication and temperature on barley steeping for malt production. **Journal of Cereal Science**, v. 82, p. 138–145, 1 jul. 2018.

CATHERINE NICOLLE et al. Genetic Variability influences carotenoid, vitamin, phenolic, and mineral content in whitem yellow, purple, orage, and dark-orange carrot cultivars. **American Society for horticulture science**, v. 129, n. 4, p. 523–529, 2004.

CHEMAT, F.; ZILL-E-HUMA; KHAN, M. K. **Applications of ultrasound in food technology: Processing, preservation and extraction**. Ultrasonics Sonochemistry. **Anais**...Elsevier B.V., 1 jul. 2011

CORRÊA, J. L. G. et al. The Influence of Ethanol on the Convective Drying of Unripe, Ripe, and Overripe Bananas. **Drying Technology**, v. 30, n. 8, p. 817–826, 15 jun. 2012.

DA CUNHA, R. M. C. et al. Effect of ethanol pretreatment on melon convective drying. **Food Chemistry**, v. 333, p. 127502, 15 dez. 2020.

DAVID SADAVA et al. **life-the-science-of-biology-10th-edition**. 10. ed. [s.l.] WH Freeman, 2012.

DE LA FUENTE-BLANCO, S. et al. Food drying process by power ultrasound. **Ultrasonics**, v. 44, p. e523–e527, 22 dez. 2006.

DIAS DA SILVA, G. et al. Pretreatments for melon drying implementing ultrasound and vacuum. **LWT - Food Science and Technology**, v. 74, p. 114–119, 1 dez. 2016.

EGGERSDORFER, M.; WYSS, A. **Carotenoids in human nutrition and healthArchives of Biochemistry and Biophysics**Academic Press Inc., , 15 ago. 2018.

EVERT RAY FRANKLIN. **Anatomia das Plantas de ESAU**. 3. ed. São Paulo: [s.n.].

FENG, Y. et al. Improvement of the catalytic infrared drying process and quality characteristics of the dried garlic slices by ultrasound-assisted alcohol pretreatment. **LWT**, v. 116, p. 108577, 1 dez. 2019.

FERNANDES, F. A. N.; GALLÃO, M. I.; RODRIGUES, S. Effect of osmotic dehydration and ultrasound pre-treatment on cell structure: Melon dehydration. **LWT - Food Science and Technology**, v. 41, n. 4, p. 604–610, 1 maio 2008.

FIJALKOWSKA, A. et al. Ultrasound as a Pretreatment Method to Improve Drying Kinetics and Sensory Properties of Dried Apple. **Journal of Food Process Engineering**, v. 39, n. 3, p. 256–265, 1 jun. 2016.

FRATIANNI, A. et al. Kinetics of carotenoids degradation and furosine formation in dried apricots (Prunus armeniaca L.). **Food Research International**, v. 99, p. 862–867, 1 set. 2017.

FRIAS, J. et al. Influence of Drying by Convective Air Dryer or Power Ultrasound on the Vitamin C and β-Carotene Content of Carrots. **J. Agric. Food Chem**, v. 58, p. 10539–10544, 2010.

FUNEBO, T. et al. Microwave and convective dehydration of ethanol treated and frozen apple - physical properties and drying kinetics. **International Journal of Food Science and Technology**, v. 37, n. 6, p. 603–614, ago. 2002.

GAMBOA-SANTOS, J. et al. Air-borne ultrasound application in the convective drying of strawberry. **Journal of Food Engineering**, v. 128, p. 132–139, maio 2014.

GAO, R. et al. The spatial-temporal working pattern of cold ultrasound treatment in improving the sensory, nutritional and safe quality of unpasteurized raw tomato juice. **Ultrasonics Sonochemistry**, v. 56, p. 240–253, 1 set. 2019a.

GAO, R. et al. The spatial-temporal working pattern of cold ultrasound treatment in improving the sensory, nutritional and safe quality of unpasteurized raw tomato juice. **Ultrasonics Sonochemistry**, v. 56, p. 240–253, 1 set. 2019b.

GRAU, R.; ANDRES, A.; BARAT, J. M. Principles of Drying. **Handbook of Fermented Meat and Poultry: Second Edition**, p. 31–38, 2014.

HAQUE, M. et al. Estimation and comparison of total carotenoid contents in ethnic vegetables, fruits and some common non leafy vegetables of Bangladesh. **~ 5 ~ International Journal of Pharmacognosy and Life Science**, v. 1, n. 1, 2020.

HUANG, C. et al. Impact of drying method on the nutritional value of the edible insect protein from black soldier fly (Hermetia illucens L.) larvae: amino acid composition, nutritional value evaluation, in vitro digestibility, and thermal properties. **European Food Research and Technology**, v. 245, n. 1, p. 11–21, 6 jan. 2019.

KADAM, S. U. et al. Effect of ultrasound pre-treatment on the drying kinetics of brown seaweed Ascophyllum nodosum. **Ultrasonics Sonochemistry**, v. 23, p. 302–307, 1 mar. 2015.

KROKIDA, M. K.; MARINOS-KOURIS, D. Rehydration kinetics of dehydrated products. **Journal of Food Engineering**, v. 57, n. 1, p. 1–7, mar. 2003.

KUCUK, H. et al. **A Review on Thin-Layer Drying-Curve EquationsDrying Technology**Taylor and Francis Inc., , 2014.

LA FUENTE, C. I. A.; TADINI, C. C. Ultrasound pre-treatment prior to unripe banana air-drying: effect of the ultrasonic volumetric power on the kinetic parameters. **Journal of Food Science and Technology**, v. 55, n. 12, p. 5098–5105, 1 dez. 2018.

LEWIS, W. K. THE SYMPOSIUM ON DRYING The Rate of Drying of Solid Materials. **The Journal of Industrial and Engineering Chemistry**, v. 13, n. 5, p. 427–432, 1921.

LLAVATA, B. et al. Innovative pre-treatments to enhance food drying: a current review. **Current Opinion in Food Science**, v. 35, p. 20–26, 1 out. 2020.

MAGALHÃES, M. L. et al. Drying intensification combining ultrasound pre-treatment and ultrasound-assisted air drying. **Journal of Food Engineering**, v. 215, p. 72–77, 1 dez. 2017.

MALEKI, M. et al. Hot air drying kinetics of novel functional carrot snack: Impregnated using polyphenolic rich osmotic solution with ultrasound pretreatment. **Journal of Food Process Engineering**, p. 1–11, 3 dez. 2019.

MASON, T. J. **Power ultrasound in food processing - the way forward. In: Ultrasound in Food Processing**. 1. ed. London: [s.n.].

MATĚJKOVÁ, J.; PETŘÍKOVÁ, K. Variation in Content of Carotenoids and Vitamin C in Carrots. **Notulae Scientia Biologicae**, v. 2, n. 4, p. 88–91, 2010.

MCMINN, W. A. M.; MAGEE, T. R. A. Principles, methods and applications of the convective drying of foodstuffs. **Food and Bioproducts Processing: Transactions of the Institution of of Chemical Engineers, Part C**, v. 77, n. 3, p. 175–193, 1999.

MD SALEH, R. et al. Investigation of dynamic quality changes and optimization of drying parameters of carrots (<scp> *Daucus carota* </scp> var. *laguna*). **Journal of Food Process Engineering**, v. 43, n. 2, 29 fev. 2020.

MERONE, D. et al. Energy and environmental analysis of ultrasound-assisted atmospheric freeze-drying of food. **Journal of Food Engineering**, v. 283, p. 110031, 1 out. 2020.

MIANO, A. C.; IBARZ, A.; AUGUSTO, P. E. D. Mechanisms for improving mass transfer in food with ultrasound technology: Describing the phenomena in two model cases. **Ultrasonics Sonochemistry**, v. 29, p. 413–419, 1 mar. 2016.

MIANO, A. C.; ROJAS, M. L.; AUGUSTO, P. E. D. Structural changes caused by ultrasound pretreatment: Direct and indirect demonstration in potato cylinders. **Ultrasonics Sonochemistry**, v. 52, p. 176–183, 1 abr. 2019.

MICHAEL, J. M.; LU, W.; KATHRYN, L. M. Engineerering properties of foods. In: BOCA RATON: CRC PRESS (Ed.). . **Ultrasound properties**. 3. ed. [s.l: s.n.]. p. 567–605.

MOTEVALI, A.; MINAEI, S.; KHOSHTAGAZA, M. H. Evaluation of energy consumption in different drying methods. **Energy Conversion and Management**, v. 52, n. 2, p. 1192–1199, 1 fev. 2011.

MUJUMDAR, A.; OSMAN, P. Handbook of Industrial Drying Chapter 35. **Industry**, p. 1–1312, 2006.

NIETO CALVACHE, J. E. et al. Obtention of dietary fibre enriched fractions from peach bagasse using ethanol pre-treatment and microwave drying. **LWT - Food Science and Technology**, v. 62, n. 2, p. 1169–1176, 1 jul. 2015.

NOWACKA, M. et al. Drying of ultrasound pretreated apple and its selected physical properties. **Journal of Food Engineering**, v. 113, n. 3, p. 427–433, 1 dez. 2012.

NOWACKA, M.; WEDZIK, M. Effect of ultrasound treatment on microstructure, colour and carotenoid content in fresh and dried carrot tissue. **Applied Acoustics**, v. 103, p. 163–171, 1 fev. 2016.

ONWUDE, D. I. et al. Modeling the Thin-Layer Drying of Fruits and Vegetables: A Review. **Comprehensive Reviews in Food Science and Food Safety**, v. 15, n. 3, p. 599–618, 1 maio 2016.

ONWUDE, D. I. et al. The effectiveness of combined infrared and hot-air drying strategies for sweet potato. **Journal of Food Engineering**, v. 241, p. 75–87, 1 jan. 2019.

OZUNA, C. et al. Influence of material structure on air-borne ultrasonic application in drying. **Ultrasonics Sonochemistry**, v. 21, n. 3, p. 1235–1243, 1 maio 2014.

PAGE, G. E. FACTORS INFLUENCING THE MAXIMUM RATES OF AIR DRYING SHELLED CORN IN THIN LAYERS. **Theses and Dissertations Available from ProQuest**, 1 jan. 1949.

PELEG, M. Characterization of the Stress Relaxation Curves of Solid Foods. **Journal of Food Science**, v. 44, n. 1, p. 277–281, 1979.

POTOSÍ-CALVACHE, D. C.; VANEGAS-MAHECHA, P.; MARTÍNEZ-CORREA, H. A. Secado convectivo de zapallo (Cucúrbita moschata): Influencia de la temperatura y velocidad de aire sobre la difusividad efectiva de humedad, contenido de carotenoides y fenoles totales. **Dyna**, v. 84, n. 202, p. 112–119, 2017.

RASO, J. et al. Influence of different factors on the output power transferred into medium by ultrasound. **Ultrasonics Sonochemistry**, v. 5, n. 4, p. 157–162, 1 jan. 1999.

RICCE, C. et al. Ultrasound pre-treatment enhances the carrot drying and rehydration. **Food Research International**, v. 89, p. 701–708, 1 nov. 2016.

RODRIGUEZ-AMAYA, D. B. Update on natural food pigments - A mini-review on carotenoids, anthocyanins, and betalains. **Food Research International**, v. 124, p. 200–205, 1 out. 2019.

RODRÍGUEZ, Ó. et al. Effect of acoustically assisted treatments on vitamins, antioxidant activity, organic acids and drying kinetics of pineapple. **Ultrasonics Sonochemistry**, v. 35, p. 92–102, 1 mar. 2017.

RODRÍGUEZ, Ó. et al. Application of power ultrasound on the convective drying of fruits and vegetables: effects on quality. **Journal of the Science of Food and Agriculture**, v. 98, n. 5, p. 1660–1673, 30 mar. 2018.

ROJAS, M. L. et al. Peach juice processed by the ultrasound technology: Changes in its microstructure improve its physical properties and stability. **Food Research International**, v. 82, p. 22–33, 1 abr. 2016.

ROJAS, M. L.; ALVIM, I. D.; AUGUSTO, P. E. D. Incorporation of microencapsulated hydrophilic and lipophilic nutrients into foods by using ultrasound as a pre-treatment for drying: A prospective study. **Ultrasonics Sonochemistry**, v. 54, p. 153–161, 1 jun. 2019.

ROJAS, M. L.; AUGUSTO, P. E. D. Microstructure elements affect the mass transfer in foods: The case of convective drying and rehydration of pumpkin. **LWT**, v. 93, p. 102–108, 1 jul. 2018a.

ROJAS, M. L.; AUGUSTO, P. E. D. Ethanol pre-treatment improves vegetable drying and rehydration: Kinetics, mechanisms and impact on viscoelastic properties. **Journal of Food Engineering**, v. 233, p. 17–27, 1 set. 2018b.

ROJAS, M. L.; AUGUSTO, P. E. D. Ethanol and ultrasound pre-treatments to improve infrared drying of potato slices. **Innovative Food Science and Emerging Technologies**, v. 49, p. 65–75, 1 out. 2018c.

ROJAS, M. L.; SILVEIRA, I.; AUGUSTO, P. E. D. Improving the infrared drying and rehydration of potato slices using simple approaches: Perforations and ethanol. **Journal of Food Process Engineering**, p. e13089, 7 maio 2019.

ROJAS, M. L.; SILVEIRA, I.; AUGUSTO, P. E. D. Ultrasound and ethanol pre-treatments to improve convective drying: Drying, rehydration and carotenoid content of pumpkin. **Food and Bioproducts Processing**, v. 119, p. 20–30, 1 jan. 2020.

SAXENA, A. et al. Degradation Kinetics of Colour and Total Carotenoids in Jackfruit (Artocarpus heterophyllus) Bulb Slices During Hot Air Drying. **Food and Bioprocess Technology**, v. 5, n. 2, p. 672–679, 18 fev. 2012.

SILVA, M. A.; BRAGA, A. M. P.; SANTOS, P. H. S. Enhancement of fruit drying: the ethanol effect. **Proceedings of the 18th International Drying Symposium (IDS 2012)**, n. November, p. 11–15, 2012.

SIMPSON, R. et al. Understanding the success of Page's model and related empirical equations in fitting experimental data of diffusion phenomena in food matrices. **Trends in Food Science & Technology**, v. 62, p. 194–201, 1 abr. 2017.

SIUCIŃSKA, K. et al. Effects of Ultrasound Assistance on Dehydration Processes and Bioactive Component Retention of Osmo-Dried Sour Cherries. **Journal of Food Science**, v. 81, n. 7, p. C1654–C1661, 1 jul. 2016.

SMITH, B. G.; HO, C. A. L. Article 7 Moisture. **International Journal of Food Engineering**, v. 3, n. 4, 2007.

SONG, J. et al. Degradation of carotenoids in dehydrated pumpkins as affected by different storage conditions. **Food Research International**, v. 107, p. 130–136, 1 maio 2018.

SORIA, A. C.; VILLAMIEL, M. Effect of ultrasound on the technological properties and bioactivity of food: A review. **Trends in Food Science and Technology**, v. 21, n. 7, p. 323–331, jul. 2010.

STREITWISER, A.; HEATHCOCK, C. H. **Introduction to organic chemistry**. New York: [s.n.].

TAO, Y. et al. Applications of water blanching, surface contacting ultrasound-assisted air drying, and their combination for dehydration of white cabbage: Drying mechanism, bioactive profile, color and rehydration property. **Ultrasonics Sonochemistry**, v. 53, p. 192–201, 1 maio 2019.

TAO, Y.; SUN, D.-W. Enhancement of Food Processes by Ultrasound: A Review. **Critical Reviews in Food Science and Nutrition**, v. 55, n. 4, p. 570–594, 21 mar. 2015.

TATEMOTO, Y. et al. Drying characteristics of food materials injected with organic solvents in a fluidized bed of inert particles under reduced pressure. **Journal of Food Engineering**, v. 158, p. 80–85, 1 ago. 2015.

TÜFEKÇI, S.; ÖZKAL, S. G. Enhancement of drying and rehydration characteristics of okra by ultrasound pre-treatment application. **Heat and Mass Transfer/Waerme- und Stoffuebertragung**, v. 53, n. 7, p. 2279–2286, 1 jul. 2017.

VINATORU, M. Ultrasonically assisted extraction (UAE) of natural products some guidelines for good practice and reporting. **Ultrasonics Sonochemistry**, v. 25, n. 1, p. 94–95, 1 jul. 2015.

WANG, J. et al. Influence of high-intensity ultrasound on bioactive compounds of strawberry juice: Profiles of ascorbic acid, phenolics, antioxidant activity and microstructure. **Food Control**, v. 96, p. 128–136, 1 fev. 2019a.

WANG, J.; VANGA, S. K.; RAGHAVAN, V. High-intensity ultrasound processing of kiwifruit juice: Effects on the ascorbic acid, total phenolics, flavonoids and antioxidant capacity. **LWT**, v. 107, p. 299–307, 1 jun. 2019.

WANG, L. et al. Low frequency ultrasound pretreatment of carrot slices: Effect on the moisture migration and quality attributes by intermediate-wave infrared radiation drying. **Ultrasonics Sonochemistry**, v. 40, p. 619–628, 1 jan. 2018.

WANG, X. et al. Effect of vacuum and ethanol pretreatment on infrared-hot air drying of scallion (Allium fistulosum). **Food Chemistry**, v. 295, p. 432–440, 15 out. 2019b.

XIAO DONG CHEN; KAMLESH C. PATEL. Biological Changes During food drying process. In: **Drying Tecnologies in Food Processing**. Wiley Blackwell ed. [s.l: s.n.]. p. 90–109.

YARA-VARÓN, E. et al. Is it possible to substitute hexane with green solvents for extraction of carotenoids? A theoretical versus experimental solubility study. **RSC Advances**, v. 6, n. 33, p. 27750–27759, 16 mar. 2016.

ZDUNEK, A.; UMEDA, M. INFLUENCE OF CELL SIZE AND CELL WALL VOLUME FRACTION ON FAILURE PROPERTIES OF POTATO AND CARROT TISSUE. **Journal of Texture Studies**, v. 36, n. 1, p. 25–43, 1 fev. 2005.

ZIELINSKA, M.; MARKOWSKI, M. Color Characteristics of Carrots: Effect of Drying and Rehydration. **International Journal of Food Properties**, v. 15, n. 2, p. 450–466, 1 mar. 2012.

ZUBERNIK, J. et al. The Impact of the Pre-Treatment in Ethanol Solution on the Drying Kinetics and Selected Properties of Convective Dried Apples. **International Journal of Food Engineering**, v. 20, n. 1–2, 9 nov. 2019.

APPENDICES

APPENDIX A. Simple abstract/ Resumo simples/ Resumen sencillo

Simple abstract (English)

Although drying is an old method of food preservation, several challenges still require study to improve this technology. In this Master's Dissertation, we study technologies to improve the following aspects of carrot drying: processing time, energy consumption, and characteristics of the final product. Two technologies were studied, ultrasound and ethanol use, separately and together, and several evaluations were made. It was possible to carry out faster drying processes with less energy consumption, resulting in dried carrots with the same nutritional characteristics of *in nature* vegetable and better ability to absorb water.

Keywords: 1. Convective drying 2. Rehydration 3. Ethanol 4. ultrasound

Resumo Simples (Português)

Embora a secagem seja um método antigo de conservação de alimentos, diversos desafios ainda demandam estudo para melhoria dessa tecnologia. Na presente Dissertação de Mestrado, estudamos tecnologias para melhorar os seguintes aspectos da secagem de cenouras: tempo de processo, consumo de energia, e características do produto final. Duas tecnologias foram estudadas, ultrassom e uso do etanol, isoladamente e em conjunto, e diversas avaliações foram feitas. Foi possível realizar processos de secagem mais rápidos e com menor consumo de energia, resultando em cenouras secas com as mesmas características nutricionais do vegetal *in natura* e melhor capacidade de absorver água.

Palavras-chave: 1. Secagem convectiva 2. Reidratação 3. Etanol 4. ultrassom

Resumen sencillo (español)

Aunque el secado es un método antiguo de conservación de alimentos, varios desafíos aún requieren estudios para mejorar el proceso convencional de secado. En esta tesis de maestría, estudiamos tecnologías para mejorar los siguientes aspectos del secado de zanahoria: tiempo de proceso, consumo de energía y características del producto final. Se estudiaron el uso de ultrasonido y etanol, por separado y en conjunto, realizándose varias evaluaciones. Se logró obtener procesos de secado más

rápidos y con menor consumo energético, dando como resultados zanahorias deshidratadas con las mismas características nutricionales que los alimentos frescos y mayor capacidad de absorción de agua.

Palabras clave: Secado por convección ; Rehidratación ; Etanol; Ultrasonido

APPENDIX B.

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Enhancing carrot convective drying by combining ethanol and ultrasound as pre-treatments: Effect on product structure, quality, energy consumption, drying and rehydration kinetics

Karoline Costa Santos[a], Jaqueline Souza Guedes[a], Meliza Lindsay Rojas[b], Gisandro Reis Carvalho[a], Pedro Esteves Duarte Augusto[a,c,*]

[a] Department of Agri-food Industry, Food and Nutrition (LAN), Luiz de Queiroz College of Agriculture (ESALQ), University of São Paulo (USP), Piracicaba, SP, Brazil
[b] Dirección de Investigación y Desarrollo, Universidad Privada del Norte (UPN), Trujillo, Peru
[c] Food and Nutrition Research Center (NAPAN), University of São Paulo (USP), São Paulo, SP, Brazil

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ABSTRACT

Ultrasound was combined with ethanol to improve different aspects of carrot convective drying, evaluating both processing and product quality. The ultrasound in water treatment resulted in cellular swelling and small impact on texture. Differently, the ultrasound in ethanol and ethanol treatments modified both carrot microstructure (cell wall modifications of parenchymatic tissue) and macrostructure (shrinkage and resistance to perforation). Pre-treatments with ultrasound in ethanol and ethanol improved the drying kinetics, reducing the processing time (~50%) and the energy consumption (42–62%). These pre-treatments also enhanced rehydration, whose initial rate and water retention were higher than the control. In addition, the carotenoid content was preserved after drying, for all the treatments. Any impact on shrinkage was observed. A mechanistic discussion, based on structural modification (microstructure and macrostructure) and physical properties of water and ethanol, was provided. As conclusion, this work not only described positive aspects of combining the technologies of ultrasound and ethanol as pre-treatments to convective drying, but also proposed mechanisms to explain the phenomena.

1. Introduction

Drying is an important operation in the food industry, producing safe and stable food products, and also reducing post-harvest losses. This operation allows for obtaining various products such as snacks, soups and dried fruits [1], which can be consumed directly or after rehydration.

Drying has numerous advantages in food preservation. However, conventional convective drying is a long process, which also presents a high energy consumption [2]. Moreover, the long processing time, associated with high temperatures, can result in undesirable changes, such as nutrient degradation or poor rehydration capacity.

Therefore, different strategies are being studied to enhance food drying, including the application of pre-treatments [3]. In this context, both ethanol and ultrasound can be used as a promising alternative in food processing.

The pre-treatment with ultrasound has been studied in different products, while the studies using pre-treatments with ethanol are now increasing. However, the combination of both approaches (conducting ultrasound processing with ethanol) was only recently proposed. Rojas et al. [4] conducted a work combining ethanol and ultrasound (Ethanol + US) pre-treatments to convective drying of pumpkin. The combined treatment reduced the drying time and the energy consumption during processing, improved the rehydration and avoid carotenoid degradation. The same combination (Ethanol + US) was also proposed prior to apple convective drying [5], with smaller processing time (3 min). They obtained significative reduction of drying time, although the combination did not minimize the degradation the phenolic compounds. The combination Ethanol + US was also evaluated on melon, with two ethanol concentrations (50 and 100%) and convective drying at 60° C. The treatment with a higher concentration of ethanol obtained shorting drying time, but there was degradation of phenolic compounds, ascorbic acid and carotenoids, when compared to dry samples without treatment and fresh melons [6]. However, in all woks, the authors did not evaluate the product structure nor performed the pre-treatment using water in the ultrasonic bath. Moreover, the three

* Corresponding author at: Avenida Pádua Dias, 11, Piracicaba, SP 13418-900, Brazil.
E-mail address: pedro.ed.augusto@usp.br (P.E.D. Augusto).

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