



LEANDRO LEVATE MACEDO

**CONVECTIVE DRYING WITH ETHANOL PRE-
TREATMENT AND INTERMITTENT MICROWAVE DRYING
OF FRESH AND ISOMALTULOSE-ENRICHED
STRAWBERRY**

LAVRAS-MG

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Prof. Dr. Jefferson Luiz Gomes Corrêa

Orientador

Prof. Dr. Irineu Petri Júnior

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LEANDRO LEVATE MACEDO

**SECAGEM CONVECTIVA COM PRÉ-TRATAMENTO COM ETANOL E
SECAGEM POR MICRO-ONDAS INTERMITENTE DE MORANGO FRESCO E
ENRIQUECIDO COM ISOMALTULOSE**

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ABSTRACT

Strawberries are nutritionally rich, with appreciated sensory characteristics. However, as it is a highly perishable fruit, the processing of the strawberry is indicated. Osmotic dehydration (OD) is a process of mass transfers between the food and the hypertonic solution. One of these mass flows is solid incorporation, which makes OD an interesting strategy to promote the enrichment of fruits with solutes beneficial to health. Isomaltulose can be used as an osmotic agent. It is a carbohydrate with a low glycemic index and low cariogenic potential. Another mass flow of OD is water loss. However, the amount of water removed during OD is insufficient to guarantee the stability of the product for long periods. Convective drying is the most traditional method for reducing the moisture content of food. Ethanol pre-treatment is an efficient procedure in reducing drying time and minimizing the degradation caused during convective drying. In turn, the microwave method is an alternative for drying food, capable of promoting volumetric heating, which is associated with thermosensitive compounds preservation. Therefore, the aims of this work were (i) to enrich the strawberry with isomaltulose, by means of OD using osmotic solutions of 25 or 35%, at atmospheric pressure and with vacuum application during the first 10 or 20 min; (ii) to study the drying kinetics and quality of fresh and isomaltulose-enriched strawberries, pre-treated, or not, with ethanol; and (iii) to evaluate the influence of the use of intermittent microwaves in the drying of fresh and isomaltulose enriched strawberries. OD was shown to be efficient for the incorporation of isomaltulose in strawberries, especially using the osmotic solution with the highest concentration and applying a vacuum. However, some losses due to the leaching of anthocyanins, phenolic compounds and antioxidants were observed. In convective drying, the osmo-dehydrated strawberry demanded more time to complete the process and contributed to the reduction of shrinkage and hygroscopicity of the dried strawberry. The addition of ethanol contributed to the reduction of drying time and minimized the degradation of antioxidant compounds in strawberries. Drying by intermittent microwaves required less time and consumed less energy, in addition to contributing to the preservation of strawberry color and bioactive compounds and antioxidants. The osmotic process promoted the enrichment with isomaltulose and reduced the shrinkage of dried strawberries.

Keywords: Osmotic dehydration. Alternative osmotic agent. Drying kinetics. Microwave drying. Antioxidant compounds.

RESUMO

Morangos são ricos nutricionalmente, com apreciadas características sensoriais. Entretanto, por ser uma fruta altamente perecível, o processamento do morango é indicado. A desidratação osmótica (DO) é um processo de transferências de massa entre o alimento e a solução hipertônica. Um desses fluxos de massa é a incorporação de sólidos, que faz da DO uma interessante estratégia para promover o enriquecimento de frutas com solutos benéficos a saúde. A isomaltulose pode ser usado como agente osmótico. Trata-se de um carboidrato de baixo índice glicêmico e baixo potencial cariogênico. Outro fluxo de massa da DO é a perda de água. No entanto, a quantidade de água removida durante a DO é insuficiente para garantir a estabilidade do produto por longos períodos. A secagem convectiva é o método mais tradicional para redução do teor de umidade de alimentos. O pré-tratamento com etanol tem se mostrado como um procedimento eficiente na redução do tempo de secagem e na minimização de degradações causadas durante a secagem convectiva. O micro-ondas, por sua vez, é um método alternativo para secagem de alimentos, capaz de promover o aquecimento volumétrico, o que está associado a preservação de compostos termossensíveis. Diante disso, os objetivos deste trabalho foram (i) realizar o enriquecimento do morango com isomaltulose, por meio da DO usando soluções osmótica de 25 ou 35%, a pressão atmosférica e com aplicação de vácuo durante os primeiros 10 ou 20 min; (ii) estudar a cinética de secagem e a qualidade do morango fresco e enriquecido com isomaltulose, não ou pré-tratado com etanol; e (iii) avaliar a influência do uso do micro-ondas intermitente na secagem de morango fresco e enriquecido com isomaltulose. A DO mostrou-se eficiente para a incorporação de isomaltulose em morango, especialmente, usando a solução osmótica de maior concentração e aplicando-se o vácuo. Entretanto, algumas perdas por lixiviação de antocianinas, compostos fenólicos e antioxidantes foram observadas. Na secagem convectiva, o morango osmo-desidratado demandou maior tempo para conclusão do processo e contribuiu com a redução do encolhimento e higroscopicidade do morango seco. A adição de etanol contribuiu com a redução do tempo de secagem e minimizou a degradação dos compostos antioxidantes do morango. A secagem por micro-ondas intermitente demandou menor tempo e consumiu menos energia elétrica, além de contribuir com a preservação da cor e compostos bioativos e antioxidantes do morango. O processo osmótico promoveu o enriquecimento com isomaltulose e reduziu o encolhimento dos morangos secos.

Palavras-chave: Desidratação osmótica. Agente osmótico alternativo. Cinética de secagem. Secagem por micro-ondas. Compostos antioxidantes.

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FIRST SECTION

1 INTRODUCTION

Strawberry is a pseudo fruit, belonging to the group of non-climacteric fruits, of great sensory appreciation, mainly in olfactory, taste and visual terms, which makes strawberry one of the most produced and consumed fruits in the world. Moreover, strawberry has low caloric and high content of antioxidant compounds, such as vitamin C and anthocyanins, in addition to dietary fibers and various minerals. However, it has low physical resistance, high respiratory rate and high water activity, which makes the strawberry highly perishable. Strawberry processing is an important and interesting strategy to add value, develop other products and increase shelf life, reducing post-harvest losses (ALVAREZ-SUAREZ et al., 2014; ANTUNES; JÚNIOR; SCHWENGBER, 2016; BASU et al., 2014; BHAT; STAMMINGER, 2015).

One of the methods for processing strawberries is osmotic dehydration (OD) (GAMBOA-SANTOS; CAMPAÑONE, 2019). OD is a unitary operation that consists of immersing the food in a hypertonic osmotic solution. During this process, mass transfers between the food and the solution occur due to the osmotic pressure gradient. The application of vacuum at the beginning of OD, whose process is called pulsed vacuum osmotic dehydration (PVOD), has shown an effect in maximizing the rates of mass transfer throughout the osmotic process (GONZÁLEZ-PÉREZ; RAMÍREZ-CORONA; LÓPEZ-MALO, 2021).

The incorporation of solute into the food is one of the mass flows that occur during OD, which makes it an interesting strategy for the enrichment of fruits with carbohydrates good for health (MACEDO et al., 2021b), such as isomaltulose. Isomaltulose is a carbohydrate with low cariogenic and glycemic indexes, contributing to dental health and minimizing glycemic peaks, respectively. This last feature is attractive for athletes and diabetics (SAWALE et al., 2017; SHYAM; RAMADAS; CHANG, 2018).

In addition to solid gain, the water loss from the food to the osmotic solution is another mass flow that occurs during OD. However, osmotically dehydrated products generally do not have sufficiently low moisture content to guarantee stability and long shelf life, making it necessary to use a complementary drying method, such as convective and microwave drying (GONZÁLEZ-PÉREZ; RAMÍREZ-CORONA; LÓPEZ-MALO, 2021; MACEDO et al., 2021b; RAMYA; JAIN, 2017).

Convective drying is the most used method for food drying. It consists of exposing the material to a heated air flow (MAISNAM et al., 2015). However, this method has some

limitations. As a result, some procedures, such as ethanol pre-treatment (ET), have been implemented for convective drying and alternative methods, such as microwave drying, have been developed to streamline the drying process and reduce food damage (JUNQUEIRA; CORRÊA; ERNESTO, 2017; MACEDO et al., 2021a).

Ethanol can be added to foods by spraying, dipping, or immersion. ET is effective in reducing the drying time of various foods (MACEDO et al., 2021a). Thus, the food is exposed to the drying air for a shorter time, minimizing undesirable changes to the dried product, such as reducing the color change, preserving heat-sensitive compounds, such as vitamin C, betalains, carotenoids, anthocyanins, phenolic compounds and antioxidants (ARAÚJO et al., 2020; CORRÊA et al., 2012; DA CUNHA et al., 2020; ROJAS; SILVEIRA; AUGUSTO, 2020).

Microwave drying is gaining prominence nowadays, given the significant reduction in drying time caused by the rapid heating and volumetric heating of the product. Such characteristics are associated with improving the quality of the dried product, making microwave drying a promising food technology (FENG; YIN; TANG, 2012; KUMAR; KARIM, 2019). However, the continuous power supply may overheat the product. Thus, an alternative solution is to use the microwave discontinuously, that is, intermittently, in which the magnetron is turned on and off during the drying process, causing the uniform distribution of moisture and heat (HUANG et al., 2021).

The present study aimed to evaluate the influence of convective drying with ethanol pre-treatment and intermittent microwave drying on drying kinetics and quality parameters of strawberries enriched with isomaltulose. This thesis was developed in three articles. Therefore, in the first article, osmotic dehydration at atmospheric pressure and osmotic dehydration with a vacuum pulse during the first 10 or 20 min were studied, using osmotic solutions of 25 or 35% isomaltulose. The mass transfer kinetics and the qualitative parameters of the samples were evaluated. In the second article, fresh strawberries, dehydrated osmotically using the 35% osmotic solution, at atmospheric pressure and with a vacuum pulse for 20 min were subjected to ethanol pre-treatment. Samples, non and pre-treated with ethanol, were dried by convective drying at 60 °C and 1 m s⁻¹, until a moisture content of 11.50%. The drying kinetics and qualitative properties were evaluated. In the third article, fresh and osmotically dehydrated strawberries were dried using intermittent microwave drying and heated air drying, at 60 °C, until a moisture content of 10.28%. Drying and qualitative parameters were evaluated.

2 THEORETICAL REFERENCE

2.1 Strawberry

Strawberry is one of the most produced and consumed fruits in the world, whose global production was approximately 8.9 million tons in 2019. The largest national production was from China, followed by the United States, representing 36.26 and 11.50% of world production, respectively. In that same year, Brazilian production was 165 thousand tons, which represents about 1.86% of world production (FAO, 2019). Among the Brazilian states, Minas Gerais stands out as the largest strawberry producer, reaching a production exceeding 50% of the national, followed by Paraná, Rio Grande do Sul, São Paulo and Espírito Santo (ANTUNES; JÚNIOR; SCHWENGBER, 2016).

In Minas Gerais, strawberry production comes from 40 cities. The municipality of Pouso Alegre is the main producer of strawberries, with a share of more than 40%. The mean productivity is 41 t ha⁻¹, involving more than 5.9 thousand producers and generating more than 39 thousand direct and indirect jobs in Minas Gerais. In the south of the state, the strawberry culture represents expressive participation in the region's economy (ANTUNES; JÚNIOR; SCHWENGBER, 2016).

Although it is often referred to as a fruit, the strawberry is a pseudofruit, since the pericarp is developed from an accessory organ (CHITARRA; CHITARRA, 2005). In the case of strawberries, fertilization stimulates the growth of the receptacle, which, in turn, becomes fleshy, constituting a pseudofruit (ANTUNES; JÚNIOR; SCHWENGBER, 2016).

Strawberries fall into the group of non-climacteric fruits, as well as citrus fruits, cherry, grape and pineapple. This characteristic is due to the gradual reduction of respiration and there is no production of endogenous ethylene. Ripening is still complete in the plant, before harvesting. Thus, strawberries should be harvested already mature, which reduces their lifetime (ANTUNES; JÚNIOR; SCHWENGBER, 2016; CHITARRA; CHITARRA, 2005).

The color and flavor of the strawberry are attractive in sensory terms and, together with the nutritional properties, leads to it arousing interest in its consumption (DU et al., 2011). The sensory appreciation of the strawberry is the result of multiple interactions between the constituents of the strawberry, such as sugars, acids and pigments, and the human senses, such as taste, sight, touch and smell (SCHWIETERMAN et al., 2014). This makes the strawberry the fifth most consumed fruit in the United States, behind only bananas, apples, oranges and grapes (BASU et al., 2014; FAO, 2019).

The nutritional composition of fresh strawberries is rich and diversified, with several minerals and vitamins, as shown in Table 1.

Table 1 – Centesimal composition, minerals and vitamins present in 100 g of fresh strawberry

Centesimal composition		Mineral content		Vitamin content	
Water	90.95 g	Calcium	16.00 mg	Vitamin C	58.80 mg
Energy	32.00 kcal	Iron	0.41 mg	Thiamin	0.02 mg
Protein	0.67 g	Magnesium	13.00 mg	Riboflavin	0.02 mg
Ash	0.40 g	Phosphorus	24.00 mg	Niacin	0.39 mg
Total lipid	0.30 g	Potassium	153.00 mg	Pantothenic acid	0.13 mg
Carbohydrate	7.68 g	Sodium	1.00 mg	Vitamin B ₆	0.05 mg
Sugars	4.89 g	Zinc	0.14 mg	Folate	24.00 µg
Sucrose	0.47 g	Copper	0.048 mg	Choline	5.70 mg
Glucose	1.99 g	Manganese	0.39 mg	Vitamin A	1.00 µg
Fructose	2.44 g	Selenium	0.40 µg	Lutein + zeaxanthin	26.00 µg
Dietary fiber	2.00 g			Vitamin E	0.29 mg
				Vitamin K	2.20 µg

Source: Giampieri; Alvarez-Suarez; Battino (2014).

Strawberry has an attractive activity antioxidant. The antioxidant compounds contribute to the strawberry being classified as a functional food with various preventive and therapeutic health benefits, with positive effects on various diseases. The antioxidant power of strawberries is attributed to their concentration of polyphenols. The high concentration of these compounds in the strawberry makes it included among the 100 dietary sources richest in polyphenols and is classified among the foods that provide more than 1 mg of polyphenols per serving (BASU et al., 2014).

Anthocyanins belong to the group of flavonoids and are among the main polyphenols found in strawberries. Anthocyanins aid in the protection of the brain, liver and kidney, and act in the prevention of cardiovascular diseases, obesity control and the treatment of cancer. In addition to these beneficial health properties, anthocyanins are water-soluble pigments that give blue, purple and red colors, giving the product an attractive color (BASU et al., 2014; BENDOKAS et al., 2019).

Like the vast majority of fruits, strawberries have a high moisture content, high water activity and high physical fragility. These significantly contribute to increased perishability, by

favoring the development of microorganisms and oxidative reactions, especially when subjected to physical damage during harvesting, transportation and marketing, causing nutritional, sensory and economic losses (CHITARRA; CHITARRA, 2005). Strawberries, when stored at room temperature, have a very short shelf life of 1 to 2 days (MULEY; SINGHAL, 2020).

Several strawberry processing techniques have been studied, such as modified atmosphere packaging, active packaging, edible coatings and films and ultrasound (BAICU; POPA, 2018). In addition to these methods, strawberry drying is a good alternative for strawberry processing. Several methods have been used, such as osmotic dehydration (KOWALSKA et al., 2018), pulsed vacuum osmotic dehydration (CHENG et al., 2014), foam mat drying (GUAZI; LAGO-VANZELA; CONTI-SILVA, 2019; VIMERCATI et al., 2019), convective drying (MÉNDEZ-LAGUNAS et al., 2017), freeze-drying (CIURZYŃSKA et al., 2018), spray drying (PELLICER et al., 2019), microwave drying (GAMBOA-SANTOS; CAMPAÑONE, 2019) and infrared drying (ADAK; HEYBELI; ERTEKIN, 2017).

2.2 Osmotic dehydration

Osmotic dehydration (OD) is a unitary operation that consists of immersing the material, either in slices, cubes, or whole, in a concentrated (hypertonic) solution. During this process, mass transfers occur between the food and the osmotic solution, due to the osmotic pressure gradient between both systems. The main flows are the flow of water from the food into the solution and the incorporation of solids from the solution into the food. In addition to these two flows, a third can also occur simultaneously (Figure 1), but it is insignificant in quantitative terms. In this third flow, for example, leaching of solutes, such as sugars, organic acids, minerals and vitamins, occurs from the food to the solution. However, the leaching of these compounds can culminate in the loss of sensory and nutritional quality of the product (AHMED; QAZI; JAMAL, 2016; AKBARIAN; GHASEMKHANI; BRANCH, 2014; GONZÁLEZ-PÉREZ; RAMÍREZ-CORONA; LÓPEZ-MALO, 2021; RAMYA; JAIN, 2017).

The water flow occurs at a higher rate initially, becoming slower over time, as the difference in osmotic pressure decreases, reducing the driving force of the OD. Furthermore, the flow of water is quantitatively greater than that of solids, due to the vegetable tissue of the food being semipermeable and the small size of the water molecule (AKBARIAN; GHASEMKHANI; BRANCH, 2014).

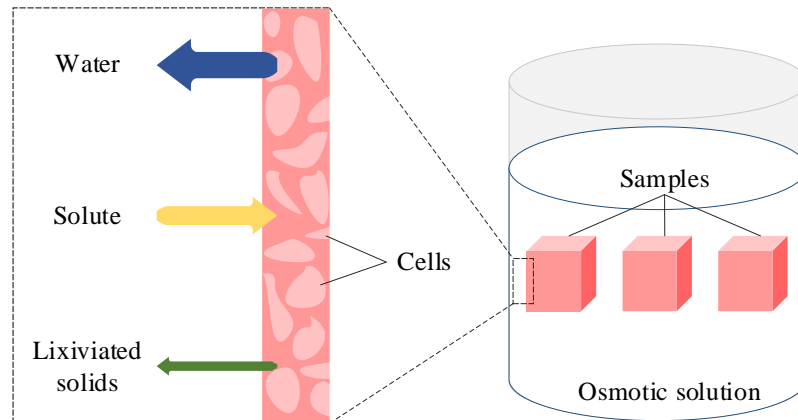


Figure 1 – Mass transfers between the product and the osmotic solution during osmotic dehydration.

During OD, water loss and solids gain increase exponentially. However, after a certain time, the flow rates decrease, continuing until equilibrium is reached (YADAV; SINGH, 2014). According to Ahmed; Qazi; Jamal (2016), mass transfers occur more quickly during the first two hours of OD. At the end of the osmotic process, the product still has an intermediate moisture content, between 65 and 75% (CHANDRA; KUMARI, 2015). For this reason, OD is used as a pre-treatment, as the amount of water removed from the food is not able to guarantee, on its own, the stability of the product for long periods. Therefore, it is recommended to use a drying method to complement the osmotic process (AHMED; QAZI; JAMAL, 2016; MACEDO et al., 2019, 2021b; RAMYA; JAIN, 2017).

The incorporation of solids can be conducted to add functional, nutritional, technological, or sensory properties to the product, to meet the growing consumer demand for foods with different characteristics. Moreover, by contributing to such properties, the enriched product has a greater added value (HUERTA-VERA et al., 2017; MACEDO et al., 2021b; RASCÓN et al., 2018). Isomaltulose (PEINADO et al., 2013), fructooligosaccharides (MACEDO et al., 2021b), polyols (MENDONÇA et al., 2017), honey (KHUBBER et al., 2020) are among these osmotic agents.

The mass transfers that occur during OD are influenced by several factors, whether related to the applied pre-treatment, the food, the osmotic solution and/or the process (CHANDRA; KUMARI, 2015; RAMYA; JAIN, 2017; SHETE et al., 2018; YADAV; SINGH, 2014).

The concentration of the osmotic solution is one of the main factors of OD, in which, generally, the more concentrated solution has a higher driving force, resulting in higher and faster transfers of water and solute. However, solutions with excessively high concentrations

become viscous, making it difficult for solutes to penetrate (AHMED; QAZI; JAMAL, 2016; RAMYA; JAIN, 2017; YADAV; SINGH, 2014).

OD is traditionally conducted at atmospheric pressure. However, the vacuum can be applied at the beginning of the process. This combination of vacuum and OD is called pulsed vacuum osmotic dehydration (PVOD). The application of vacuum causes the expansion of the food and, mainly, the removal of the air naturally present inside the material. Upon returning to atmospheric pressure, the osmotic solution penetrates the pores of the food, filling the volume previously occupied by the air, which leads to an increase in the contact area between the food and the solution. Therefore, this mechanism can decrease the time and increase the mass transfers of the osmotic process (CORRÊA et al., 2014; RAMALLO; HUBINGER; MASCHERONI, 2013; ŞAHIN; ÖZTÜRK, 2016).

2.3 Isomaltulose

6-O- α -D-glucopyranosyl-D Fructofuranose (C₁₂H₂₂O₁₁), known as isomaltulose and Palatinose[®], is a carbohydrate classified as a disaccharide, consisting of glucose and fructose (Figure 2), with a molecular weight of 360.32. Isomaltulose is an isomer of sucrose and was discovered in the 1950s, therefore considered as an emerging carbohydrate. It is naturally present in honey and sugar cane juice, however, in small quantities, making extraction difficult. Isomaltulose can be produced from sucrose, by an enzymatic isomerase process (LINA; JONKER; KOZIANOWSKI, 2002; SAWALE et al., 2017; SHYAM; RAMADAS; CHANG, 2018).

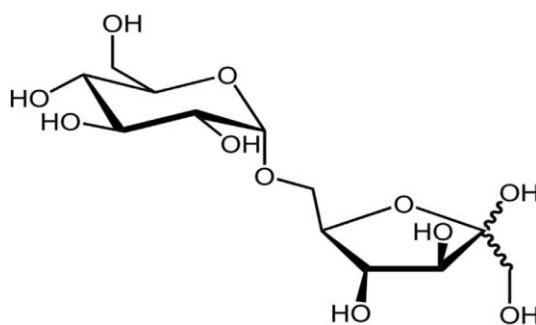


Figure 2 – Molecular structure of isomaltulose.

Source: Sawale et al. (2017).

The sweetness, caloric value and glycemic index of isomaltulose and other sugars are shown in Table 2.

Table 2 – Sweetness, caloric value (kcal g⁻¹) and glycemic index (GI) of different sugars

Sugar	Sweetness	Caloric value	GI
Glucose	0.5	4	100
Fructose	1.5-1.8	4	19-23
Sucrose	1	4	61-65
Erythritol	0.7	0.2	0
Xylitol	1	3	7-13
Maltitol	0.5-0.9	3	35-52
Lactitol	0.35-0.4	2.4	6
Sorbitol	0.6	2.6	9
Isomaltulose	0.3-0.4	4	32

Source: Sawale et al. (2017).

In the small intestine, isomaltulose is cleaved in a slower process compared to sucrose. As a result, it has a low glycemic index when compared to sucrose, which is the most traditional sugar in Brazil, and glucose, which is the base sugar for calculating the glycemic index. This characteristic is favorable to diabetics, due to the attenuation of blood glucose and insulin response, therefore being a good alternative in substitution to sucrose. Furthermore, the low glycemic index prolongs the feeling of satiety and promotes a balanced release of energy to the body, favoring physical and mental performance (LINA; JONKER; KOZIANOWSKI, 2002; SAWALE et al., 2017). Furthermore, isomaltulose has a caloric value similar to that of sucrose, fructose and glucose, of 4 kcal g⁻¹ (SAWALE et al., 2017).

Isomaltulose is considered “tooth-friendly”, as it has a low potential cariogenic index. This index is given by the acid production capacity from the oral fermentation of carbohydrates. The lactic acid produced by the fermentation of sugar acts in the demineralization of the dental tissues, causing dental caries, thus destroying the tooth. Resistance to isomaltulose fermentation is due to the stability of the α -1,6 bond between glucose and fructose monomers since this bond cannot be broken by most bacteria present in the mouth, especially in dental plaques. In sucrose, this bond is α -1,2 (LINA; JONKER; KOZIANOWSKI, 2002; SAWALE et al., 2017; SHYAM; RAMADAS; CHANG, 2018).

2.4 Food drying

As described in topic 2.2, osmotic dehydration is not able to reduce the moisture content of the product sufficiently to guarantee its stability for long periods. The use of a drying method

after osmotic dehydration is the best way to complement the reduction in moisture content (RAMYA; JAIN, 2017).

Drying is one of the most traditional methods in food preservation, to increase the shelf life of products, going from days or weeks to months or even years. Simultaneously, it contributes to the reduction of demands on packaging, transport and storage, implying cost reduction. Drying refers to a thermal process applied to remove moisture present in the food, converting solid, semi-solid, or liquid materials into solids with low moisture content. Removal is based on the difference in vapor pressure between the food and the surrounding air (KARAM et al., 2016a; MACEDO et al., 2020a, 2020b; OMOLOLA; JIDEANI; KAPILA, 2017).

Many drying methods have been developed over the years, where each method has been developed to meet the specific needs of each food. However, inevitably, regardless of the method, from the simplest and oldest, to the most sophisticated and current method, drying not only changes the moisture content of food but can also change physical, sensory and nutritional properties. In most cases, these changes are undesirable and irreversible, even after rehydration (KARAM et al., 2016a; OMOLOLA; JIDEANI; KAPILA, 2017; SAGAR; SURESH KUMAR, 2010). However, many studies have been developed to minimize these changes, to maintain the beneficial properties of the pre-drying product (ARAÚJO et al., 2020; KARAM et al., 2016b; MACEDO et al., 2020a, 2020b, 2021b).

2.4.1 Drying with ethanol pre-treatment

Pre-treatments applied to fruits before drying generally aim to improve the drying process and with the preservation of compounds and characteristics of the food (WANG et al., 2019). Among these pre-treatments, the addition of low-boiling organic solvents, such as ethanol, isopropanol and ethyl acetate, has been carried out on the wet material by immersion in a solution with the organic solvent (ROJAS; AUGUSTO, 2018a, 2018b; WANG et al., 2019) or dripped/spread under the surface of the food (ARAÚJO et al., 2020; CORRÊA et al., 2012; MACEDO et al., 2021a; SILVA; CELEGHINI; SILVA, 2018) or even spraying the organic solvent during drying, changing the composition of the atmospheric air (BRAGA et al., 2009, 2010; SANTOS; SILVA, 2009).

Ethyl alcohol or ethanol is an organic compound of molecular formula $\text{CH}_3\text{CH}_2\text{OH}$, belonging to the family of alcohols, presenting a boiling temperature of 78 °C. This compound has been the most used solvent with as a pre-treatment to convective drying, as it presents, mainly, low toxicity and low cost (MACEDO et al., 2021a). The U.S. Food and Drug Administration considers ethanol a GRAS (generally recognized as safe) substance (FDA, 2019).

The addition of ethanol acts on four main aspects: reducing the heat of water vaporization; the opening of channels; increasing permeability of cellular membrane; and the Marangoni effect (CORRÊA et al., 2012; FUNEBO et al., 2002; MACEDO et al., 2021a; ROJAS; AUGUSTO, 2018b; SANTOS et al., 2021).

- i. The addition of ethanol reduces the heat of vaporization of the water present in the food, due to the mixture of the water+ethanol formed. As a result, water evaporation is increased;
- ii. The rapid evaporation of ethanol and the ethanol+water mixture forms flow channels and pores in the food, facilitating the displacement of pure water from inside the product to the drying air;
- iii. Ethanol increases the permeability of the cell wall of the food, both in the sense of dehydration and for rehydration, since the use of ethanol causes structural changes in the tissue, promoting the dissolution and disorganization of the cell wall compounds;
- iv. The Marangoni effect promotes the transfer of mass at an interface between two fluids of different surface tension. Water has a higher surface tension than ethanol. In food pre-treated with ethanol, the surface tension gradient causes the ethanol, when it evaporates first, to increase the displacement of the water.

Studies have shown several beneficial effects of pre-treatment with ethanol, both in the process and in the characteristics of the dried product. The reduction of drying time is the main contribution of ethanol in the process. Studies have observed reductions between 10% and 60% in drying time with the inclusion of this pre-treatment (MACEDO et al., 2021a; ROJAS; AUGUSTO, 2018b; ROJAS; AUGUSTO; CÁRCEL, 2020; SANTOS et al., 2021). Reducing process time decreases energy consumption, reducing equipment usage time and labor time.

Reducing the drying time reduces the exposure of the material to drying conditions, such as high temperatures. As a result, less degradation of thermosensitive compounds has been observed, such as minimizing the color change (CORRÊA et al., 2012), decreased shrinkage (SILVA; CELEGHINI; SILVA, 2018), reduced degradation of ascorbic acid (SANTOS; SILVA, 2009; WANG et al., 2019), cumarin (SILVA; CELEGHINI; SILVA, 2018), volatile compounds (BRAGA et al., 2009, 2010), higher aromas retention (WANG et al., 2019), increased preservation of antioxidant compounds (ARAÚJO et al., 2020) and better rehydration (WANG et al., 2019).

2.4.2 Intermittent microwave drying

The term “microwave” refers to electromagnetic waves in the frequency range 300 MHz to 300 GHz with a wavelength from 1 mm to 1 m. Microwave drying is gaining increasing interest in food drying in recent years. This drying method is based on a unique volumetric heating mode facilitated by electromagnetic radiation at 915 or 2450 MHz. The waves penetrate the material, causing agitation of the molecules and, consequently, heating is generated throughout the product, producing steam, which moves due to the internal pressure gradient (FENG; YIN; TANG, 2012; KUMAR; KARIM, 2019).

The heating generated by microwaves occurs mainly by two mechanisms: dipolar reorientation and ionic conduction. In the first, the dipolar molecule, like water, tries to orient itself through the electric field. However, the electric field generated by the microwave changes direction 2.45 billion times per second, causing agitation of the molecules, generating friction and heating inside the product, especially in materials with high moisture content, such as fruits. In the second mechanism, the ions present in the food move according to the electric field, generating heating. However, in food, the heating generated by the agitation of the molecules is more expressive than that generated by the ions (FENG; YIN; TANG, 2012; KUMAR; KARIM, 2019).

Microwave drying features reduced processing time, low energy consumption, precise process control and uniform heating (DA COSTA et al., 2021; FENG; YIN; TANG, 2012; JUNQUEIRA; MENDONÇA; CORRÊA, 2016; KUMAR; KARIM, 2019). Furthermore, because microwaves mainly heat the wet areas of the food, this method can preserve quality characteristics, such as color, density, rehydration capacity, texture and nutritional parameters (CORRÊA et al., 2011; FENG; YIN; TANG, 2012; GUCLU et al., 2021; İZLI; POLAT, 2020). Another highlight of this drying method is that bound water molecules which are difficult to remove can also be excited by microwaves, facilitating their removal (KUMAR; KARIM, 2019).

Continuous exposure to microwaves can cause the product to overheat, leading to the degradation of various compounds or even the burning of the product. Thus, the batch process is called intermittent microwave drying (IMD) and has been an interesting alternative for drying food. In this process, the magnetron is turned on and off periodically, allowing moisture and temperature to be evenly distributed during drying, preventing overheating of the food. IMD has less energy consumption than convective drying and its use is associated with the production of dehydrated foods of good physical, nutritional and chemical qualities (CAO et

al., 2019; GUCLU et al., 2021; HUANG et al., 2021; JUNQUEIRA; CORRÊA; ERNESTO, 2017; MOUNIR et al., 2020; TEPE; TEPE, 2020).

Cao et al. (2019) observed that the inclusion of intermittent microwaves during vacuum drying of litchi fruits saved at least 31% of the energy needed, reduced the product browning and increased the sensory acceptance of the dry product. Guclu et al. (2021) observed that IMD was more effective in creating new aroma compounds in sweet red peppers than infrared and hot air drying.

3 GENERAL CONSIDERATIONS

The high perishability of strawberries requires processing methods to be used, aiming to extend the shelf life and also preserve the qualitative parameters of the product. One of the ways to process the strawberry is subjecting it to osmotic dehydration, immersing the fruit in a hypertonic solution. Simultaneously with the reduction of the moisture content, during the osmotic process, there is also the incorporation of solute, making osmotic dehydration a simple and low-cost method for fruit enrichment. The most used solute for the preparation of this solution is sucrose. However, some undesirable characteristics are linked to this solute. Isomaltulose appears as an alternative to sucrose, as it is a carbohydrate with low glycemic and cariogenic indexes.

Osmotic dehydration is not able to significantly extend the shelf life of the product. For this reason, it is generally used as a pre-treatment to some drying method. Drying, especially the method using heated air, stands out among the various food processing techniques. Ethanol pre-treatment can help reduce drying time and retain antioxidant compounds in fruits, simply and inexpensively. In this same context, microwave drying has stood out for its short process time, low energy consumption, and good preservation of the qualitative parameters of the product.

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SECOND SECTION**ARTICLE 1 – THE IMPACT OF USING VACUUM AND ISOMALTULOSE AS AN OSMOTIC AGENT ON MASS EXCHANGE DURING OSMOTIC DEHYDRATION AND THEIR EFFECTS ON QUALITATIVE PARAMETERS OF STRAWBERRIES**

Running title: Osmotic dehydration using isomaltulose

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Abstract

The impregnation of isomaltulose is an interesting strategy for the enrichment of strawberries since it is a carbohydrate with low glycemic and cariogenic indexes. The osmotic enrichment of strawberries with isomaltulose was performed considering, or not, the vacuum pulse in the first 10 and 20 min in a total process time of 300 min, at 25°C. The tested solution concentrations were 25 and 35% (w w⁻¹) of isomaltulose. The kinetics of solid gain, water loss, moisture content and weight reduction were obtained. The fresh and final products had some physics and nutritional parameters evaluated. Osmotic processes resulted in a reduction of moisture content, water activity, acidity, total anthocyanins and phenolic contents and antioxidant capacity, increased pH, influenced colorimetric parameters and caused shrinkage of the samples. The higher impregnation of palatinose was obtained with the vacuum pulse, which was 82% higher than the osmotic process without vacuum pulse. The atmospheric process resulted in higher bioactive compounds levels and lower shrinkage and lower total color difference.

Keywords: Osmotic dehydration; Mass transfer kinetics; Shrinkage; Antioxidant activity; Bioactive compounds.

Practical application

Osmotic dehydration is a unitary operation that promotes solid incorporation into the food and water loss from the food, which makes this process an interesting pre-treatment to pre-dehydrate and enrich fruits, especially when using solutes with beneficial properties to the organism, such as isomaltulose. The vacuum application during the first minutes is a simple and low-cost procedure, able of increasing mass exchanges during the osmotic process. However, important qualitative changes occur during the osmotic process.

1 INTRODUCTION

Isomaltulose (6-O- α -D-glucopyranosyl-D-fructose), commercially known as palatinose, is a reducing disaccharide with a low glycemic index, since the release of monosaccharides by isomaltulose occurs slowly, due to its difficulty to be hydrolyzed by gastrointestinal enzymes (Lina et al., 2002; Sawale et al., 2017; Shyam et al., 2018). Thus, glycemic spikes are minimized, which is a beneficial property for human health, especially for diabetic patients, besides prolonging the satiety period (Sawale et al., 2017). Furthermore, isomaltulose has a low cariogenic index, contributing to dental health. Compared to sucrose, isomaltulose has equal caloric value and less sweetening power. Due to these characteristics, isomaltulose is considered a healthy sweetener, attracting interest of food manufacturers seeking innovation (Lina et al., 2002; Sawale et al., 2017; Shyam et al., 2018).

Isomaltulose impregnation is an interesting strategy to promote fruit enrichment, since there is a great demand from consumers for healthy and functional foods, especially fruit, due to the growing concern with health and well-being. As a result, food is no longer consumed only to satisfy hunger (Betoret et al., 2011; Peinado et al., 2013; Sawale et al., 2017; Tylewicz et al., 2020). Among the fruits, strawberries are a good source of fibers, minerals, vitamins and bioactive compounds, such as anthocyanins. All these offer several health benefits, such as reduction of oxidative, inflammatory, hyperlipidemic, hypertensive or proliferative effects (Basu et al., 2014), besides presenting a high sensory appreciation, especially for the attractive aroma, flavor, color and appearance (Amami et al., 2017; Basu et al., 2014; Giampieri et al., 2014; Tylewicz et al., 2020). Such characteristics make the strawberry one of the most produced and consumed fruits in the world (Basu et al., 2014). The development of new products with functional properties is usually an expensive process (Betoret et al., 2011). However, the present study proposed for the first time the impregnation of isomaltulose in strawberries by vacuum application on osmotic dehydration (OD).

OD is a unitary operation that consists of immersing the food in a solution with higher osmotic pressure, with mass transfers between the osmotic solution and the food, due to the osmotic pressure gradient. In addition to the effect of reducing the moisture content, there is also the impregnation of solids from the solution into the food. One of the advantages of OD is the lack of sophisticated technologies and equipment, making it a simple and low-cost method for promoting food enrichment (Corrêa et al., 2010; de Mello et al., 2019; Grajales-Lagunes et al., 2019; Macedo et al., 2020; Mendonça et al., 2017; Ramya & Jain, 2017; Şahin & Öztürk, 2016). However, the loss of water-soluble compounds can occur, such as anthocyanins, due to the time of immersion in the osmotic solution (Ahmed et al., 2016; Ramya & Jain, 2017).

Several techniques can be combined with OD, aiming to increase solid impregnation and water loss, in addition to reducing the time necessary for the completion of the process (Ramya & Jain, 2017). The pressure reduction in the first minutes of OD, usually up to 20 min (Ahmed et al., 2016; Vieira et al., 2012), followed by the resumption of atmospheric pressure, promotes the filling of the spaces occupied by the air inside the food with the osmotic solution. Thereby, the contact area between the osmotic solution and the food is increased, favoring mass transfer rates (Betoret et al., 2011; de Jesus Junqueira et al., 2018; de Mello et al., 2019; Mendonça et al., 2017; Ramya & Jain, 2017). Therefore, this work aimed to study the effect of the isomaltulose solution concentration and the vacuum pulse time on OD and physicochemical and bioactive characteristics of strawberry.

2 MATERIALS AND METHODS

2.1 Preparation of the samples

Fresh strawberries (*Fragaria x ananassa* cv. Camino Real) were purchased from the local market, selected for ripeness degree (predominantly red fruits), integrity and size, rinsed in running tap water, sanitized with chlorinated water (200 ppm for 10 min), rinsed again and drained. The strawberries were stored at 4°C for up to 2 days. The skin was then removed and the strawberry was cut into 10 mm cubes.

2.2 Osmotic dehydration (OD) and pulsed vacuum osmotic dehydration (PVOD)

Isomaltulose (Beneo-Palatinit, Mannheim, Germany) solutions were prepared in concentrations of 25 and 35% (w w⁻¹) (Table 1), by dissolving the solute in deionized water. Isomaltulose has low water solubility, making it impossible to use high concentration osmotic solutions.

OD and PVOD were performed by immersing the strawberry cubes in the osmotic solutions, in the proportion of 1:20 (w w⁻¹), to avoid significant dilution of the medium. For PVOD treatments, the vacuum pressure (160 mbar) was applied in the first 10 or 20 min (Table 1), since these vacuum pulse times are generally used in osmotic processes (Ahmed et al., 2016; Vieira et al., 2012). Then, atmospheric pressure was resumed. OD was performed under atmospheric pressure throughout the process. OD and PVOD were carried out in a temperature-controlled oven (Solab SL104/40, Piracicaba, Brazil) at 25°C, until completing the total time of 300 min.

Table 1 – Levels of osmotic solution concentration and vacuum pulse time

Sample code	Concentration (% w w ⁻¹)	Vacuum time (min)
OD25	25	0
OD35	35	0
PVOD25-10	25	10
PVOD35-10	35	10
PVOD25-20	25	20
PVOD35-20	35	20

OD: osmotic dehydration; PVOD: pulsed vacuum osmotic dehydration.

At 20, 40, 60, 90, 120, 180, 240 and 300 min, the samples were removed from the osmotic solution, immersed in ice water for 10 s to stop the osmotic process. The sample surfaces were then drained with absorbent paper.

2.3 Mass transfer

The solid gain (SG), water loss (WL) and weight reduction (WR), were calculated according to Equations (1), (2) and (3).

$$SG(\%) = \frac{W_t(1-M_t) - W_0(1-M_0)}{W_0} \times 100 \quad (1)$$

$$WL(\%) = \frac{W_0M_0 - W_tM_t}{W_0} \times 100 \quad (2)$$

$$WR(\%) = \frac{W_0 - W_t}{W_0} \times 100 \quad (3)$$

Where W is the weight of the sample (kg); M is the moisture content of the sample (kg water kg sample⁻¹); the sub-indexes “0” and “t” indicate the initial and t times, respectively.

2.4 Characterization of samples

2.4.1 Moisture content (M)

The M of the samples was determined by the gravimetric method, at 70°C under vacuum (AOAC, 2010), according to method 934.06.

2.4.2 Water activity (a_w)

The a_w of the samples was determined by an electronic hygrometer (Aqualab, series 3TE, Washington, USA), at 25°C (Macedo et al., 2020).

2.4.3 Shrinkage

The fresh and osmotically dehydrated samples dimensions were measured at three points on each axis (x, y, z) of the cube. The measurements were performed with the aid of a digital Vernier caliper (± 0.01 mm) (Western, DC-60 model, Zhejiang, China) (Şahin & Öztürk, 2016). The volume was calculated by the product of the average of the measurements. Shrinkage was calculated according to Equation (4) (Junqueira, Corrêa, et al., 2017).

$$\text{Shrinkage} = 1 - \frac{V}{V_0} \quad (4)$$

Where V and V_0 are the volumes (m^3) after and before OD, respectively.

2.4.4 pH and acidity

The pH determination of the samples was carried out using a digital pH meter (Tecnal, TEC-5, Piracicaba, Brazil). Acidity was determined by titration of sodium hydroxide, up to pH 8.20 (Araújo et al., 2020). The result was expressed in %, kg of citric acid per 100 kg of sample on a dry basis.

2.4.5 Color

The color determination of the samples was performed by direct reading using a colorimeter (Konica Minolta, model CR-400, Osaka, Japan), with illuminant D65, using color scale CIELab. The parameters L^* , a^* , b^* , C^* and h° were obtained. The total color difference (ΔE) was calculated according to the Equation (5) (Pathare et al., 2013). considering the control as the fresh sample, whose parameter is represented by the sub-index “0”.

$$\Delta E = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2} \quad (5)$$

2.4.6 Total anthocyanins content (TAC)

TAC was quantified according to the differential pH method, according to Equation (6) (Giusti & Wrolstad, 2001).

$$\text{TAC} \left(\frac{\text{mg}}{100\text{g}} \right) = \left(\frac{((A_{510 \text{ nm}} - A_{700 \text{ nm}})_{\text{pH}1} - (A_{510 \text{ nm}} - A_{700 \text{ nm}})_{\text{pH}4.5}) \times \text{MW} \times \text{DF}}{\epsilon \times L} \right) \quad (6)$$

Where MW is the molecular weight of cyanidin-3-glycoside (449.2 g mol^{-1}); DF is the dilution factor; ϵ is the molar absorptivity (26900 mol^{-1}); L is the path length in centimeters.

2.4.7 Total phenolics content (TPC)

TPC was determined by the spectrophotometric method using the Folin-Ciocalteu method, as described by Waterhouse (2003). TPC was expressed in mg of gallic acid per 100 g on a dry basis.

2.4.8 Antioxidant capacity

The antioxidant capacity of the samples was determined by the ferric reducing antioxidant power capacity (FRAP) and 2,2-diphenyl-1-picrylhydrazyl (DPPH) methods. The results were expressed in μmol of ferrous sulphate per g of sample, on a dry basis, and g sample per g of DPPH (Rufino et al., 2010).

2.5 Statistical analysis

The experiment was carried out in a completely randomized design, in a complete factorial scheme 2x3 (Table 1), with five replications.

The data were submitted to analysis of variance (ANOVA) and then the Tukey test for comparison among treatments. The Dunnett test was used to compare each treatment individually with fresh fruit.

A multivariate analysis was conducted using principal component analysis (PCA) to correlate the mass transfer responses after osmotic processes, moisture content, water activity, shrinkage, pH, acidity, color parameters, total anthocyanins content, total phenolics content, and antioxidant capacity by FRAP and DPPH methods responses with concentration of osmotic solution (25 or 35%) and vacuum pulse time (0, 10 or 20 min).

Statistical analyzes were carried out at the level of 5% probability of error and with the aid of the Statistica software (StatSoft Inc., Tulsa, OK, USA).

3 RESULTS AND DISCUSSION

3.1 Mass transfer

The kinetics of solid gain (SG), water loss (WL), weight reduction (WR) and moisture content (M) during osmotic dehydration (OD) and pulsed vacuum osmotic dehydration (PVOD) are shown in Figure 1.

SG, WL and WR increased over time, especially in the first minutes (Figures 1a to 1c). Moreover, higher SG, WL and WR values were obtained with the highest osmotic solution (OS) concentration, as also observed by Corrêa et al. (2010), Ferrari et al. (2011) and de Mello et al. (2019). This is due to the higher osmotic pressure gradient between the most concentrated OS

and the food (de Mello et al., 2019). It was also observed that the M decreased along with the OD and PVOD (Figure 1d), due to the increase in WL and SG.

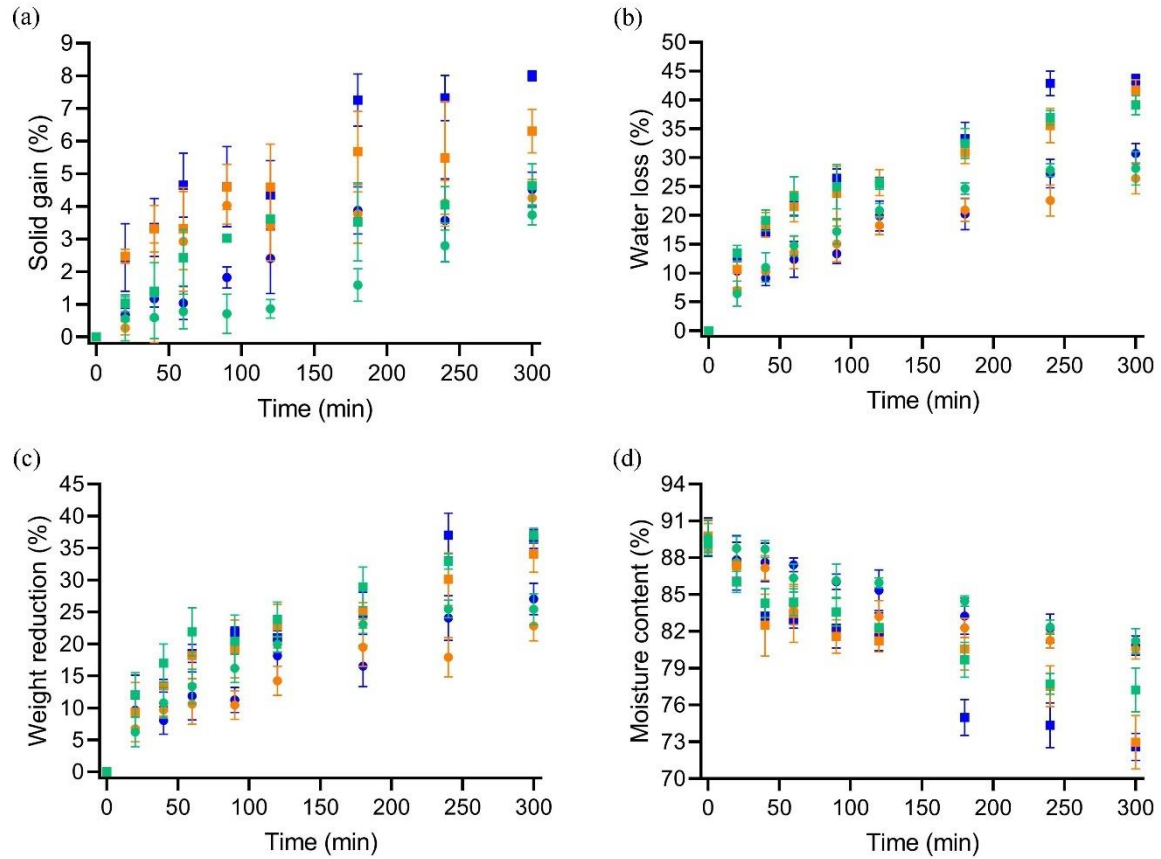


Figure 1 – Solid gain (a) water loss (b), weight reduction (c) and moisture content (d) during OD and PVOD.

Green circle: OD25; green square: OD35; orange circle: PVOD25-10; orange square: PVOD35-10; blue circle: PVOD25-20; blue square: PVOD35-20. The bar represents the standard deviation.

In addition to the OS concentration factor, the treatments with pulsed vacuum showed a trend of higher SG, WL and WR values (Figures 1a to 1c) and lower value of M (Figure 1d) during the osmotic processes, especially using vacuum pulse time (VPT) of 20 min. The use of vacuum causes the ejection of occluded air in the strawberry, followed by the occupation of these sites by the OS, favoring the contact between the OS and the food (Corrêa et al., 2016).

The comparison among treatments for SG, WL and WR in the final time (300 min) is shown in Figure 2. The SG, WL and WR were between 3.73 and 8.00%, 28.15 and 43.53%, 25.43 and 36.96%. The osmotic solutions concentrations used in the present study were lower than those commonly used in other studies, due to the low solubility of isomaltulose compared to other carbohydrates. Even so, similar results were found in several other studies, such as in

Castelló et al. (2006) using glucose solutions from 15 to 30°Brix; Mendonça et al. (2017) using solutions of xylitol, maltitol, erythritol, isomalt and sorbitol; and Corrêa et al. (2016) and Ferrari et al. (2011) using sucrose solutions.

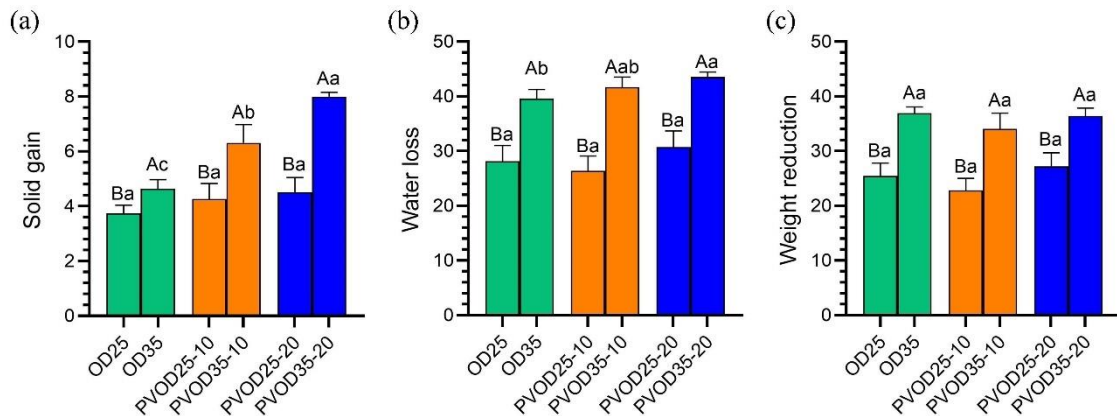


Figure 2 – Solid gain (a) water loss (b) and weight reduction (c) after 300 min OD and PVOD. The same uppercase letter indicates no significant difference between the concentrations of the osmotic solution in the same vacuum pulse time; and the same lowercase letter indicates no significant difference between vacuum pulse times at the same concentration as the osmotic solution, by the Tukey test ($p < 0.05$).

The 35% OS resulted in higher SG for both OD samples and those treated with pulsed vacuum, for both times (10 and 20 min) (Figure 2a). Furthermore, for the OS of 35%, the SG was higher with the increase in the VPT, reaching a maximum response (8.00%) by applying the PVOD35-20 treatment, the best treatment to enrich the strawberry with isomaltulose.

The highest OS concentration resulted in a higher WL at the end of the processes (Figure 2b), as also reported by Amami et al. (2017) in OD of strawberry using commercial sugar. The vacuum pulse application for 20 min resulted in an increased WL compared to treatment at atmospheric pressure, using a 35% OS. However, using the 25% OS, although the vacuum pulse increases the contact between the food and the OS, non-significant differences were observed for WL, as for SG (Figures 2a and 2b).

The WR was influenced only by the concentration of the OS, where the OS with the highest concentration resulted in the highest WR. However, the application of vacuum pulse did not significantly influence WR for both tested concentrations (Figure 2c).

3.2 Characterization of the samples

3.2.1 Moisture content (M)

The fresh strawberry showed high M, of 89.39 kg of water 100 kg of sample⁻¹. Similar values were found by Amami et al. (2017), Giampieri et al. (2014) and Basu et al. (2014).

All studied treatments were effective in reducing M (Figure 3a). Samples submitted to treatments with 35% OS showed lower responses than those with 25%. This is due to the fact that the higher OS concentration has a higher osmotic pressure gradient in relation to the product, increasing the driving force of the migration of water from food to the OS (de Mello et al., 2019). This can be seen by the strong correlation between M and WL, and between M and SG, due to the increase in the dry matter content of the product, reducing the M.

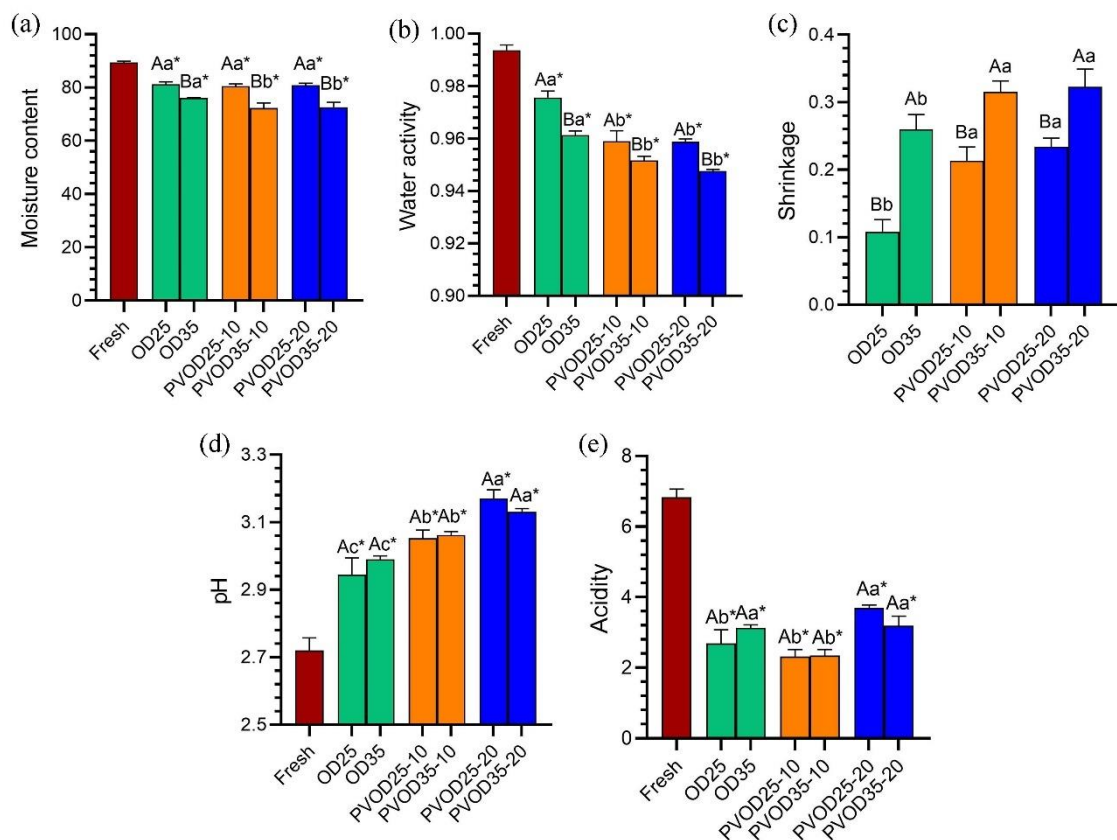


Figure 3 – Moisture content (a), water activity (b), shrinkage (c), pH (d), acidity (e) and hardness (f) of fresh and osmotically dehydrated strawberries.

The same uppercase letter indicates no significant difference between the concentrations of the osmotic solution in the same vacuum pulse time; and the same lowercase letter indicates no significant difference between vacuum pulse times at the same concentration as the osmotic solution, by the Tukey test ($p < 0.05$); The asterisk indicates a significant difference between each treatment and the fresh sample, by the Dunnett test ($p < 0.05$).

In both VPTs, the application of vacuum pulse reduced the moisture content of the samples when using the OS by 35%. However, for the 25% OS, the use of vacuum did not

influence the moisture content of the samples. The treatments PVOD35-10 and PVOD35-20 resulted in the lowest moisture content.

3.2.2 Water activity (a_w)

The fresh strawberry presented a_w of 0.994 (Figure 3b). Similar results were found by Dermesonlouoglou et al. (2016), Garcia-Noguera et al. (2014), and Tylewicz et al. (2020). This value makes the fresh strawberry susceptible to microbial deterioration (Núñez-Mancilla et al., 2014), which gives the strawberry high perishability and highlights the need for using conservation methods. The osmotically dehydrated strawberries showed a_w lower than the fresh strawberry, between 0.948 and 0.976. Thus, although the osmotic process is not able to exclusively guarantee microbiological stability (Corrêa et al., 2016), the combination of vacuum pulse with the highest concentration OS contributed to fruit conservation. Castelló et al. (2006) performed OD and PVOD of strawberry using glucose OS with a concentration of 15 to 30°Brix, obtaining similar values to those found in the present study, since they used OS with a concentration below that commonly used (40 to 60°Brix) (Ramya & Jain, 2017), as well as the present study.

The highest concentration OS resulted in samples with lower a_w , due to the higher WL and SG during OD (de Mello et al., 2019). The same was observed by de Mello et al. (2019), Corrêa et al. (2010) and Castelló et al. (2006). The a_w reduction showed a correlation WL, reducing the proportion of free water, and with SG, as SG increases the dry matter content in the product, increasing the interaction between water and material.

The use of the vacuum pulse reduced a_w for both OS, since the vacuum pulse allows the OS to fill the places occupied by the occluded air, increasing the area of contact between the OS and the food, promoting greater WL, which resulted in samples with lower a_w (Castelló et al., 2006; Corrêa et al., 2010). The VPT did not significantly influence a_w , for both OS concentrations. The treatments PVOD35-10 and PVOD35-20 resulted in samples with lower a_w .

3.2.3 Shrinkage

The shrinkage of the samples was between 0.11 and 0.31 (Figure 3c). It was influenced by the OS concentration and the VPT. Similar results were found by Junqueira et al. (2017). Among the same VPTs, the concentration of 35% resulted in higher shrinkage compared to treatments at 25%. In addition, treatments using PVOD resulted in higher shrinkage than OD,

as also observed by de Jesus Junqueira et al. (2018a). For both concentrations, increasing the VPT from 10 to 20 min did not change the sample shrinkage.

Shrinkage is a response strongly related to mass transfers that occur during osmotic processes. Junqueira et al. (2017) observed a reduction in the thickness of sweet potatoes after OD, attributing this change to WL during the osmotic process, since, as in the present study, WL is the main flow in mass transfer, in quantitative terms.

During osmotic processes, water and solids flow between the OS and the material. The water output is quantitatively greater than the incorporation of solids, causing the food matrix to move towards space previously filled with water, causing the material to shrink (Nahimana et al., 2011).

Strawberry has a porous structure (Amami et al., 2017). This characteristic makes food more susceptible to shrinkage, especially when applying the vacuum pulse, due to the intensification of mass transfer during the osmotic process (Junqueira et al., 2020).

Although it is an important response in dehydrated foods {Formatting Citation}, few studies have evaluated shrinkage in foods subjected to osmotic dehydration, hindering comparison with other studies. It is interesting to note that the shrinkage was almost isotropic, that is, the sample volume reduction occurred similarly in all directions (x, y and z axes).

3.2.4 pH and acidity

The fresh strawberry presented a pH of 2.72 (Figure 3d) and acidity of 6.83% (Figure 3e), showing the typical acid characteristic of fresh strawberries. All treatments were responsible for increasing the pH value and reducing acidity in relation to fresh strawberries. Reduced acidity contributes to improve taste and increase sensory acceptance of strawberries (Resende et al., 2008), increasing the consumption of fruit rich in several bioactive compounds and enriched with isomaltulose. The increase in pH and the reduction in acidity are due to the leaching of H⁺ ions and organic acids from the product to the OS. de Jesus Junqueira et al. (2017) observed greater potassium leaching using vacuum pulse in relation to OD.

The concentration of the OS did not influence the pH and acidity of the samples (Figures 3d and 3e). However, the increase in the VPT increased the pH at all concentrations of the OS (Figure 3d). Possibly, the VPT increased the leaching of H⁺ ions to OS. Regarding acidity, the time of 20 min presented the highest value among OS, 25%. For the 35% solution, the PVOD35-20 treatment did not differ from the OD35, with the lowest acidity value observed for the PVOD35-10 treatment.

3.2.5 Color

The samples submitted to OD25 and OD35 showed L^* statistically equal to the fresh sample (Figure 4a). The other samples showed lower L^* . The use of OS 25 and 35% did not influence the L^* of the samples. On the other hand, PVOD reduced L^* in relation to OD, for both concentrations of OS.

The samples treated by OD25, PVOD35-10 and PVOD35-20 showed a^* similar to that of the fresh sample (Figure 4b). The OS of 35 resulted in samples with a higher a^* , indicating more redness of the strawberry. In addition, PVOD, both 10 and 20 min, resulted in a lower a^* . The a^* parameter is an important quality attribute in strawberries since it can correlate with the concentration of anthocyanins, which are important red pigments found in strawberries (Giampieri et al., 2014; Pathare et al., 2013).

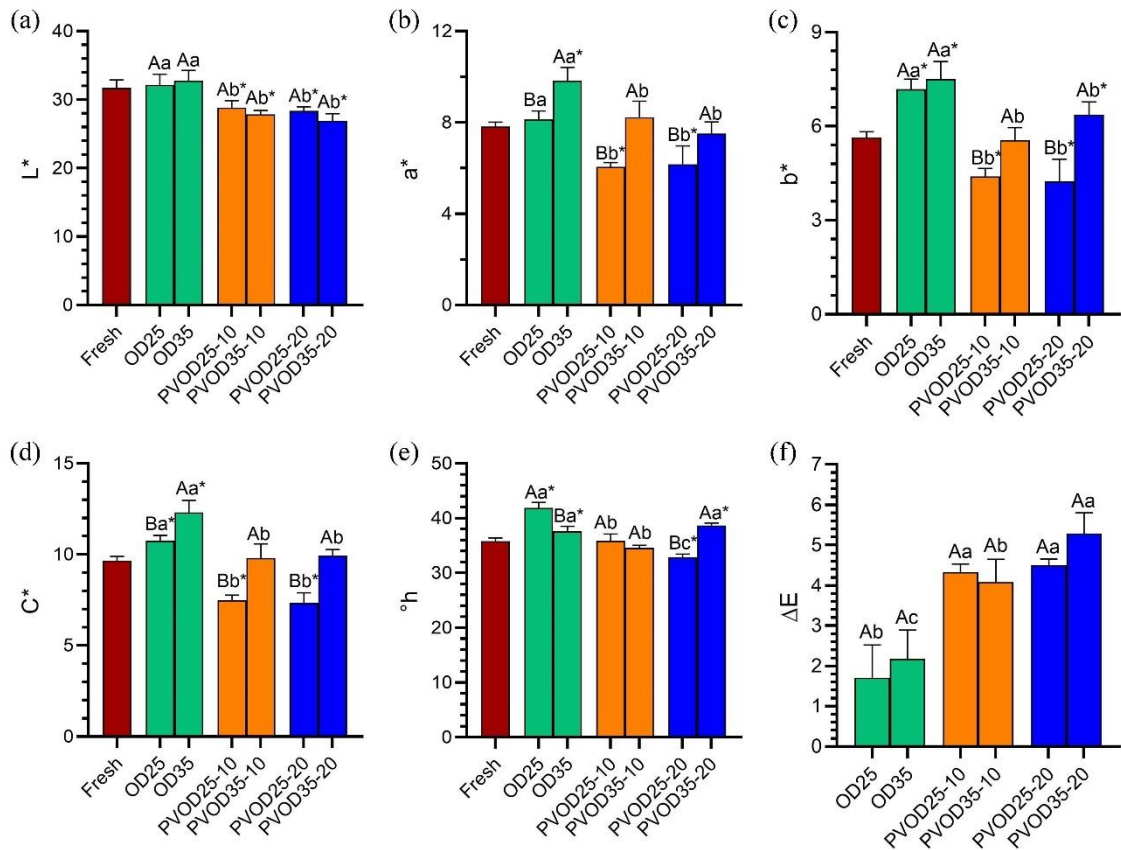


Figure 4 – Colorimetric parameters (L^* (a), a^* (b), b^* (c), C^* (d), $^{\circ}h$ (e) and ΔE (f)) of fresh and osmotically dehydrated strawberries.

The same uppercase letter indicates no significant difference between the concentrations of the osmotic solution in the same vacuum pulse time; and the same lowercase letter indicates no significant difference between vacuum pulse times at the same concentration as the osmotic solution, by the Tukey test ($p < 0.05$); The asterisk indicates a significant difference between each treatment and the fresh sample, by the Dunnett test ($p < 0.05$).

The PVOD35-10 treatment was the only one with a b^* value equal to that of the fresh sample (Figure 4c). Between the OSs, OD did not influence b^* . However, the application of the vacuum pulse (10 or 20 min) caused the OS of 35% to present a higher b^* .

The C^* indicates the color intensity perceived by humans (Pathare et al., 2013). OD25 and OD35 resulted in a higher C^* than the fresh sample (Figure 4d). The treatments PVOD25-10 and PVOD25-20 resulted in lower C^* than the fresh sample. Samples submitted to the treatments PVOD35-10 and PVOD35-20 presented C^* equal to the fresh sample. The 35% OS produced strawberries with a higher C^* than the 25% OS. In addition, the application of vacuum pulse (10 or 20 min) reduced C^* compared to OD treatments.

The term $^{\circ}h$ represents the shade of the product. The closer to 0° (or 360°), the greater the red hue, which is desirable in strawberries. Therefore, $^{\circ}h$ is an important color attribute for fruit evaluation (Pathare et al., 2013). The PVOD treatments for 10 min did not influence the $^{\circ}h$ in relation to the fresh sample. The PVOD25-20 treatment reduced the $^{\circ}h$. The other treatments increased the $^{\circ}h$, reducing the red hue. Using the 25% OS, the increase in the VPT reduced the $^{\circ}h$. On the other hand, with the OS of 35%, 10 min of vacuum pulse had the lowest $^{\circ}h$.

The treated samples showed values of total color difference (ΔE) between 1.71 to 5.29. According to Pathare et al. (2013), $1.5 < \Delta E$, $1.5 < \Delta E < 3$ and $\Delta E > 3$ indicate a small difference, distinct, and very distinct, respectively, as to perceivable color. The ΔE was not influenced by the concentration of the OS. However, OD samples showed lower ΔE than PVOD. At for the OS of 25%, the increase in the VPT from 10 to 20 min did not affect the ΔE . However, it increased the ΔE using the OS by 35%.

3.2.6 Total anthocyanins content (TAC)

The fresh strawberry showed a TAC of $14.565 \text{ mg } 100\text{g}^{-1}$ (Figure 5a). This value is lower than those presented in the literature (Zeliou et al., 2018) because the peripheral part of the fruits, that presents more TAC, was removed in the samples obtainment. This procedure was important for the diffusive phenomena of impregnation.

Compared to fresh strawberries, OD and PVOD significantly reduced TAC (Figure 5a). Anthocyanins are water-soluble compounds (Krga & Milenkovic, 2019). Therefore, anthocyanins are easily leached from strawberry to OS, due to WL during the osmotic process. As a result, treatments using higher concentration OS and the application of vacuum pulse resulted in lower TAC.

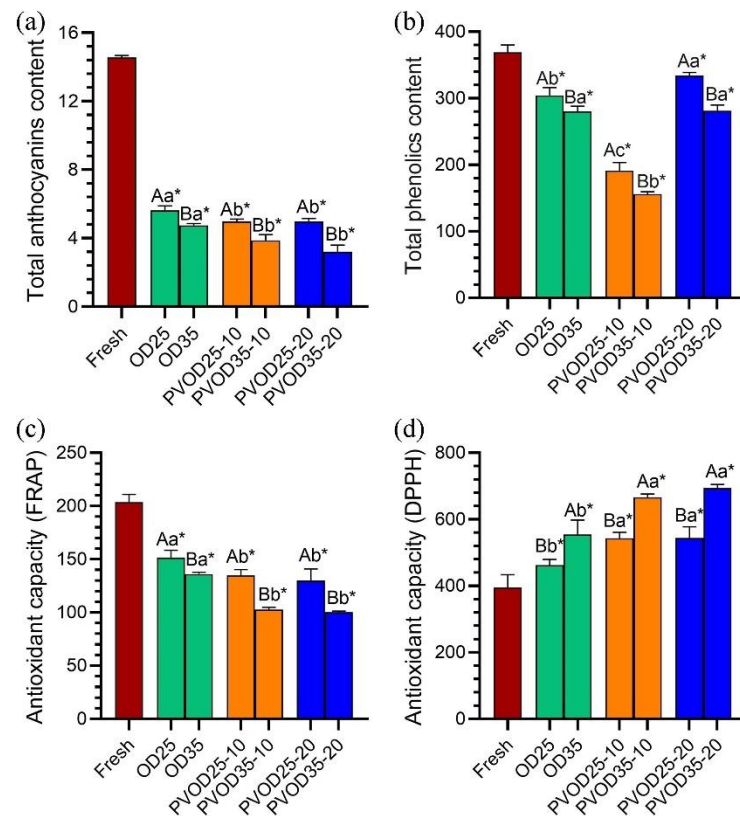


Figure 5 – Bioactive and antioxidant compounds of fresh and osmotically dehydrated strawberries.

The same uppercase letter indicates no significant difference between the concentrations of the osmotic solution in the same vacuum pulse time; and the same lowercase letter indicates no significant difference between vacuum pulse times at the same concentration as the osmotic solution, by the Tukey test ($p < 0.05$); The asterisk indicates a significant difference between each treatment and the fresh sample, by the Dunnett test ($p < 0.05$).

The leaching of some solutes, such as organic acids, minerals, fragrances and colorants is termed as the third flow in osmotic processes. This flow is neglected in quantitative terms (Ahmed et al., 2016). However, as observed in the present study, leaching can be qualitatively significant. According to Figure 5a, the TAC was reduced up to 78%.

The maintenance of anthocyanins is important for the appearance of the product, since anthocyanins are the main pigments of the strawberry, giving it a red color. In addition, these compounds are important for human health, since they are considered as pharmaceuticals providing various beneficial health effects on the human cardiovascular system, brain, liver, pancreas and kidney (Bendokas et al., 2019).

3.2.7 Total phenolics content (TPC)

The samples submitted to OD and PVOD showed a reduction in TPC compared to fresh strawberries, with a mean value of 368.47 mg of gallic acid per 100 g of sample on a dry basis (Figure 5b). Close values were found in other studies (Méndez-Lagunas et al., 2017; Núñez-Mancilla et al., 2014).

TPC was lower in samples treated with a higher concentration OS (Figure 5b). This increased reduction may be associated with the leaching of these compounds (de Jesus Junqueira et al., 2018). since it was observed that these samples had higher WL during the osmotic process. In addition, the VPT influenced the TPC, in which both treatments conducted with pulsed vacuum for 10 min (PVOD25-10 and PVOD35-10) had the lowest values in their respective OS concentrations (Figure 5b). The application of the vacuum pulse can act in the increase of the WL, as occurred using the treatments with OS of 35%, which would result in a greater reduction of TPC due to leaching. However, the application of vacuum reduces the contact of TPC with oxygen, due to the removal of occluded air, minimizing oxidative reactions (Ramya & Jain, 2017), therefore, there is a compensatory effect. Therefore, PVOD25-20 resulted in the highest TPC.

Polyphenols represent a considerable group of natural compounds found mainly in fruits, such as strawberries. In this group, there are anthocyanins, which belong to the subgroup known as flavonoids (Bendokas et al., 2019). In strawberries, about 40 phenolic compounds have already been identified, making strawberries one of the main food sources of these compounds (Basu et al., 2014).

3.2.8 Antioxidant capacity

The antioxidant capacity by the FRAP method is expressed by the antioxidant power ratio per sample mass, different from the DPPH method, in which the result is expressed by the amount of sample needed to reduce the mass of DPPH (Rufino et al., 2010). Thus, higher values of responses by the FRAP method and lower by the DPPH method indicate higher antioxidant capacity (Vimercati et al., 2020).

The antioxidant capacity of the fresh strawberry was higher than those osmotically dehydrated, by both determination methods (Figures 5c and 5d), presenting values of 203.67 μmol of ferrous sulfate per g of sample and 434.50 g sample per g DPPH, for FRAP methods and DPPH, respectively. Similar results were found by Pineli et al. (2011). The use of higher OS concentration and the application of vacuum resulted in samples with less TAC and antioxidant capacity.

Strawberries are considered a functional food due to their various benefits to health and basic nutrition, linked to the antioxidant capacity of these fruits. Antioxidant compounds act to reduce oxidative stress, responsible for causing several neurodegenerative diseases (Basu et al., 2014; Bendokas et al., 2019).

3.3 Principal component analysis (PCA)

PCA was used to help discriminate treatments through a multivariate analysis of responses regarding mass transfer and qualitative parameters. The score and loading plots are shown in Figures 6a and 6b, respectively.

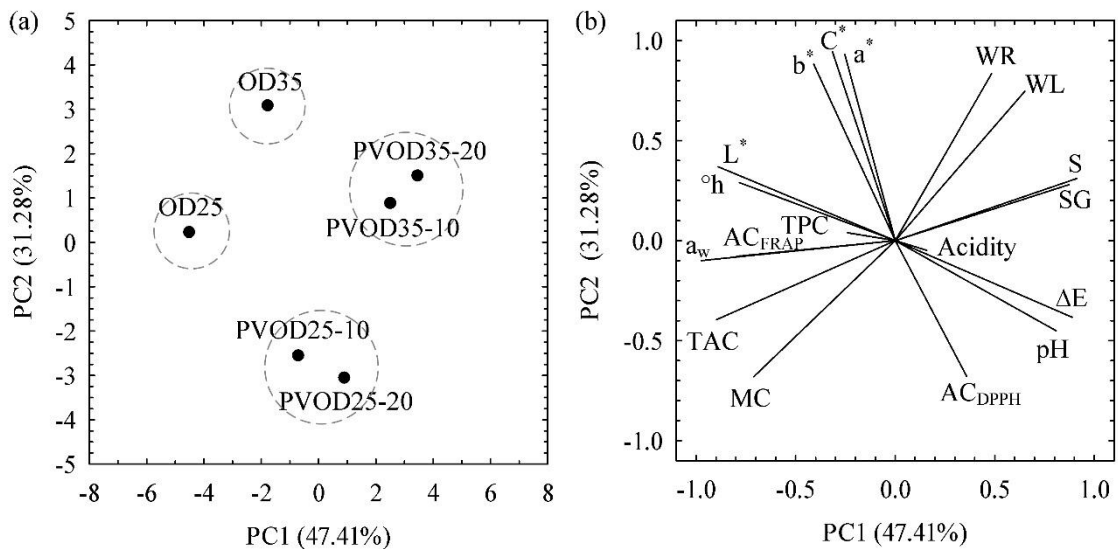


Figure 6 – Score (a) and loading (b) plots based on principal components analysis (PCA).

The dashed line circles represent the groups formed by the cluster analysis. SG: solid gain; WL: water loss; WR: weight reduction; MC: moisture content; a_w : water activity; S: shrinkage; TAC: total anthocyanins content; TPC: total phenolics content; AC_{FRAP}: antioxidant capacity by FRAP method; AC_{DPPH}: antioxidant capacity by DPPH method.

The first two principal components (PC1 and PC2) explained 78.69% of the total variance in the data set (Figure 6). Cluster analysis allowed for the distinction of treatments into four groups (Figure 6a). PVOD25-10 and PVOD25-20 treatments were grouped together, as well as PVOD35-10 and PVOD35-20 treatments, indicating similarity between both treatments in each group. Thus, it can be observed that the vacuum times of 10 and 20 min resulted in strawberries with similar characteristics. On the other hand, the osmotic processes carried out entirely at atmospheric pressure presented distinct from the processes in which the vacuum was applied.

According to the score plot (Figure 6a), it was possible to observe a distinction between the samples regarding the osmotic solution concentration, proving the strong influence that this factor exerted on the evaluated responses.

The responses regarding mass transfers (SG, WL, and WR) were close to each other, situated on the positive sides of both axes (Figure 6b). PVOD35-10 and PVOD35-20 treatments were located in this same region of the graph, corresponding to high values of SG, WL, and WR. Furthermore, these treatments are also associated with high shrinkage, low a_w , TAC, and MC.

OD35 treatment was associated with high values of a^* , b^* , and C^* . The OD25 treatment was associated with strawberries with high values of L^* , $^{\circ}h$, AC_{FRAP} , and low pH and ΔE values.

4 CONCLUSIONS

Strawberries impregnated with isomaltulose could be obtained by osmotic dehydration, mainly using vacuum application and higher concentrated osmotic solution. However, these conditions caused more loss of bioactive compounds and less antioxidant capacity. Therefore, the use of vacuum should occur when isomaltulose impregnation is the aim and the standard atmosphere process is indicated when high anthocyanins content and antioxidant capacity are wanted.

DECLARATIONS OF INTEREST

No potential conflict of interest was reported by the authors.

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ARTICLE 2 – CONVECTIVE DRYING WITH ETHANOL PRE-TREATMENT OF STRAWBERRY ENRICHED WITH ISOMALTULOSE

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(Elaborated in accordance to Food and Bioprocess Technology)

Abstract

Convective drying of strawberries was performed with or without ethanol pre-treatment (ET) in their fresh state or enriched with isomaltulose osmotic dehydration (OD) or by pulsed vacuum osmotic dehydration (PVOD). Both osmotic processes were executed by the immersion of 10 mm strawberry cubes in the osmotic solution for 300 min. The PVOD was done at reduced pressure (160 mbar) during the first 20 min. The ET was conducted by immersion of the samples in 95% ethanol for 2 min. Convective drying (60 °C and 1 m s⁻¹) was performed until a final moisture content of 11.50%_{w.b.}. The convective drying required from 195 to 300 min. The non-osmotically dehydrated dried sample required a shorter drying time and produced samples with lower water activity, hardness and color change, and higher total anthocyanin content (TAC), total phenolics content (TPC), and antioxidant capacity (AC). In addition to promoting enrichment with isomaltulose, both osmotic processes reduced shrinkage and hygroscopicity compared to non-osmotically dehydrated dried samples. ET reduced drying time by up to 30%, reduced hygroscopicity, and contributed to TAC, TPC, and AC preservation, showing as an interesting procedure to be applied in the convective drying of strawberries. However, the ET increased the shrinkage of the samples.

Keywords: Palatinose; Pre-treatment with ethanol; Heated air drying; Drying kinetics; Antioxidant capacity.

1 INTRODUCTION

Osmotic dehydration (OD) is considered a unitary operation of mass transfer between the food and the osmotic solution where the food is immersed. In this process, the two main flows are solid gain and water loss. The solid gain flow represents the incorporation of the solute from the osmotic solution to the food (González-Pérez et al., 2021; Ramya & Jain, 2017). This flow makes OD an interesting technology to enrich fruits, especially when the solute is beneficial to human health, such as isomaltulose (also known as palatinose), which is a low glycemic and cariogenic index carbohydrate (Lazou et al., 2020; Sawale et al., 2017).

The other flow refers to the diffusion of water from the food to the osmotic solution. A third flow also occurs, which is the leaching of compounds, such as bioactive and aromatic compounds. This flow is quantitatively negligible, but it can result in significant changes qualitatively (Ramya & Jain, 2017). To maximize solid gain and water loss flows, the vacuum application at the beginning of the osmotic process promotes the replacement of the occluded air by osmotic solution, increasing the area of contact with the osmotic solution (González-Pérez et al., 2021).

OD reduces the moisture content and water activity of the food, but not enough to ensure stability and significantly extend the shelf life of the product (Ramya & Jain, 2017). Therefore, convective drying is commonly applied after OD (Kroehnke et al., 2021; Macedo, da Silva Araújo, et al., 2021; Mendonça et al., 2017; Ramya & Jain, 2017; Turkiewicz et al., 2020), where heated air is used to complete the removal of water from the food. This is a low cost, simple structure and convenient operation process (Liu et al., 2020; Wang et al., 2019). However, drying can influence the quality parameters of the food, causing loss of heat-sensitive nutrients, such as bioactive compounds, present in fruits (Macedo et al., 2020; Mendonça et al., 2017; Omolola et al., 2017).

Strawberries are non-climacteric fruits of high sensory appreciation, due to their attractive color and their characteristic aroma and flavor. Furthermore, the strawberry has high levels of antioxidant compounds, such as phenolics and anthocyanins. These compounds are beneficial to health, as they are associated with reduced risk of several diseases, such as cancer, cardiovascular disorders, neurodegenerative and other chronic diseases (Basu et al., 2014). However, strawberries are very perishable, with a shelf life of a few days postharvest. Therefore, the use of preservation methods, such as convective drying, is essential for this fruit. However, studies have shown that convective drying has resulted in significant losses in the content of anthocyanins, phenolics, and antioxidant activity in strawberries (Karam et al., 2016; López-Ortiz et al., 2020; Méndez-Lagunas et al., 2017).

Treatments applied before drying fruits have been studied, aiming to accelerate the process of convective drying to reduce the time of exposure of the product to heated air (Wang et al., 2019). The addition of low-boiling organic solvents, such as ethanol, acetic acid, and isopropanol, is a pre-treatment that reduces the boiling point of water, due to the mixture of solvent+water. Furthermore, the solvent increases cell wall permeability, aiding the removal of water (Corrêa et al., 2012). As a low-cost and non-toxic solvent, ethanol has been the most used (Tatemoto et al., 2015). Studies have reported beneficial effects of ethanol pre-treatment on the drying of apple (M. L. Rojas, Augusto, et al., 2020), banana (Corrêa et al., 2012), guaco leaves (Silva et al., 2018), melon (da Cunha et al., 2020), pineapple (A. M. P. Braga et al., 2009; Alice M. P. Braga et al., 2010; Santos & Silva, 2009), pitaya (Araújo et al., 2020; Macedo, Corrêa, et al., 2021), potato (Meliza L. Rojas et al., 2019; Meliza Lindsay Rojas & Augusto, 2018a), pumpkin (Carvalho et al., 2020; M. L. Rojas, Silveira, et al., 2020; Meliza Lindsay Rojas & Augusto, 2018c) and scallion (Wang et al., 2019).

This work aimed to evaluate the influence of (i) osmotic dehydration, using isomaltulose as a solute, with or without pulsed vacuum, and (ii) ethanol pre-treatment on the drying kinetics and quality parameters, such as moisture content, water activity, shrinkage, hygroscopicity, color, total anthocyanins, total phenolics and antioxidant capacity of strawberry.

2 MATERIAL AND METHODS

2.1 Raw material

Fresh strawberries fruits, cultivar “Camino Real”, were purchased from the local market, selected for color (predominantly red skin), integrity (no injury), weight (19.41 ± 5.41 g), and maturation stage ($8.28 \pm 0.63^\circ$ Brix). The strawberries were then washed in running water, sanitized with chlorinated water (200 ppm for 10 min), rinsed, drained with absorbent paper, and stored at 4 °C for up to 2 days. The fruit skin was removed and the inside was cut into 10 mm cubes (Fig. 1).

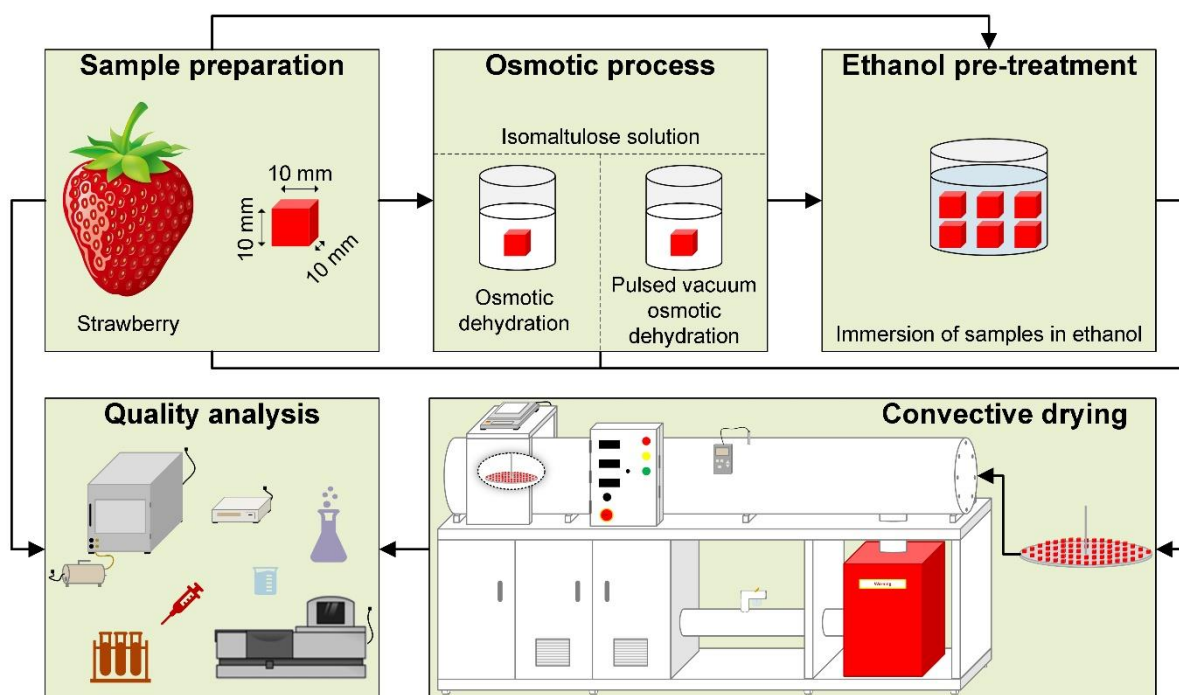


Fig. 1 Strawberry sample preparation, osmotic process, ethanol pre-treatment, convective drying and quality analysis.

2.2 Osmotic dehydration (OD) and pulsed vacuum osmotic dehydration (PVOD)

The osmotic solution of 35% (w w⁻¹) of isomaltulose (Beneo-Palatinit, Mannheim, Germany) was prepared and used for OD and PVOD of strawberry cubes, by immersing the cubes in the osmotic solution (Fig. 1), in the proportion of 1:20 (w w⁻¹), at 25 °C, for 300 min. OD was performed at atmospheric pressure throughout the process. PVOD was performed with vacuum application (160 mbar, absolute pressure) during the first 20 min. Then, atmospheric pressure was resumed. After the osmotic processes, the samples were immersed in an ice bath for 10 s to interrupt the mass flow. The sample surfaces were drained with absorbent paper (Corrêa et al., 2016).

2.3 Ethanol pre-treatment

Fresh and osmotic dehydrated by OD or PVOD samples were ethanol pre-treated (Fig. 1). The ethanol pre-treatment was performed by immersing the sample in 95% ethanol (v v⁻¹), in the proportion of 1:5 (w v⁻¹), for 2 min, at 25 °C.

2.4 Convective drying

2.4.1 Drying operation

Samples, fresh and dehydrated by OD or PVOD, non or ethanol pre-treated were dried in a tunnel dryer (Eco Engenharia Educacional, MD018 model, Brazil) (Fig. 1), with the parallel flow at 60 °C and 1.0 m s⁻¹. For each drying, 60.38±0.44 g of sample was used. The sample was weighed during drying, at 15 min intervals, with the aid of an analytical balance (Marte Científica, AD33000 model, Brazil) (accuracy ±0.01 g) coupled to the sample holder. Drying was carried out until the samples had a moisture content of 11.50±0.96%_{w.b.}. This value was established so that the samples of all treatments had water activity below 0.6 (Jay et al., 2005).

2.4.2 Moisture ratio

The moisture ratio (MR) of the samples during drying was calculated according to Eq. (1). The moisture content at equilibrium (X_e) was determined by drying the samples for 1440 min.

$$MR = \frac{X_t - X_e}{X_0 - X_e} \quad (1)$$

Where X₀, X_t, and X_e represent the dry basis moisture (kg water kg sample_{d.b.}⁻¹) at the initial, time t, and the equilibrium stage, respectively.

2.4.3 Page model

The Page model (Page, 1949), represented by Eq. (2), has been successfully fitted to represent the drying behavior (M. L. Rojas, Silveira, et al., 2020; Meliza Lindsay Rojas & Augusto, 2018c; Simpson et al., 2017).

$$MR = e^{-kt^n} \quad (2)$$

Where MR is the moisture ratio; t is time (min); k is the drying rate parameter (min⁻¹); n is the dimensionless drying parameter.

2.4.4 Peleg model

The two-parameter model proposed by Peleg (1988) (Eq. 3) was also used to predict the drying kinetics.

$$MR = MR_0 - \frac{t}{k_1 + k_2 t} \quad (3)$$

Where MR is the moisture ratio; MR₀ is initial moisture ratio (equal to 1); t is time (min); k₁ is the Peleg rate parameter (min⁻¹) and k₂ is the Peleg capacity parameter.

2.4.5 Drying rate (DR)

The DR was determined according to the moisture content on a dry basis, according to Eq. (4) (Macedo et al., 2020).

$$DR = \frac{X_t - X_{t+\Delta t}}{\Delta t} \quad (4)$$

Where DR is the drying rate, X_t and $X_{t+\Delta t}$ are the moisture content (kg water kg dry solid⁻¹) at t and $t+\Delta t$, respectively, t is the time (min) and Δt is the time difference (min).

2.5 Quality analysis

The fresh and dried samples were characterized in terms of moisture content, water activity, shrinkage, hardness, hygroscopicity, color, total anthocyanins, total phenolics, and antioxidant capacity.

2.5.1 Moisture content

The moisture content was determined by the gravimetric method established by method 934.06 of AOAC (2010), wherein the samples were placed in an oven at 70 °C, under vacuum.

2.5.2 Water activity (a_w)

The a_w of the samples was determined on an electronic hygrometer (Aqualab, series 3TE, Washington, USA), at 25°C (Corrêa et al., 2016).

2.5.3 Shrinkage

Sample volumes were determined using a toluene displacement method (Gamboa-Santos et al., 2014). The volumetric shrinkage of the samples was calculated according to Eq. (5).

$$\text{Shrinkage (\%)} = \left(1 - \frac{V}{V_0}\right) \times 100 \quad (5)$$

Where V and V_0 are the volumes (m³) of dried and fresh samples, respectively.

2.5.4 Hardness

The hardness of the samples was measured using a texture analyzer (Stable Micro Systems, TA-X2T, Surrey, England), equipped with a 50 kg load cell, a 6 mm diameter probe, test speed of 2 mm s⁻¹, and penetration distance of 3 mm. The hardness was expressed in Newton (N).

2.5.5 Hygroscopicity

The hygroscopicity of the dried samples was determined by placing approximately 1.0 g of sample in a desiccator with saturated sodium chloride solution (75% of moisture content) at 25 °C, for 7 days. Afterward, the samples were weighed and the hygroscopicity was calculated by the difference in weight of the sample, expressed in % (g of absorbed moisture per 100 g dry solids) (Sette et al., 2016).

2.5.6 Color

The colorimetric parameters of the samples were obtained by direct reading on a colorimeter (Konica Minolta, model CR-10, Osaka, Japan), with illuminant D65, using color scale CIELab.

The L^* parameter indicates the lightness, ranging from 0 (black) to 100 (white). C^* represents chromaticity or color saturation, in which the higher the C^* values, the higher the perceived color intensity. The h° indicates the hue of the sample, where the angles of 0° or 360°, 90°, 180°, and 270° represent red, yellow, green, and blue angles, respectively. The total color difference (ΔE) of dried strawberries was calculated according to Eq. (6) taking as reference the fresh strawberry, whose parameters are represented by the subscript “0” (Macedo et al., 2019).

$$\Delta E = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2} \quad (6)$$

Where L^* , a^* and b^* are CIE lightness coordinate, CIE red(+)/green(-) color attribute and CIE yellow(+)/blue(-) color attribute, respectively.

2.5.7 Total anthocyanins content (TAC)

The preparation of the extract for the quantification of TAC was performed by mixing 2g in 20 mL of ethanol acidified with HCl (0.1%). The mixture was centrifuged at 25400 xg, for 15 min. TAC was quantified by the differential pH method, according to Eq. (7) (Giusti & Wrolstad, 2001).

$$\text{TAC} \left(\frac{\text{mg}}{100\text{g}} \right) = \left\{ \frac{[(A_{510 \text{ nm}} - A_{700 \text{ nm}})_{\text{pH1}} - (A_{510 \text{ nm}} - A_{700 \text{ nm}})_{\text{pH4.5}}] \times \text{MW} \times \text{DF}}{\varepsilon \times L} \right\} \quad (7)$$

Where MW is the molecular weight of pelargonidin-3-glucoside (433.0 g mol⁻¹); DF is the dilution factor; ε is the molar absorptivity (22400 mol⁻¹); L is the path length in centimeters.

2.5.8 Total phenolics content (TPC)

The extract for quantification of TPC was prepared by mixing 5g in 40 mL of 50% methanol (v v⁻¹). The homogenized mixture was left to stand for 1h, protected from light, at

room temperature. The mixture was centrifuged at 25400 xg for 15 min and the supernatant was collected. The residue from the centrifugation was mixed with 40 mL of 70% acetone (v v⁻¹) and left to stand for 1 h, protected from light, at room temperature. The mixture was centrifuged at 25400 xg for 15 min. The supernatant was collected and mixed with the first. The final volume was completed with deionized water up to 100 mL (Rufino et al., 2010).

TPC was quantified using the Folin-Ciocalteu method (Waterhouse, 2003) and the response was expressed in mg of gallic acid per 100 g sample_{d.b.}.

2.5.9 Antioxidant capacity (AC)

The same extract prepared for the TPC analysis was used for the AC analysis (Rufino et al., 2010). AC was quantified by the ferric reducing antioxidant power capacity (FRAP) and 2,2-diphenyl-2-picryl-hydrazyl (DPPH) methods. The responses were expressed in μmol of ferrous sulfate per g of sample_{d.b.} and g sample_{d.b.} per g of DPPH (Rufino et al. 2010).

2.6 Statistical analysis

The experiment was conducted in a completely randomized design, in a factorial scheme (2 \times 3) (Table 1) and three repetitions. The data of the quality analyzes were submitted to analysis of variance (ANOVA), followed by the Tukey test. The Dunnett test was used to compare the fresh sample with each treatment individually.

Table 1 Experimental conditions

Code	Osmotic process	Ethanol pre-treatment	Convective drying
Fresh	No	No	No
CD	No	No	Yes
ET+CD	No	Yes	Yes
OD+CD	OD	No	Yes
OD+ET+CD	OD	Yes	Yes
PVOD+CD	PVOD	No	Yes
PVOD+ET+CD	PVOD	Yes	Yes

CD: convective drying; ET: ethanol pre-treatment; OD: osmotic dehydration; PVOD: pulsed vacuum osmotic dehydration.

A multivariate analysis was conducted by means of principal component analysis (PCA) on the drying time, water activity, shrinkage, hardness, hygroscopicity, colorimetric

parameters, total anthocyanins content, total phenolics content, and antioxidant capacity by FRAP and DPPH methods responses to analyze their particular interrelationships as influenced by osmotic process (none, OD and PVOD) and ethanol pre-treatment.

Statistical analyzes were carried out at the level of 5% probability of error, with the aid of software Statistica (StatSoft Inc., Tulsa, OK, USA).

3 RESULTS AND DISCUSSION

3.1 Drying kinetics

The drying times and drying kinetics of non-osmotically dehydrated and subjected to osmotic dehydration (OD) or pulsed vacuum osmotic dehydration (PVOD) strawberries, non- or ethanol pre-treated, are shown in Fig. 2. The time required to finish the drying was from 195 to 300 min (Fig. 2). Fig. 2a indicates the percentages of increasing drying time due to the inclusion of each osmotic process compared to the respective drying of fresh strawberry (CD or ET+CD). Fig. 2b presents the percentages of reduction of drying time due to the application of the ethanol pre-treatment compared to non-ethanol pre-treated drying.

The osmotically dehydrated (by OD or PVOD) samples required a longer time to complete the drying in relation to the drying of the fresh (non-osmotically dehydrated) samples showing longer drying time by up to 33% (Fig. 2a). Fresh strawberry had a higher moisture content than both osmotically dehydrated strawberries. However, the water present in the fresh strawberry is mostly free, which facilitates its removal, requiring less time to complete the convective drying process.

Between osmotically dehydrated non-ethanol pre-treated samples (OD+CD and PVOD+CD), the process in which vacuum was applied (PVOD) required a longer drying time compared to OD. Although PVOD has resulted in higher water loss during the osmotic process, PVOD provides greater incorporation of solute than OD (González-Pérez et al., 2021). Macedo et al. (2020a) also observed that samples in which there was greater incorporation of solids showed a trend towards a demand for longer drying time. This is due to the increased incorporation of solute during osmotic processes increasing the amount and strength of water bonds with the material, making it difficult to get out of the water from the material (Macedo, da Silva Araújo, et al., 2021). However, between osmotically dehydrated ethanol pre-treated samples (OD+ET+CD and PVOD+ET+CD), the OD process required a longer drying time (Fig. 2a).

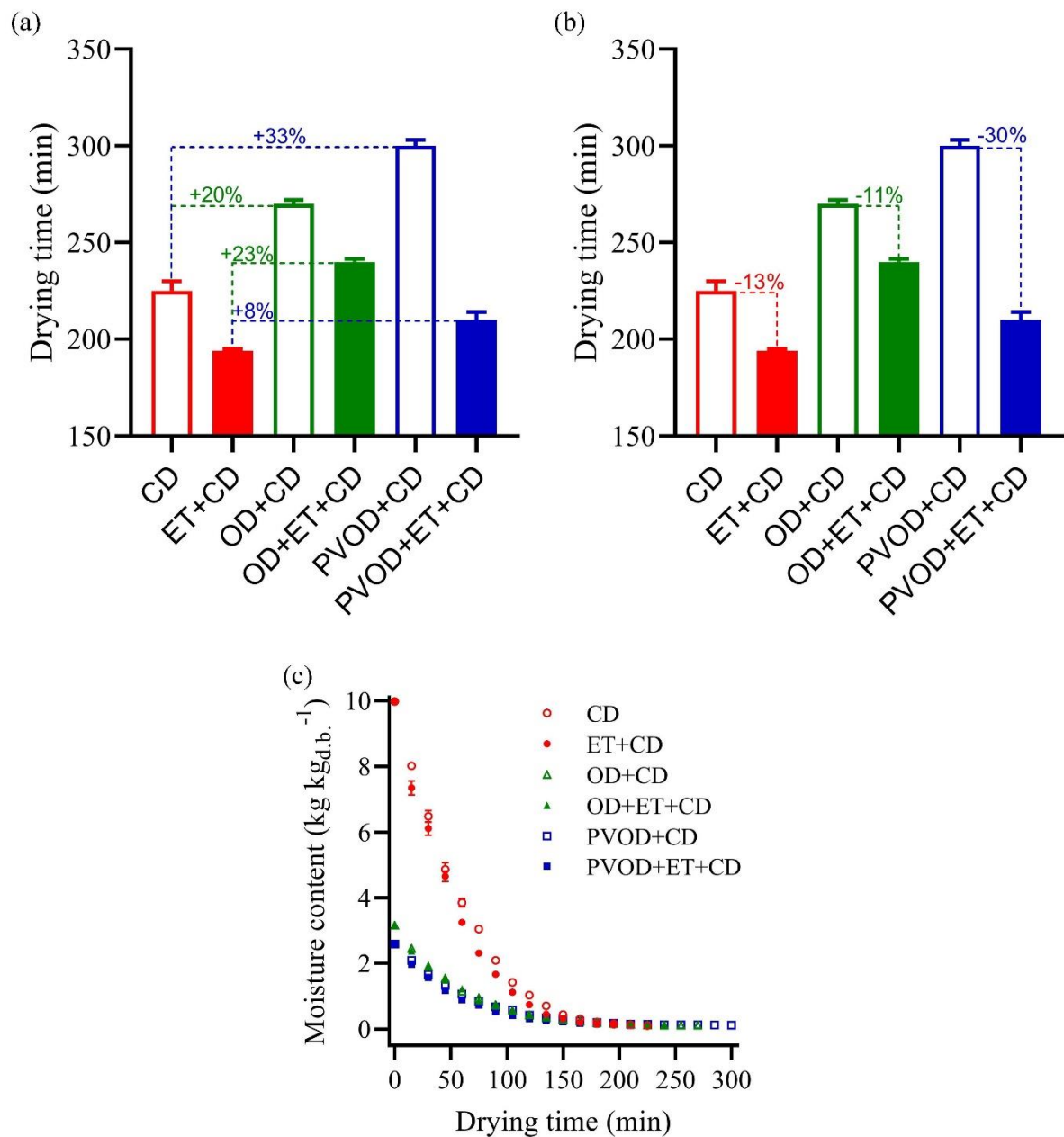


Fig. 2 Percentage of increase in the drying time of the osmotic processes in relation to that of the non-osmotically dehydrated dried sample (a), percentage of reduction with the application of the pre-treatment with ethanol (b) and drying kinetics of non- and osmotically dehydrated strawberries (c).

Compared to their respective non-ethanol pre-treated samples, the ethanol pre-treatment (ET) reduced the drying time of fresh samples and those submitted to OD and PVOD (Fig. 2b). ET reduced drying time by up to 30%. This reduction corresponds to the drying of the sample subjected to PVOD, in which the sample ethanol pre-treated (PVOD+ET+CD) required 210 min to complete the drying, while the non-ethanol pre-treated sample (PVOD+CD) required

300 min. The fresh and OD-submitted samples had their drying times reduced by 13 and 11%, respectively. Similar reductions were observed in other studies (Carvalho et al., 2020; Macedo, Corrêa, et al., 2021). It is possible to observe that ET is simple and low-cost processing, with a good capacity to reduce the time and cost of the drying process (M. L. Rojas, Silveira, et al., 2020). The added ethanol can reduce the drying time because the ethanol mixes with water, reducing the vaporization heat of the mixture (water+ethanol). Furthermore, the evaporation of ethanol (or the water+ethanol mixture) present in the surface layers of the material opens pores, facilitates the evaporation of water present in the innermost layers of the material. A third consequence of added ethanol is the Marangoni effect. This effect is due to the gradient of surface tension between water and ethanol, causing ethanol to promote water displacement, facilitating drying (Macedo, Corrêa, et al., 2021; M. L. Rojas, Augusto, et al., 2020; M. L. Rojas, Silveira, et al., 2020).

The moisture content of the samples reduced exponentially during drying (Fig. 2c). Initially, fresh and pre-dehydrated by OD and PVOD strawberries presented moisture content of 90.59, 75.95, and 72.12%_{w.b.}, respectively. It is possible to observe an abrupt reduction in the moisture content of both non-osmotically dehydrated samples (CD and ET+CD) during the first minutes of drying. In just 120 min, the moisture content of samples submitted to CD and ET+CD treatments was approximately 50.71%_{w.b.} and 42.62%_{w.b.}, respectively. This rapid reduction is due to the large proportion of free water present in the fresh strawberry initially, which facilitates water removal. On the other hand, the reduction in the moisture content of the osmotically dehydrated (both OD and PVOD) samples was mild, both for the non- and ethanol pre-treated samples.

3.2 Page model

The values of the Page model parameters are presented in Table 2. Page model fitted very well to the data, presenting high R^2 -values and low SE values (Table 2), indicating excellent similarity between the predicted and experimental data, as observed in other studies (Macedo, da Silva Araújo, et al., 2021; M. L. Rojas, Augusto, et al., 2020; Meliza Lindsay Rojas & Augusto, 2018c). The goodness of fit of Page model to the data justifies the great success of this equation when representing the migration of water during the drying of food (Simpson et al., 2017).

According to Table 2, the osmotically pre-dehydrated samples presented a higher value of the k parameter in relation to the control sample (not pre-dehydrated). There were no significant differences among the values of the k parameter between the osmotic processes,

whether pre-treated or not, with ethanol. Furthermore, the ET did not significantly influence the value of the k parameter. According to Simpson et al. (2017), parameter k may be associated with the diffusion coefficient and sample geometry.

Table 2 Values of the parameters (k and n), coefficient of determination (R^2) and standard error of estimate (SE) of the Page model

		CD	OD+CD	PVOD+CD
k	Non-ET	0.0078±0.0004 Ab	0.0175±0.0028 Aa	0.0139±0.0003 Aa
	ET	0.0113±0.0017 Ab	0.0201±0.0018 Aa	0.0178±0.0002 Aa
n	Non-ET	1.1828±0.0095 Aa	0.9924±0.0356 Ab	1.0217±0.005 Ab
	ET	1.1300±0.0330 Aa	0.9607±0.0194 Ab	0.9979±0.0029 Ab
R^2	Non-ET	0.9982±0.0006	0.9990±0.0009	0.9992±0.0001
	ET	0.9961±0.0021	0.9987±0.0008	0.9994±0.0001
SE	Non-ET	0.0137±0.0024	0.0087±0.0045	0.0083±0.0002
	ET	0.0200±0.0006	0.0105±0.0033	0.0077±0.0001

Values are mean±standard deviation, n=3. CD: convective drying; ET: ethanol pre-treatment; OD: osmotic dehydration; PVOD: pulsed vacuum osmotic dehydration. The same uppercase letter indicates no significant difference between non and ethanol pre-treated samples; and the same lowercase letter indicates that there is no significant difference among non-osmotically dehydrated, submitted to OD and PVOD dried samples, by the Tukey.

Osmotic processes reduced n values in relation to the values of the non-osmotically dehydrated samples (CD and ET+CD) (Table 2). A significant difference was not observed between both osmotic processes. Parameter n is related to the type of diffusion and microstructure of the material, where values above 1 indicate super-diffusion and below 1 indicate sub-diffusion (Macedo, da Silva Araújo, et al., 2021; Meliza Lindsay Rojas & Augusto, 2018c; Simpson et al., 2017). Rojas, Silveira, Augusto (2020) stated that when the value of n is different from 1, other mechanisms than diffusion are important. The drying of the samples submitted to the treatments OD+CD, OD+ET+CD and PVOD+ET+CD showed $n < 1$, and samples submitted to the treatment PVOD+CD showed $n > 1$. However, these values were very close to 1 (Table 2). On the other hand, the n values of the non-osmotically dehydrated samples (CD and ET+CD) were higher than 1, indicating super-diffusion, associated with an important influence of another mechanism, such as capillarity, as reported in other studies (M. L. Rojas, Silveira, et al., 2020; Meliza Lindsay Rojas & Augusto, 2018b, 2018c).

3.3 Peleg model

Peleg model is used to describe moisture sorption curves. This model fitted well with the experimental data of the moisture ratio kinetics during the convective drying of strawberries, presenting high values of R^2 and low values of SE (Table 3), as also reported in other studies (Aydar, 2020; Cruz et al., 2016).

Table 3 Values of the parameters (k_1 and k_2), coefficient of determination (R^2) and standard error of estimate (SE) of the Peleg model

		CD	OD+CD	PVOD+CD
k_1	Non-ET	52.8302+0.6889 Aa	45.8673+1.8166 Ac	49.5744+0.2745 Ab
	ET	46.0989+1.0722 Ba	45.0801+0.9429 Aa	44.9435+0.1139 Ba
k_2	Non-ET	0.7220+0.0043 Ab	0.8064+0.0106 Aa	0.8141+0.0019 Aa
	ET	0.7291+0.0080 Ac	0.8084+0.0062 Aa	0.7876+0.0006 Bb
R^2	Non-ET	0.9892+0.0009	0.9932+0.0013	0.9895+0.0002
	ET	0.9909+0.0025	0.9947+0.0017	0.9945+0.0004
SE	Non-ET	0.0339+0.0015	0.0245+0.0026	0.0299+0.0003
	ET	0.0312+0.0043	0.0214+0.0036	0.0229+0.0007

Values are mean±standard deviation, n=3. CD: convective drying; ET: ethanol pre-treatment; OD: osmotic dehydration; PVOD: pulsed vacuum osmotic dehydration. The same uppercase letter indicates no significant difference between non and ethanol pre-treated samples; and the same lowercase letter indicates that there is no significant difference among non-osmotically dehydrated, submitted to OD and PVOD dried samples, by the Tukey test ($p<0.05$).

As shown in Table 3, there was a trend of lower k_1 with the ET. The exception was the OD+CD treatment, in which the non and submitted to ET samples did not differ from each other ($p>0.05$). Cruz et al. (2016) observed that dryings that required less drying time had a lower value of parameter k_1 . Among the samples not submitted to ET, the non-osmotically dehydrated (CD) sample had the highest k_1 . The OD+CD sample had the lowest. On the other hand, no significant difference was observed in relation to the value of k_1 among samples submitted to ET.

The k_2 parameter showed a significant difference only for those osmotically dehydrated with vacuum application between non (PVOD+CD) and pre-treated ethanol (PVOD+CD+ET) samples. In the other treatments, no significant differences were observed between non and submitted to ET samples. Furthermore, non-dehydrated strawberries had lower k_2 values than

osmotically pre-treated ones. According to Cruz et al. (2016), the k_2 parameter is related to the characteristics of the material and does not depend on the drying conditions. This can be seen from the fact that the value of the k_2 parameter was mainly influenced by the use of osmotic processes, which causes mass exchanges between the osmotic solution and the food, changing the composition of the material in relation to fresh one (not osmotically dehydrated).

3.4 Drying rate (DR)

The DR during sample dryings is shown in Fig. 3. At the beginning of drying, the strawberries (especially the non-osmotically dehydrated) present high moisture content, aiding the removal of water, which results in high DR values.

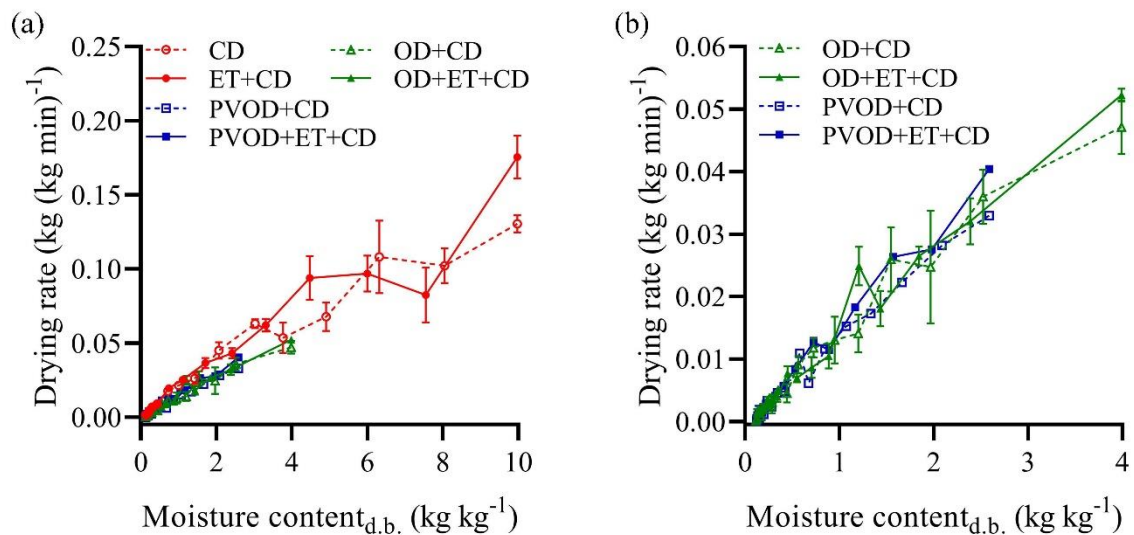


Fig. 3 Drying rates of the samples.

During drying, the DR reduced, as the amount of water to be removed reduces throughout the process, making it increasingly difficult to remove the water.

Non-osmotically dehydrated dried strawberries (CD and ET+CD) showed higher DR compared to osmotically dehydrated samples (Fig. 3a), as also observed by Tylewicz et al. (2019). This is due to the amount of free water for removal being larger in fresh material compared to samples that were previously dehydrated by osmotic processes. Furthermore, the incorporation of solids during osmotic processes increases the interactions of water with the material, hindering the outflow of water, reducing the DR (Macedo, da Silva Araújo, et al., 2021).

The DR values of the osmotically dehydrated samples are shown separately in Fig. 3b to facilitate the visualization of data behavior. Among osmotically dehydrated strawberries, the DR values were close throughout the dryings, except at the beginning of the drying process, when the OD process resulted in higher DR during drying (Fig. 3b). This is due to the PVOD process which results in higher water loss during the osmotic process, resulting in a sample with lower moisture content at the beginning of convective drying. Furthermore, the PVOD process provides higher solid gain during the osmotic process (González-Pérez et al., 2021). Therefore, in the samples submitted to PVOD, the amount of water to be removed is lower and the interactions of water with the constituents of the material are higher, by increasing dry matter.

ET showed a trend towards a higher DR compared to non-ethanol pre-treated samples, especially for non-osmotically dehydrated strawberries. This is due to the effects of the addition of ethanol in favor of facilitating the removal of water, as mentioned above.

Periods with constant or near-constant DR were rare or even non-existent. The same was observed by Gamboa-Santos et al. (2014) in convective drying of strawberries at different air temperatures.

3.5 Water activity (a_w)

The fresh material presented a high value of a_w , 0.986 ± 0.004 (Fig. 4a), as is the characteristic of strawberry (Dermesonlouoglou et al., 2016; Tylewicz et al., 2020). The dryings significantly ($p < 0.05$) reduced the a_w of the non- and osmotically dehydrated samples. After drying, dried strawberries presented a_w between 0.423 and 0.585. Similar results were observed by Amami et al. (2017) when performing convective drying of strawberries to reduce the moisture content by 90%.

The non-osmotically dehydrated dried samples (CD and ET+CD) presented lower a_w values compared to samples pre-dehydrated by OD or PVOD (Fig. 4a). During the osmotic processes, there was incorporation of solids (isomaltulose), changing the composition and structure of the food, which makes the water interact differently with the material, influencing the fugacity of the water and consequently the a_w (Damodaran, 2017).

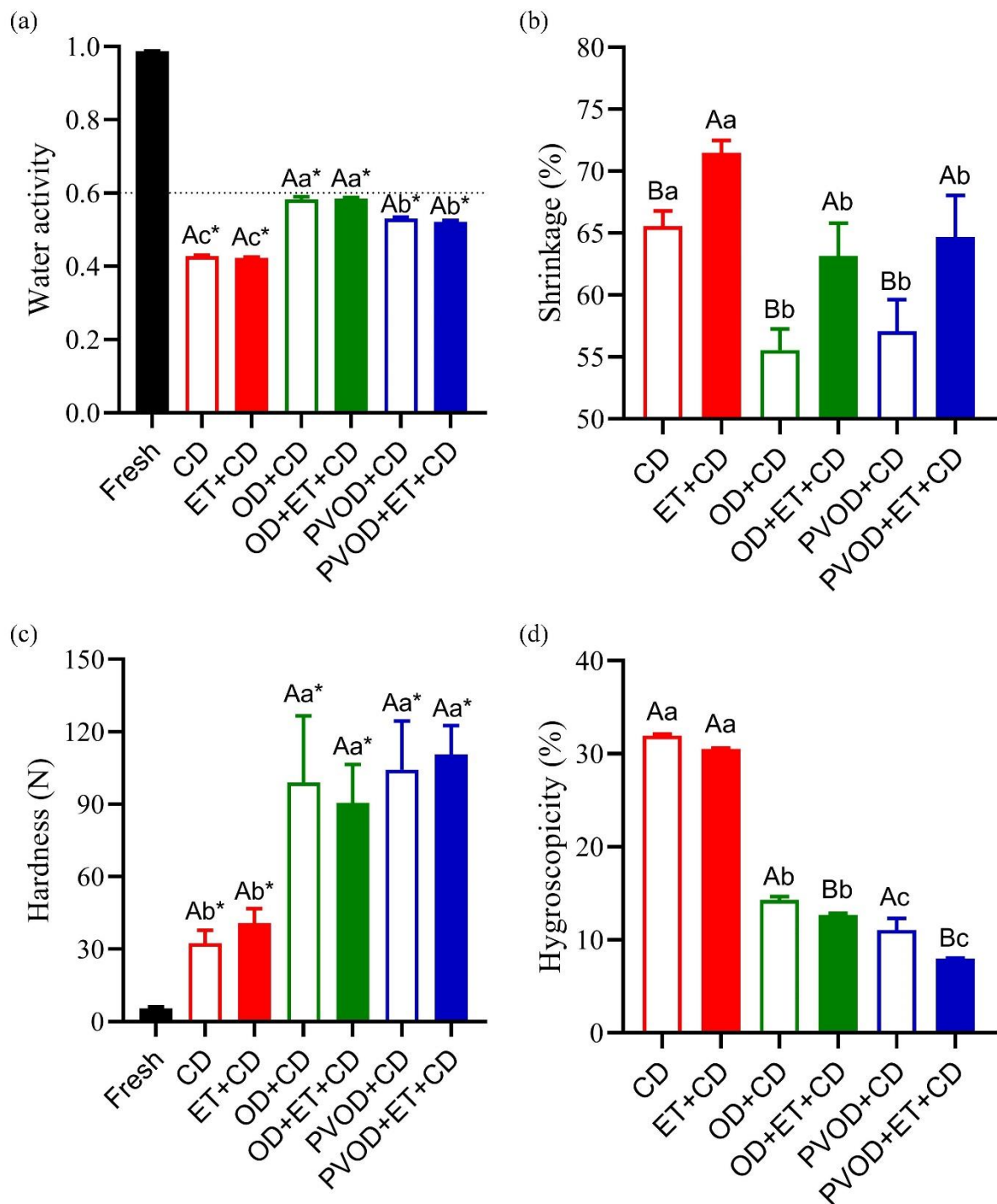


Fig. 4 Water activity (a), shrinkage (b), hardness (c) and hygroscopicity (d) of the samples.

Values are mean \pm standard deviation, n=3. The same uppercase letter indicates no significant difference between non and ethanol pre-treated samples; and the same lowercase letter indicates that there is no significant difference among non-osmotically dehydrated, submitted to OD and PVOD dried samples, by the Tukey test ($p < 0.05$). The asterisk indicates a significant difference between each treatment and the fresh sample, by the Dunnett test ($p < 0.05$).

Among the osmotic processes, the application of the vacuum at the beginning of the osmotic process (PVOD) resulted in a lower a_w value in dried strawberry (Fig. 4a). The same behavior was observed by Deng and Zhao (2008). This behavior can be explained by the lower a_w value of the sample submitted to PVOD than to OD, before convective drying (data not shown). In addition, the application of vacuum during the osmotic process promotes changes in the structure of the food matrix. Therefore, among the samples submitted to OD and PVOD, OD caused less change in the food, resulting in higher a_w .

The ethanol pre-treated samples did not present a significant difference in a_w in relation to their respective non-pre-treated ethanol samples (Fig. 4a).

The a_w is an important parameter in food preservation and quality. According to Jay et al. (2005), microorganisms and enzymes need water to become active. Drying is a preservation method by reducing the moisture content to the point of inhibiting the activity of microorganisms that deteriorate food and cause food poisoning (Jay et al., 2005). The range of the a_w values of dried strawberries indicates that they are microbiologically stable and safe since they presented a value under 0.6 (value represented by the dotted line in Fig. 4a).

3.6 Shrinkage

Dried strawberries showed shrinkage from 55.56 to 71.49% (Fig. 4b). During drying, water is removed. the viscoelastic matrix moves towards space previously occupied by water, causing the material to shrink (Dehghannya et al., 2018). In processes where there is a high removal of water, as in the present study (90.59 to 11.50%_{w.b.}), shrinkage occurs in a high proportion (Nahimana et al., 2011). Gamboa-Santos et al. (2014) found that shrinkage has a strong correlation with moisture ratio in strawberry drying.

OD and PVOD contributed to the reduction of shrinkage in relation to non-osmotically dehydrated strawberries (Fig. 4b). The incorporation of solids that occurs during osmotic processes reduces the mobility of the solid matrix of the food, giving the material an elastic behavior typical of the vitreous state, which reduces shrinkage along convective drying (Nahimana et al., 2011).

The ET use increased the shrinkage, both for non-osmotically dehydrated and pre-dehydrated by OD or PVOD samples (Fig. 4b). The same was observed in other studies (Silva et al., 2018). According to Silva et al. (2018), the rapid evaporation of water during the drying with ET causes greater damage to the material structure and consequently more shrinkage.

3.7 Hardness

Hardness can be defined physically as the force necessary to cause a deformation in the material. Sensorially, hardness represents the force necessary to compress a solid food between the molar teeth. Therefore, it is an important parameter of dried food texture (Macedo, da Silva Araújo, et al., 2021; Szczesniak, 2002).

The hardness of the strawberries is shown in Fig. 4c. Similar values were found in other studies (Deng & Zhao, 2008; Khubber et al., 2020; Li et al., 2019; Schwieterman et al., 2014; Tylewicz et al., 2019). The dried samples showed higher hardness than the fresh strawberry, as also observed by Tylewicz et al. (2019). This is due to physical changes, such as shrinkage and distortion, which occur due to the moisture content reduction during the drying of the strawberry, resulting in increased hardness.

Among the dried strawberries, the osmotic processes increased the hardness of the dried strawberries. Some studies have observed that the inclusion of the osmotic process prior to convective drying can cause softening of the dried strawberry (Tylewicz et al., 2019). Khubber et al., (2020) studied different solutes for carrot osmotic dehydration, noting that the solute used influences the reduction or increase in the hardness of the dried product. In the present study, it was observed that the incorporation of isomaltulose resulted in dried strawberries with higher hardness (Fig. 4c). This same effect was observed by Lyu et al. (2017). Between both osmotic processes (OD and PVOD), significant differences were not observed, indicating that the vacuum application did not affect the hardness of the osmotically dehydrated dried strawberries.

ET had no significant influence on the hardness of dried strawberries, compared to the respective non-pre-treated samples (Fig. 4c).

3.8 Hygroscopicity

The hygroscopicity of the samples was influenced by the treatments studied. Osmotic processes, especially PVOD, reduced dried strawberry hygroscopicity compared to non-osmotically dehydrated dried samples (Fig. 4d). Isomaltulose is a sugar of very low hygroscopicity, giving this characteristic to the product in which it is added (Sawale et al., 2017). Thus, the samples that incorporate more isomaltulose, that is, those submitted to PVOD, resulted in lower hygroscopicity.

ET contributed to the reduction of hygroscopicity of osmotically dehydrated by OD and PVOD samples. However, ET had no significant influence on non-osmotically dehydrated dried samples (Fig. 4d).

Hygroscopicity is an important property in dehydrated foods. It is the capacity of the product to absorb moisture from the air. Foods with high hygroscopicity can have their stability

reduced quickly, due to increased water absorption from air, increasing their water activity, reducing the shelf life of the product (Sette et al., 2016). Therefore, in the case of the present study, it is desirable that the dried strawberry has a low hygroscopicity.

3.9 Color

Color is an important attribute of the quality of a product since it is the first parameter evaluated by the consumer and directly influences their choices and preferences. In the case of fruits, color is an attribute that can be associated with the degree of maturation and freshness of the fruit, influencing consumer acceptance. In strawberries, the consumer expects the color to be predominantly red (Pathare et al., 2013).

The influence of the treatments studied in relation to the colorimetric parameters of the samples is presented in Fig. 5.

Non-osmotically dehydrated dried samples (CD and ET+CD) showed L^* statistically equal to L^* of fresh strawberry. The samples submitted to OD and PVOD showed high L^* . This may be due to the pigments leaching during osmotic processes. The ET influenced the L^* only in the PVOD treatment sample, in which the ET resulted in a higher value of this parameter (Fig. 5a).

The dried samples presented higher C^* than fresh strawberries (Fig. 5b). The drying process causes a concentration of the constituents of the material, due to the reduction of moisture content, resulting in increased color saturation. The sample submitted to the OD+CD treatment presented the highest C^* value. The ET influenced only the sample submitted to OD (Fig. 5b).

The dried samples showed a higher value of $^{\circ}h$ than fresh strawberries, indicating that the red hue was lower in the dried samples (Fig. 5c). It is due to the leaching of anthocyanins during osmotic processes and the degradation of anthocyanins during CD since anthocyanins are the pigments that confer red coloration to strawberries. ET did not influence non-osmotically dehydrated dried strawberry $^{\circ}h$ but influenced dried samples that were osmotically dehydrated (Fig. 5c).

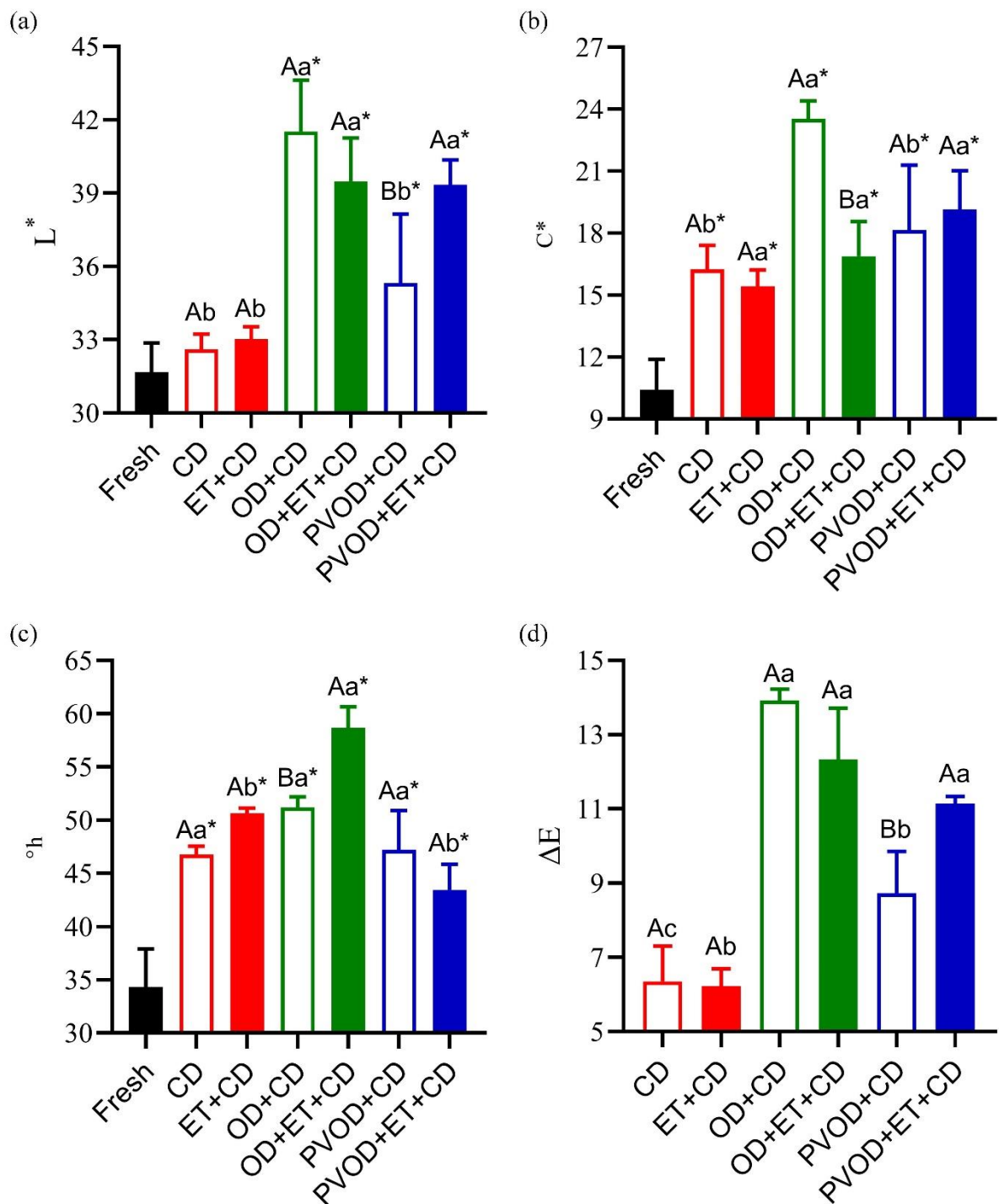


Fig. 5 Colorimetric parameters of the samples.

Values are mean \pm standard deviation, n=3. The same uppercase letter indicates no significant difference between non and ethanol pre-treated samples; and the same lowercase letter indicates that there is no significant difference among non-osmotically dehydrated, submitted to OD and PVOD dried samples, by the Tukey test ($p < 0.05$). The asterisk indicates a significant difference between each treatment and the fresh sample, by the Dunnett test ($p < 0.05$).

The ΔE is used to express the magnitude of the color difference between the samples and a control sample (fresh strawberry). The non-osmotically dehydrated dried samples (CD and ET+CD) showed the lowest ΔE values. The ET had influence only in the treatment of PVOD, increasing the ΔE (Fig. 5d). Differences in perceptible color can be classified as very distinct ($\Delta E > 3$), distinct ($1.5 < \Delta E < 3$), and small differences ($1.5 < \Delta E$) (Pathare et al., 2013). Based on this classification, the color difference between the dried samples and the fresh strawberry was very distinct.

Exposure to heated air can result in chemical reactions responsible for changing the color of the food, whether related to discoloration, due to degradation of heat-sensitive pigments, such as anthocyanins in strawberries; to non-enzymatic browning; to enzymatic browning by polyphenol oxidase (PPO) activity (López-Ortiz et al., 2020; Omolola et al., 2017). However, it can be observed that the dried samples showed equal or higher L^* , and higher C^* than the fresh strawberry, indicating that the browning reactions (enzymatic or non-enzymatic) did not occur excessively (Mierzwa & Kowalski, 2016).

3.10 Total anthocyanins content (TAC), total phenolics content (TPC), and antioxidant capacity (AC)

The TAC, TPC, and AC by the FRAP and DPPH methods are presented in Figs. 6a, 6b, 6c, and 6d, respectively. Fresh strawberries presented as a good source of bioactive and antioxidant compounds (Figs. 6a to 6d). These compounds contribute to the strawberry being classified as a functional food with various preventive and therapeutic health benefits, with positive effects in various diseases, such as hypertension, inflammation, cancer, cardiovascular and neurodegenerative diseases (Basu et al., 2014).

Fresh strawberries presented higher TAC, TPC, and AC than dried strawberries (Figs. 6a to 6d), since convective drying causes degradation of these compounds. During drying, the reduction of moisture content and physical changes favor oxidative reactions, which degrade the bioactive and antioxidant compounds of strawberry (Méndez-Lagunas et al., 2017).

The TAC was the group of compounds that suffered the greatest degradation. This is due to the loss of anthocyanins by leaching during osmotic processes since these compounds are very water soluble. Furthermore, anthocyanins are very sensitive, suffering high losses during exposure to drying air temperature, due to the change in the structure of anthocyanins, in addition to oxidative processes (Méndez-Lagunas et al., 2017). According to Méndez-Lagunas et al. (2017), anthocyanins can be degraded enzymatically by polyphenol oxidase, as

they are relatively heat-stable enzymes. Méndez-Lagunas et al. (2017) found a significant reduction in TAC after convective drying of strawberries, mainly at the beginning of drying.

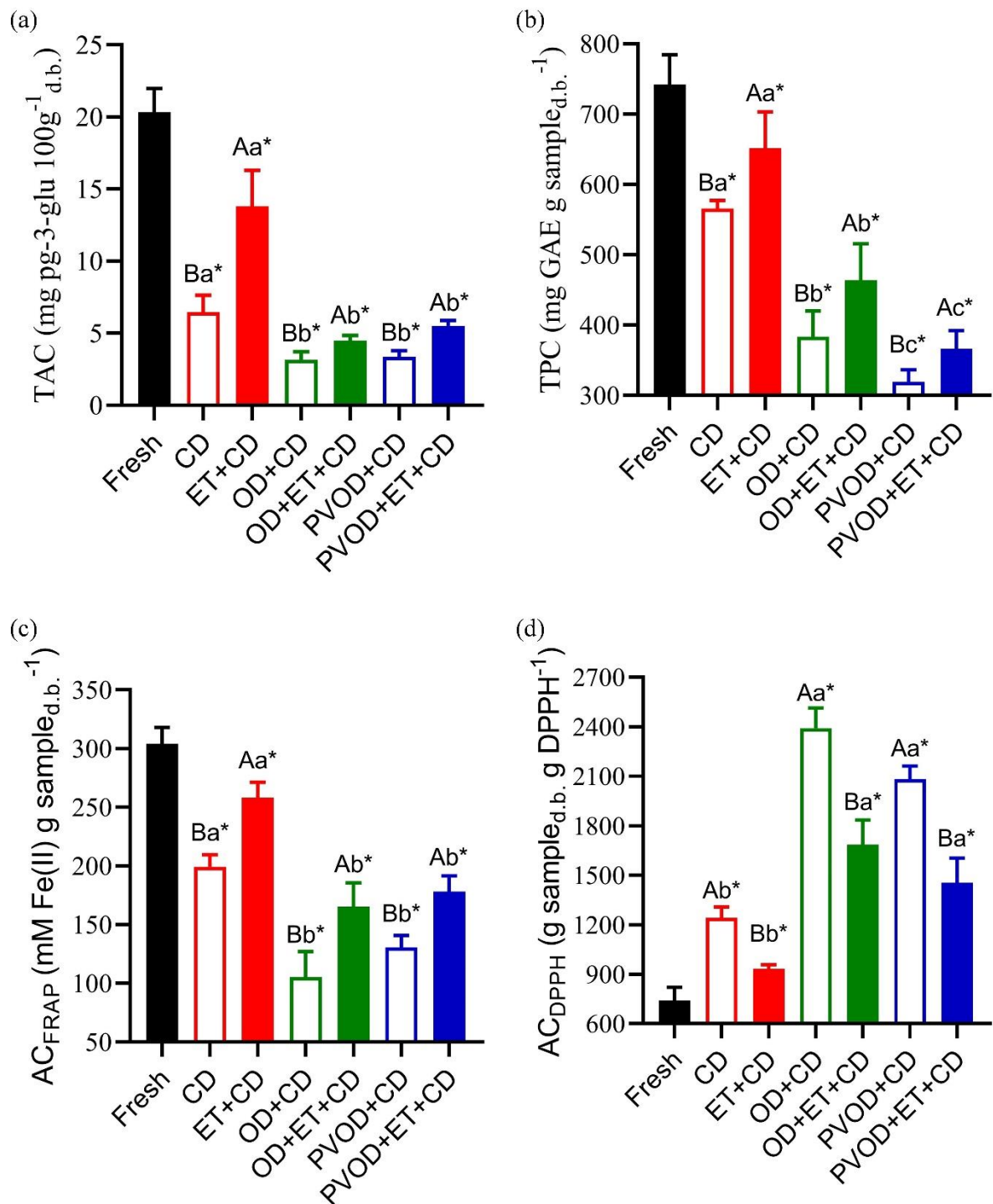


Fig. 6 Total anthocyanins content (a), total phenolics content (b) and antioxidant capacity by FRAP (c) and DPPH (d) methods of the samples.

Values are mean±standard deviation, n=3. TAC: total anthocyanins content; TPC: total phenolics content. The same uppercase letter indicates no significant difference between non and ethanol pre-treated samples; and the

same lowercase letter indicates that there is no significant difference among non-osmotically dehydrated, submitted to OD and PVOD dried samples, by the Tukey test ($p < 0.05$). The asterisk indicates a significant difference between each treatment and the fresh sample, by the Dunnett test ($p < 0.05$).

Osmotic processes and convective drying also significantly reduced the TPC and CA of the samples (Figs. 6a to 6d). Méndez-Lagunas et al. (2017) and Tylewicz et al. (2019) also observed a reduction in TPC and AC after strawberry convective drying. According to Méndez-Lagunas et al. (2017), during drying, thermal degradation of phenolic compounds occurs. Furthermore, bound phenolic compounds are released and partial degradation of lignin occurs, promoting the release of phenolic acid derivatives, increasing the thermal degradation of these compounds (Méndez-Lagunas et al., 2017).

The ET had a beneficial effect on the preservation of TAC, TPC, and AC (Figs. 6a to 6d). The effect of ethanol on the preservation of bioactive and antioxidant compounds may be related to the reduction of drying time, reducing the exposure time of the product to hot air (Araújo et al., 2020). Some studies report that ET reduces the content of some bioactive compounds. However, this effect may be related to the long time of the ET process, with the extraction of these compounds (da Cunha et al., 2020).

3.11 Principal component analysis

The principal component analysis (PCA) was used to investigate the influence of both studied factors on drying time, water activity, shrinkage, hardness, hygroscopicity, color parameters, total anthocyanins content, total phenolics content, and antioxidant capacity by FRAP and DPPH methods responses, as illustrated in Figs. 7a and 7b.

The first (PC1) and second (PC2) axes were responsible for 71.78% and 12.11%, respectively, giving a total of 83.89% of the explained variance. As seen in the scores plot (Fig. 7a), the osmotically dehydrated dried samples were on the negative side of the PC1 axis, while the non-osmotically dehydrated dried samples (CD and ET+CD) were on the positive side of the PC1 axis, evidencing the differences between the properties of non- and osmotically dehydrated samples after drying process.

The shrinkage, TAC, TPC, AC_{FRAP} responses were observed to positively correlated (Fig. 7b). It can be observed that the highest values of these responses were associated with the ET+CD treatment (Figs. 7a and 7b). As expected, AC_{DPPH} was opposite to TAC, TPC, AC_{FRAP} responses, as low AC_{DPPH} response values indicate high AC. High values of anthocyanic,

phenolic and antioxidant compounds were obtained when drying time was short, indicating that longer drying times resulted in more degradation of these compounds. (Fig. 7b).

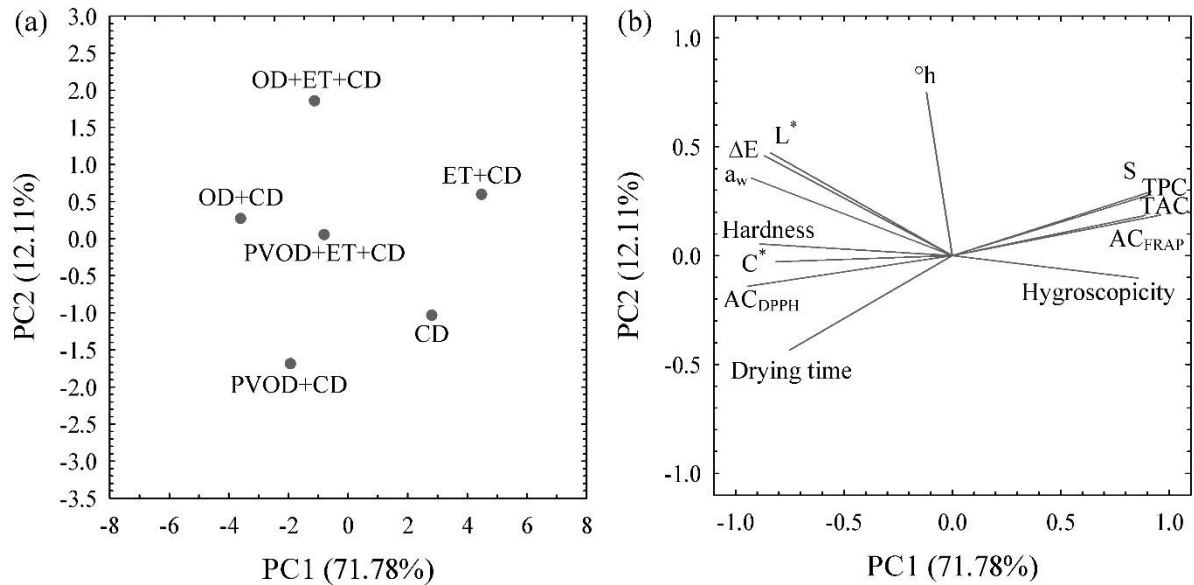


Fig. 7 Principal component analysis of the dried strawberries responses in effect of osmotic processes and ethanol pre-treatment: (a) score plot and (b) loading plot.

S: shrinkage; TAC: total anthocyanins content; TPC: total phenolics content; AC_{FRAP}: antioxidant capacity by FRAP method; AC_{DPPH}: antioxidant capacity by DPPH method.

In general, the osmotically dehydrated samples (by OD or PVOD, non-or ethanol pre-treated) were associated with high values of drying time, a_w , hardness, L^* , C^* , ΔE , and AC_{DPPH}. Therefore, when searching for strawberries with maximum content of bioactive and antioxidant compounds, the choice should be made for the ethanol pre-treated dried fruit, non-osmotically dehydrated. However, if the main interest is in a dried strawberry enriched with isomaltulose, the inclusion of the osmotic process previously convective drying is recommended.

4 CONCLUSION

Osmotic dehydration, with or without vacuum application, enriched strawberries with isomaltulose and contributed to the reduction of shrinkage and hygroscopicity of the dried product. However, osmotic processes increased drying time, water activity, hardness and caused higher color change in relation to non-osmotically dehydrated strawberry, besides resulting in samples with lower total anthocyanin and total phenolics contents and antioxidant capacity.

Ethanol pre-treatment proved to be an interesting procedure to be applied in the convective drying of strawberries, as it was efficient to reduce drying time, contributed to the reduction of hygroscopicity, and minimized the degradation of total anthocyanins and total phenolics contents and antioxidant capacity. However, the use of ethanol caused higher shrinkage of the samples.

DECLARATIONS

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Conflicts of interest/Competing interests

The authors have no conflicts of interest to declare that are relevant to the content of this article.

Availability of data and material

The authors chose not to present the data and material.

Code availability

Not applicable.

Author contributions

Leandro Macedo: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Project Administration, Validation, Visualization, Writing - Original Draft.

Jefferson Corrêa: Conceptualization, Funding acquisition, Investigation, Methodology, Project Administration, Supervision, Writing - Review & Editing.

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Wallaf Vimercati: Formal analysis, Investigation, Methodology, Writing - Review & Editing.

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**ARTICLE 3 – INTERMITTENT MICROWAVE DRYING AND HEATED AIR
DRYING OF FRESH AND ISOMALTULOSE ENRICHED STRAWBERRY**

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(Elaborated in accordance to LWT)

Abstract

Fresh and osmotically pretreated (using a 35% isomaltulose solution) strawberries were dried by intermittent microwave drying (IMD) and heated air drying (HAD). The samples were dried to a moisture content of 10.28%_{w.b.}. The sample temperature was monitored during IMD, adopting a setpoint temperature of 60°C and continuous airflow (0.5 m s⁻¹, room temperature). The HAD was performed by exposing the samples to air (0.5 m s⁻¹ and 60 °C). IMD stood out for being faster (up to 64.91%) and consuming less energy (up to 73.16%) than HAD. During IMD, the magnetron was off for most of the time (up to 74.13%). The isomaltulose impregnation reduced drying rates in both methods, increasing drying time. Dried samples showed a_w within the safe range ($a_w < 0.6$). The impregnation process was carried out to a reduced shrinkage product besides the enrichment with isomaltulose. Non-pre-treated strawberries had lower a_w , hardness, and ΔE , and higher levels of bioactive compounds and antioxidants, especially with the IMD method. Using IMD, retentions of up to 47.46%, 70.26%, and 81.18% of anthocyanins, phenolic compounds, and antioxidant capacity were obtained, respectively, concerning fresh strawberries.

Keywords: Convective drying; Microwave drying; Drying kinetics; Anthocyanins; Antioxidant compounds.

1 INTRODUCTION

The increase in concern and care for health and well-being has made consumers increasingly seek healthier foods nowadays (Tylewicz et al., 2020). Among healthy foods, fruits stand out due to the richness of nutrients, such as fiber, vitamins, minerals, and antioxidant compounds (Basu et al., 2014). The strawberry is one of the most consumed fruits because, besides its nutritional composition, it has desirable sensory characteristics. However, it is a highly perishable and fragile fruit. Therefore, strawberry processing is an essential and interesting strategy to add value, produce products and increase shelf life, reducing post-harvest losses (Bhat & Stamminger, 2015).

Osmotic dehydration (OD) is a unitary operation performed by immersing the food in a hypertonic solution; mass transfers occur between the osmotic solution and the food due to the osmotic pressure gradient (González-Pérez et al., 2021; Junqueira et al., 2017). During OD, the solute impregnation has made this process widely used to promote fruit enrichment with beneficial health solutes (Macedo, da Silva Araújo, et al., 2021). Among the various solutes that can be used in OD, isomaltulose, also known as palatinose, is a carbohydrate that has aroused much interest due to the low cariogenic index, contributing to dental health, and the low glycemic index, minimizing glycemic peaks and insulin production, being an attractive feature for athletes and diabetics (Sawale et al., 2017).

The reduction of the moisture content during OD is insufficient to guarantee the stability of the osmo-dehydrated product for long periods. Several drying methods can be applied after OD (Ramya & Jain, 2017), being heated air drying (HAD) the most used one. It consists of exposing the wet material to heated air (Araújo et al., 2020). However, HAD has some limitations that led to the implementation of new drying technologies, such as microwave drying (Junqueira et al., 2017).

Microwave drying consists of wet material absorbing microwave energy and converting it to heat. In foods, the heat is generated mainly by dipolar and ionic mechanisms; these tend to be oriented by the oscillating electric field generated by microwaves, resulting in the volumetric heating of the material (Kumar & Karim, 2019). This is an interesting method for drying fruits due to its high drying rate, low energy consumption, and preservation of heat-sensitive compounds. However, continuous product exposure to microwaves can generate overheating (Huang et al., 2021).

Intermittent microwave drying (IMD) is a discontinuous process in which the magnetron is turned on and off periodically. This allows moisture and temperature to be evenly distributed

during the process, preventing overheating of the food. The IMD has lower energy consumption than heated air drying, and its use is associated with the production of dried foods of suitable physical, nutritional and chemical qualities (Huang et al., 2021; Junqueira et al., 2017; Keser et al., 2020; Mounir et al., 2020; Tepe & Tepe, 2020). In this context, this study aimed to evaluate the drying kinetic, energy consumption, and quality parameters of fresh and isomaltulose enriched strawberry dried by intermittent microwave drying and heated air drying.

2 MATERIAL AND METHODS

2.1 Raw material

Fresh strawberries (*Fragaria x ananassa* cv. Camino Real) were purchased from the local market (Lavras, MG, Brazil) and selected for color, integrity, and maturation stage. The fruits were washed in running water, sanitized with chlorinated water (200 ppm for 10 min), rinsed, drained with absorbent paper, and stored at 4 °C for up to 2 days. The fruit skin was removed. Then, the inside of the strawberries was cut into 10 mm cubes.

The fresh cubes had a moisture content of $90.31 \pm 0.85\%_{w.b.}$. The moisture content was determined by the gravimetric method established by method 934.06 of AOAC (2010), wherein the samples were placed in an oven at 70 °C, under vacuum.

2.2 Osmotic pre-treatment

The strawberry cubes were immersed in an osmotic solution of 35% ($w w^{-1}$) of isomaltulose (Beneo-Palatinit, Germany), in the proportion of 1:20 ($w w^{-1}$, fruit:osmotic solution), at 25 °C, for 300 min. The osmotic process was carried out at 160 mbar for the first 20 min, followed by atmospheric pressure until completing the total time. This process is called pulsed vacuum osmotic dehydration and is based on removing occlude gases in the interior of the samples (Junqueira et al., 2017). Then, the samples were removed from the osmotic solution and immersed in an ice bath for 10 s to interrupt the mass flow. The sample surfaces were drained with absorbent paper.

The conditions under which the osmotic process was carried out were determined in previous tests to obtain the maximum mass transfer rates. In the chosen condition, the isomaltulose impregnation was $8.00 \pm 0.16\%$. After the osmotic pre-treatment, the strawberries showed a moisture content of $76.56 \pm 1.02\%_{w.b.}$.

2.3 Drying procedures

Fresh (non-osmotically pre-treated) and osmotically pre-treated strawberries were subjected to intermittent microwave drying (IMD) and heated air drying (HAD). Each drying was performed using 60.02 ± 0.69 g of sample. During drying, the sample masses were measured at 5 min intervals. Drying processes were completed when the samples had a moisture content of $10.28 \pm 0.12\%$ w.b..

2.3.1 IMD

The IMD was performed in a hexagonal microwave drying cavity, 12 cm wide, 24.5 cm high. The equipment had a 12 cm long WR340 waveguide centered on one of the walls, magnetron 2M319J, perforated cover to allow the passage of drying air through the cavity (da Costa et al., 2021). A fan was attached to the cavity base to promote continuous air circulation at 0.5 m s^{-1} at room temperature ($22.89 \pm 1.17 \text{ }^\circ\text{C}$). A layer of silica was used to decrease the relative moisture of the air.

The samples were placed in an 18.5 cm diameter perforated circular sample holder, positioned 3.0 cm high and in the center of the cavity. The cavity was closed. The sample holder was attached to a digital scale (Ohaus Adventurer, ARC 120, USA) to monitor the sample mass during the process.

The sample temperature was continuously measured with a type K thermocouple inserted in a strawberry cube. The microwave magnetron was triggered to heat the samples to the setpoint temperature (60°C). Upon reaching the setpoint temperature, the magnetron was automatically turned off. With air circulation at room temperature, the sample temperature decreased below the setpoint, causing the magnetron to fire again. The ON/OFF system allowed keeping the sample temperature close to the setpoint temperature throughout the process (da Costa et al., 2021). Operating parameters were controlled by a system developed using a NI-USB6009 board and LabView software.

2.3.2 HAD

HAD was performed in a tunnel dryer (Eco Engenharia Educacional, MD018 model, Brazil), with the airflow at $60 \text{ }^\circ\text{C}$ and 0.5 m s^{-1} . The samples were placed in an 18.5 cm diameter perforated circular sample holder. A digital scale (Ohaus Adventurer, ARC 120, USA) coupled to the sample holder monitored the mass of the samples during drying.

2.4 Drying rate (DR)

The DR was determined according to the moisture content_{d.b.} according to Eq. (1) (Macedo et al., 2020; Macedo, Corrêa, et al., 2021).

$$DR = \frac{X_t - X_{t+\Delta t}}{\Delta t} \quad (1)$$

where DR is the drying rate (kg water kg dry solid⁻¹ min⁻¹), X_t and $X_{t+\Delta t}$ are the moisture content (kg of water kg of dry sample⁻¹) at t and $t+\Delta t$, respectively, t is the time (min) and Δt is the time difference (min).

2.5 Energy consumption

The electrical energy consumed during drying was measured with an ammeter (Hikari, HA-266 model, Shenzhen, China). The energy consumption required to remove 1 kg of water was calculated according to Eq. (2).

$$EC = \frac{EC_t}{m_0 - m_f} \quad (2)$$

where EC is the energy consumption per kg of water, EC_t is the total energy consumption (kWh) of each drying, m_0 and m_f are the sample mass (kg) at the beginning and end of drying, respectively.

2.6 Quality analysis

The fresh and dried samples were characterized in terms of water activity, shrinkage, hardness, color, total anthocyanins, total phenolics, and antioxidant capacity.

2.6.1 Water activity (a_w)

The a_w of the samples was determined on an electronic hygrometer (Aqualab, series 3TE, Washington, USA) at 25 °C.

2.6.2 Shrinkage

The shrinkage of the dried samples was determined by the toluene displacement method (Dehghannya et al., 2018) and calculated according to Eq. (3).

$$\text{Shrinkage (\%)} = \left(1 - \frac{V}{V_0}\right) \times 100 \quad (3)$$

where V and V_0 are the volumes (m³) of dried and fresh samples, respectively.

2.6.3 Hardness

The hardness was measured using a texture analyzer (Stable Micro Systems, TA-X2T, Surrey, England), equipped with a 50 kg load cell, a 6 mm diameter probe, test speed of 2 mm s⁻¹, and penetration distance of 3 mm. The response was expressed in Newton (N).

2.6.4 Color

The colorimetric parameters of the samples were obtained with the aid of a colorimeter (Konica Minolta, model CR-300, Osaka, Japan), with illuminant D65 and 10° viewing angle, using color scale CIELab. The L* parameter represents the lightness, ranging from 0 (black) to 100 (white). C* indicates chromaticity or color saturation. The °h represents the hue of the sample, where the angles of 0° (or 360°), 90°, 180°, and 270° represent red, yellow, green, and blue angles, respectively. The total color difference (ΔE) of the samples concerning the fresh sample whose parameter is represented by the sub-index “0” was calculated according to Eq. (4).

$$\Delta E = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2} \quad (4)$$

2.6.5 Total anthocyanins content (TAC)

The extract was prepared by mixing 2g in 20 mL of acidified ethanol, using HCl (0.1%). The mixture was centrifuged at 25400 xg for 15 min. TAC was quantified by the differential pH method, according to Eq. (5) (Giusti & Wrolstad, 2001). The result was expressed in mg of cyanidin 3-glycoside per g sample_{d.b.}.

$$\text{TAC} \left(\frac{\text{mg}}{100\text{g}} \right) = \left(\frac{((A_{510 \text{ nm}} - A_{700 \text{ nm}})_{\text{pH}1} - (A_{510 \text{ nm}} - A_{700 \text{ nm}})_{\text{pH}4.5}) \times \text{MW} \times \text{DF}}{\varepsilon} \right) \quad (5)$$

where MW is the molecular weight of cyanidin 3-glycoside (433.0); DF is the dilution factor; ε is the molar absorptivity (22400).

2.6.6 Total phenolics content (TPC)

The extract for quantification of TPC was prepared by mixing 5g in 40 mL of 50% methanol (v v⁻¹). The homogenized mixture was left to stand for one hour, protected from light, at room temperature. The mixture was centrifuged at 25400 xg for 15 min, and the supernatant was collected. The residue from the centrifugation was mixed with 40 mL of 70% acetone (v v⁻¹) and left to stand for one hour, protected from light, at room temperature. The mixture was centrifuged at 25400 xg for 15 min. The supernatant was collected and mixed with the first. The final volume was completed with deionized water up to 100 mL (Rufino et al., 2010). The extracts were kept at -20 °C until the analysis was carried out.

TPC was quantified using the Folin-Ciocalteu method (Waterhouse, 2003), and the result was expressed in mg of gallic acid per 100 g sample_{d.b.}.

2.6.7 Antioxidant capacity (AC)

The same extract prepared for the TPC analysis was used for the AC analysis (Rufino et al., 2010; Vimercati et al., 2020). AC was quantified by the ferric reducing antioxidant power capacity (FRAP) and 2,2-diphenyl-2-picryl-hydrazyl (DPPH) methods. The results were expressed in μmol of ferrous sulfate per g of sample_{d.b.}, and g sample_{d.b.} per g of DPPH (Rufino et al. 2010).

2.7 Statistical analysis

The experiments were conducted in a completely randomized design, in a 2×2 complete factorial scheme (Table 1). Data were evaluated by ANOVA. In the case of significant effects, the means were compared using the Tukey test. Each treatment was compared individually with the control (fresh) sample by the Dunnett test.

Table 1 – Experimental conditions

Code	Osmotic pre-treatment	Drying method
Fresh	No	None
IMD	No	IMD
HAD	No	HAD
OP-IMD	Yes	IMD
OP-HAD	Yes	HAD

IMD: intermittent microwave drying; HAD: heated air drying; OP: osmotic pre-treatment.

Statistical analyses were performed using the 5% probability level, using the Statistica software (Statsoft, Tulsa, USA).

3 RESULTS AND DISCUSSION

Dried strawberry samples, non- or osmotically pre-treated, by IMD or HAD, are shown in Fig. 1.



Fig. 1 – Non- and osmotically pre-treated (OP) dried strawberries using IMD and HAD methods.

3.1 Drying

3.1.1 Drying kinetic

Reductions in strawberry moisture content during the drying processes are shown in Fig. 2a. Initially, non-osmotically pre-treated (fresh) and osmotically pre-treated strawberries had a moisture content of 90.31%_{w.b.} and 76.56%_{w.b.}, respectively. Moisture contents reduced exponentially throughout the drying (Fig. 2a), as is characteristic of the drying of fruits (Dehghannya et al., 2018; Junqueira et al., 2017; Macedo et al., 2020; Macedo, Corrêa, et al., 2021; Macedo, da Silva Araújo, et al., 2021; Tepe & Tepe, 2020).

A relevant reduction of moisture content was observed in the first minutes of drying of non-pre-treated strawberries, especially by the IMD method (Fig. 2a). This is due to the high moisture content of the fruit at the beginning of the process, presenting a large amount of free water that can be easily removed (Huang et al., 2021). Concerning the pre-treated samples, the rate of reduction of moisture content with time was more slightly. It is due to the water loss and the interaction of moisture with the incorporated solid.

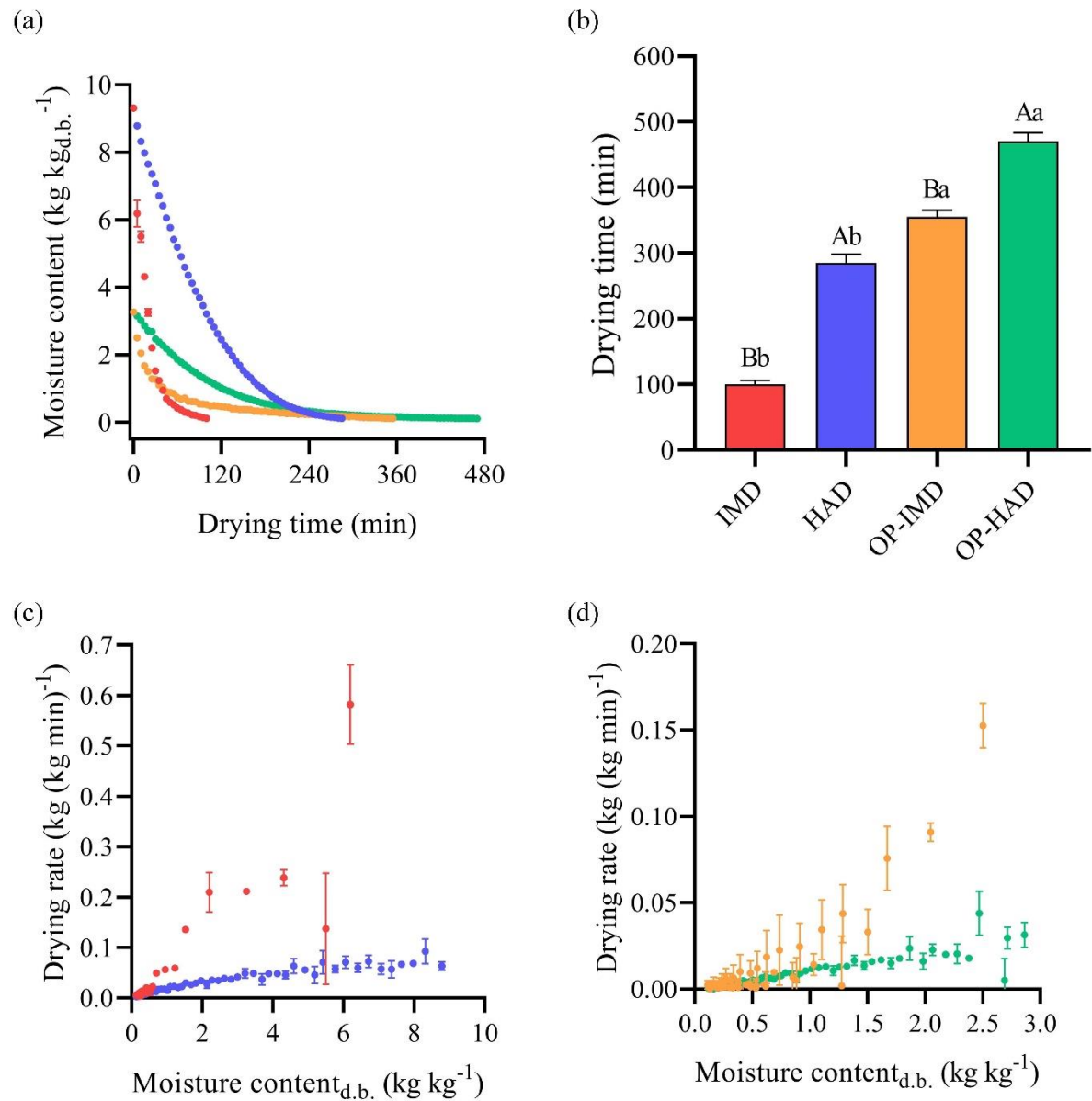


Fig. 2 – Kinetic of moisture content, on a dry basis (a), drying time (b), and drying rate of non-osmotically pre-treated (c) and osmotically pre-treated (d) samples.

Red: IMD; blue: HAD; orange: OP-IMD; green: OP-HAD. Values are mean±standard deviation, n=3. The same uppercase letter indicates no significant difference between IMD and HAD samples. The same lowercase letter indicates no significant difference between non- and osmotically pre-treated samples by the Tukey test ($p < 0.05$).

In just 50 min, drying by IMD managed to reduce the moisture content of the non-pre-treated strawberry by 90.31%_{w.b.} to 37.19%_{w.b.}, whereas, at the same drying time, the sample had 85.23%_{w.b.} when dried by HAD (Fig. 2a). This highlights the IMD method is due to the volumetric heating generated mainly by the agitation of water molecules (bipolar rotation) due to the electric magnetic field, leading to a high water vapor pressure gradient between the internal and surface of the wet material, accelerating the transfer of moisture (Huang et al.,

2021; Junqueira et al., 2017; Kumar & Karim, 2019; Zielinska et al., 2020). In addition to resulting in a large water evaporation capacity, the intermittent system (power on/power off mode) minimizes the risk of sample overheating, which would result in the degradation of fruit compounds or even burning (Dehghannya et al., 2018; Zielinska et al., 2020).

The drying processes required from 100 min to 470 min to reduce the moisture content of the samples to 10.28%_{w.b.} (Fig. 2a and 2b). Similar drying times were found in other studies when drying strawberries (Changrue et al., 2008; Gamboa-Santos et al., 2014; Gamboa-Santos & Campañone, 2019; Méndez-Lagunas et al., 2017). For non- and osmotically pre-treated strawberry drying, IMD was the method that required less time to complete the process. IMD was faster than HAD by 64.91% and 24.47% for drying non- and osmotically pre-treated strawberries, respectively (Fig. 2b). The same was observed in other studies (Huang et al., 2021; Tepe & Tepe, 2020). As discussed earlier, IMD results in volumetric heating of the material. On the other hand, drying by the HAD method is slow, as, initially, the surface of the material is heated by receiving heat from the heated air, by convection, starting the evaporation of surface water. Furthermore, there is also the conduction of heat from the surface to the interior of the material, but it is a slow mechanism, which contributes to the delay in drying. Subsequently, moisture moves from the interior to the surface of the material by mainly diffusion mechanisms due to a concentration and temperature gradient between the surface and the interior (Dehghannya et al., 2018; Huang et al., 2021; Omolola et al., 2017).

The application of osmotic pre-treatment increased the drying time for both drying methods, as shown in Fig. 2b. The IMD method, including osmotic dehydration as a pre-treatment, prolonged the drying time by 3.55 times and 1.65 times by the HAD method. Osmotic dehydration promoted a partial reduction in moisture content, as observed in the present study (90.31 to 76.56%_{w.b.}). Reducing the volume of moisture in a material to be removed by complementary drying would facilitate the completion of this process. However, during the osmotic process, the solute is incorporated from the solution to the material, increasing the interaction of water with the material and decreasing the diffusivity of water, resulting in increased drying time. This same effect of osmotic dehydration on convective drying time was also observed in other studies (An et al., 2019; Lyu et al., 2017; Macedo, da Silva Araújo, et al., 2021).

3.1.2 Drying rate (DR)

The DR of non- and osmotically pre-treated strawberries are shown in Fig. 2c and 2d, respectively. At the beginning of the drying, the DR was higher than the end of the processes due to the large volume of water present in the samples initially (Macedo, Corrêa, et al., 2021).

The DR showed decreasing behavior during most of the drying, with short periods of DR close to constant. Periods of constant rate were more frequent in HAD, where the evaporation rate of water from the surface of the sample was similar to diffusion within the material to the surface (Bhandari, 2006).

Among the four drying treatments, the DR of strawberry non-pre-treated by IMD stood out, presenting the highest values during the process. In both drying methods, the DR of osmotically pre-treated strawberries was lower than that of non-pre-treated strawberries due to the greater volume of water in the non-pre-treated samples. The DR by the IMD method was higher than by the HAD, mainly at the beginning of the drying. This is due to the volumetric heating capacity carried out by the electromagnetic field, causing greater water evaporation.

3.13. Sample temperature during IMD

The temperature records of the non- and osmotically pre-treated strawberry during the IMD drying are presented in Fig. 3a and 3b, respectively.

Sample temperatures oscillated within a small range, as commonly occurs in IMD (Khan et al., 2020; Pham et al., 2020), in which non- and osmotically pre-treated strawberries had mean values very close to the setpoint temperature (60 °C), as shown in Table 2.

There were high peaks in sample temperature for a short period, between times 4.8- and 6.5-min during drying of the non-pre-treated sample (Fig. 3a). This may be because non-pre-treated strawberries initially present high free water content, causing increased agitation of water molecules by microwaves, resulting in rapid heating of the sample. On the other hand, high peaks did not occur in the drying of the pre-treated sample (Fig. 3b), as initially, the pre-treated sample had a lower free water content due to water loss during the osmotic process and the interactions of the water with the incorporated solids.

Sample temperature fluctuated during IMD is due to the on/off system. When drying starts, the magnetron is turned on, causing the material to heat rapidly (Fig. 3a and 3b), as reported in other studies (Khan et al., 2020; Pham et al., 2020). In addition to the fact that the waves generate volumetric heating, the small initial size of the cubes (10 mm) favors the rapid heating of the material. As soon as the material reaches the setpoint temperature, the magnetron is automatically turned off. The strawberries cool down due to air circulation at room

temperature (22.89 ± 1.17 °C) and water evaporation. When the sample has a temperature lower than the setpoint, the magnetron is switched on again.

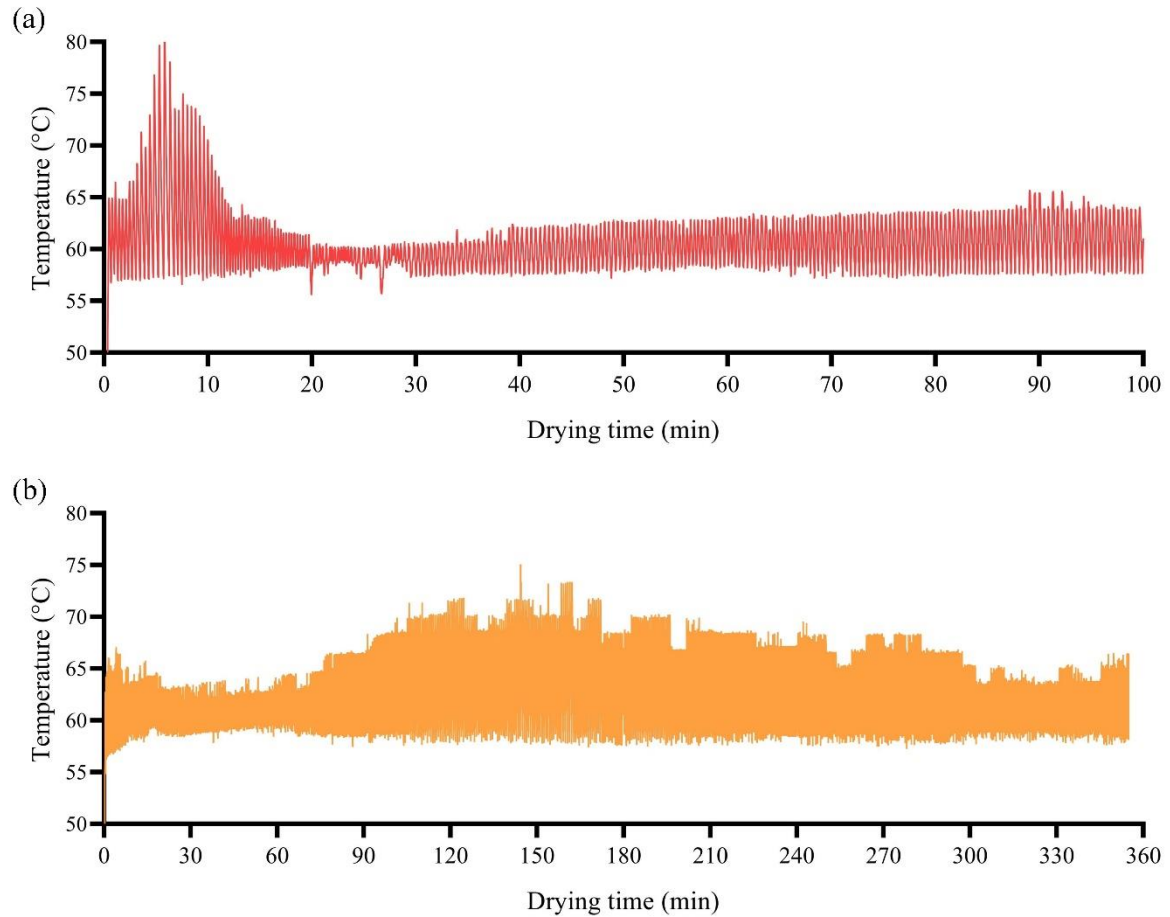


Fig. 3 – Temperature of non-osmotically pre-treated (a) and osmotically pre-treated (b) samples during IMD.

Table 2 – Sample temperature, percentage of magnetron on and off time during IMD and OP-IMD

Code	Sample temperature (°C)	On (%)	Off (%)
IMD	60.542 ± 3.312 a	46.11 ± 1.80 a	53.89 ± 1.80 b
OP-IMD	61.685 ± 3.153 a	25.87 ± 0.61 b	74.13 ± 0.61 a

Values are mean \pm standard deviation, $n=3$. The same lowercase letter indicates no significant difference between non- and osmotically pre-treated samples by the Tukey test ($p < 0.05$).

The peak and base points reflect the heating times and the tempering times (Fig. 3a and 3b), respectively; they indicate the times when the microwave is turned on and off, respectively

(Pham et al., 2020). Table 2 shows the percentage of time the magnetron was on and off for both IMD. For both the non-strawberry and the osmotically pre-treated strawberry, the magnetron remained off for most of the drying time, especially when drying the pre-treated strawberry, where the magnetron remained off for approximately $\frac{3}{4}$ of the time.

3.1.4 Energy consumption

Drying is a unit operation known as an energy-intensive process, especially methods that use heated air (Khan et al., 2020). Therefore, the evaluation of this response becomes very important, aiming to minimize it. The energy consumption for each drying is shown in Fig. 4. To complete the drying, 155.45 to 1203.64 kWh kg of water⁻¹ removed were required. Similar results were found in other studies (Huang et al., 2021).

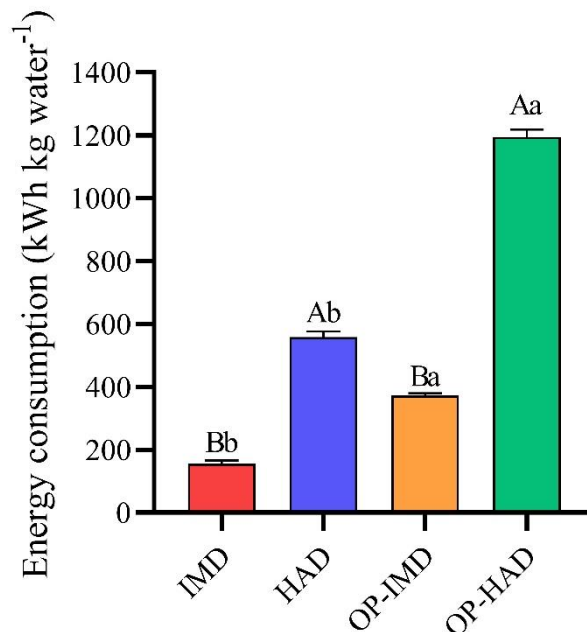


Fig. 4 – Energy consumption at different drying conditions.

Values are mean±standard deviation, n=3. The same uppercase letter indicates no significant difference between IMD and HAD samples. The same lowercase letter indicates no significant difference between non- and osmotically pre-treated samples by the Tukey test (p<0.05).

Microwave drying had lower energy consumption than HAD (Fig. 4), 73.16% and 68.99% lower in drying non- and pre-treated strawberries, respectively. This is due to the intermittent system (on/off), in which the magnetron was off for most of the drying time, as discussed above. Other studies also reported that the on/off system favors the reduction of

energy consumption (Cao et al., 2019; Huang et al., 2021). Furthermore, the convective method requires heating a large amount of air, resulting in high energy demand. Comparing the drying of non- and pre-treated strawberries, the second required a greater amount of energy, linked to a longer drying time due to greater difficulty in eliminating the bounded water.

3.2 Quality properties

3.2.1 Water activity (a_w)

The a_w of fresh and dried strawberries is shown in Fig. 5a. The fresh strawberry had a high a_w , characteristic of the fruit (Amami et al., 2017), indicating the high susceptibility to the growth of deteriorating microorganisms, making a fruit highly perishable, which justifies the study of processing and preservation methods of fruit.

A significant difference was not observed between the drying methods. However, non-pre-treated strawberries had a_w statistically lower than that of osmotically pre-treated strawberries (Fig. 5a).

Dried strawberries had a_w less than 0.60, which is generally the minimum value for microorganisms to grow. Therefore, at this value or below, the viability of the growth of microorganisms is minimal. The occurrence of microbial deterioration in the dried food is unlikely for up to 2 years (Jay et al., 2005), provided that the drying process, storage, transport, and transportation until the sale are carried out under hygienic conditions. Otherwise, some pathogens, yeast, and molds can continue to grow in the dried food. In addition to extending shelf life, drying fruit reduces packaging requirements and transport weight (Alp & Bulantekin, 2021).

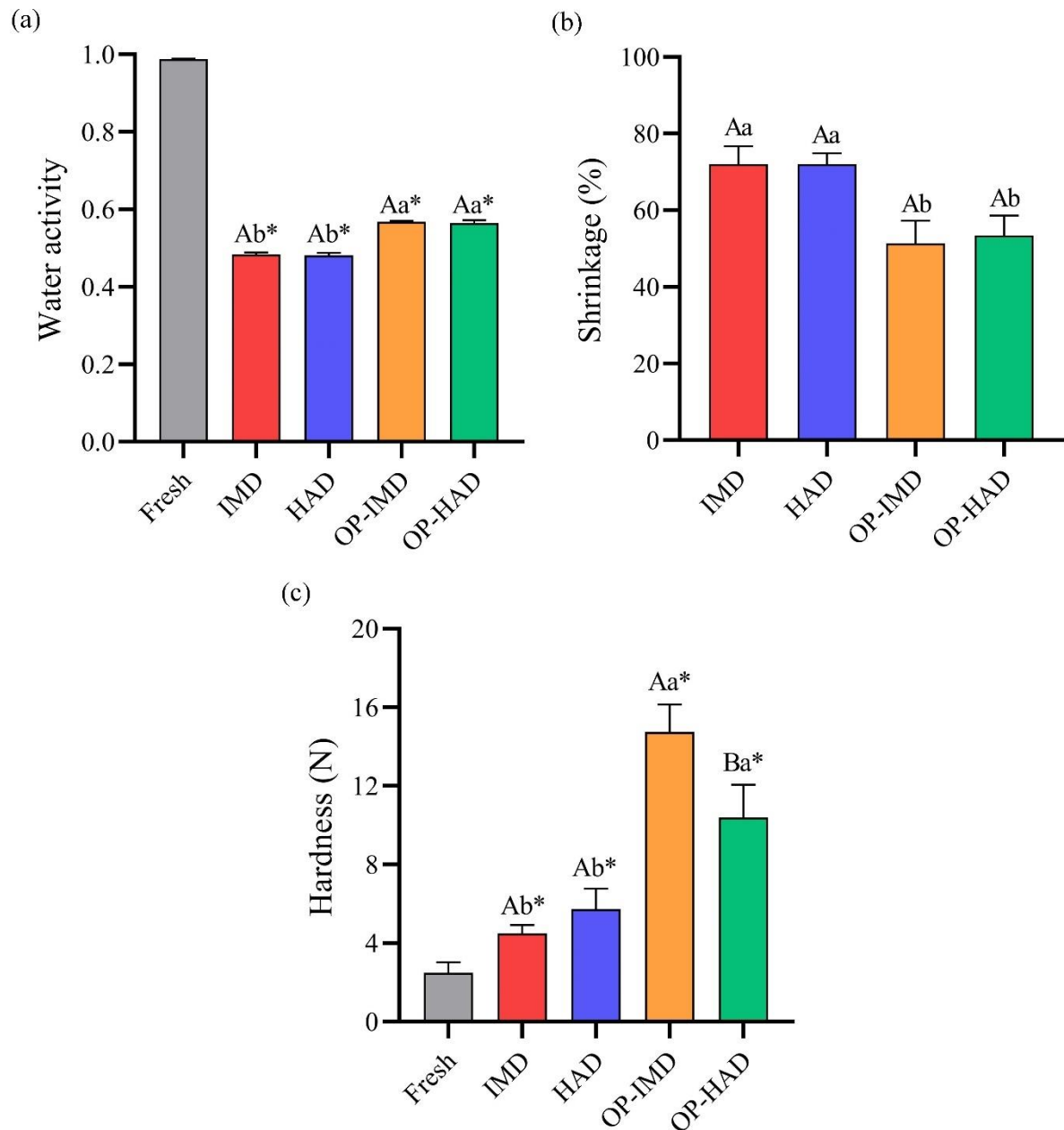


Fig. 5 – Water activity (a), shrinkage (b), and hardness (c) of the samples.

Values are mean±standard deviation, n=3. The same uppercase letter indicates no significant difference between IMD and HAD samples. The same lowercase letter indicates no significant difference between non- and osmotically pre-treated samples by the Tukey test ($p<0.05$). The asterisk indicates a significant difference between each treatment and the fresh sample by the Dunnett test ($p<0.05$).

3.2.2 Shrinkage

Shrinkage is an inherent physical alteration in drying processes. However, it is undesirable and representing an important quality parameter in dried fruit. In addition to the

negative aspect of product appearance, shrinkage prejudices moisture removal during drying and heat transfer (Dehghannya et al., 2018).

As shown in Fig. 5b, strawberries shrank from 51.72 ± 7.90 to $72.41 \pm 5.97\%$. Between the drying methods, there was no significant difference. Some studies reported that the use of microwaves could minimize shrinkage when drying fruits (Raghavan & Silveira, 2001; Tepe & Tepe, 2020), but this effect was not observed in the present study. However, osmotic pre-treatment minimized strawberry shrinkage (Fig. 5b), as also reported by other authors (Changrue et al., 2008; Raghavan & Silveira, 2001). This is due to the isomaltulose impregnation, contributing to the maintenance of the physical structure of the material (Changrue et al., 2008).

During drying, the sample volume reduction results from the material leaving the water, especially in materials such as strawberries, in which water represents a significant portion of its composition. According to Raghavan and Silveira (2001), strawberry shrinkage during drying has a linear relationship with moisture ratio.

For all dried samples, it was observed that the shrinkage was anisotropic due to deformations caused in the microstructure of the food, distorting the strawberry.

3.2.3 Hardness

The hardness of fresh and dried strawberries is shown in Fig. 5c. Fresh strawberries had a low hardness value, being one of the indicators of strawberry fragility. Dried strawberries had greater hardness than fresh strawberries, contributing to greater physical resistance of the material. However, the increase in material hardness due to drying must be controlled as products with high hardness may not be well accepted by consumers (Lyu et al., 2017).

Non-pre-treated dried strawberries showed no significant difference between both drying methods. On the other hand, between osmotically pre-treated strawberries, IMD resulted in dried strawberries with greater hardness than dried by HAD. Furthermore, pre-treated strawberries had higher hardness than non-pre-treated strawberries, regardless of the drying method (Fig. 5c).

3.2.4 Color

The colorimetric parameters of fresh and dried strawberries are shown in Fig. 6.

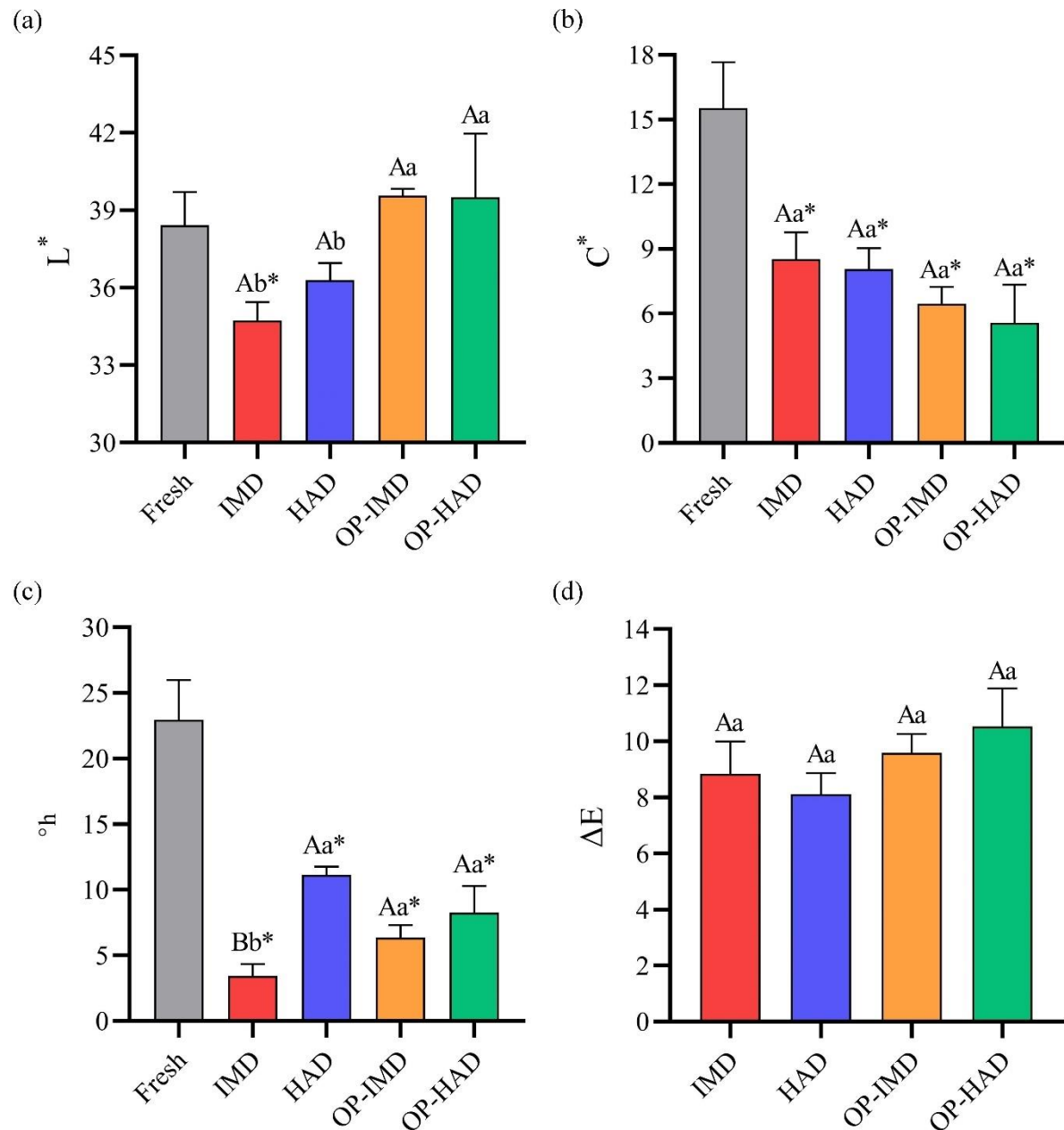


Fig. 6 – Colorimetric parameters of fresh and dried strawberries.

Values are mean \pm standard deviation, n=3. The same uppercase letter indicates no significant difference between IMD and HAD samples. The same lowercase letter indicates no significant difference between non- and osmotically pre-treated samples by the Tukey test ($p < 0.05$). The asterisk indicates a significant difference between each treatment and the fresh sample by the Dunnett test ($p < 0.05$).

The L^* parameter of fresh strawberry was equal to that of dried strawberries, except the IMD-non-pre-treated dried strawberry (Fig. 6a). Between the drying method, there was no difference in L^* value. However, the osmotic pre-treatment resulted in strawberries with higher

L^* , indicating that these samples were lighter than the non-pre-treated ones, as shown in Fig. 1. This is due to the leaching of pigments during the osmotic process.

The C^* of fresh and dried strawberries is shown in Fig. 6b. Fresh strawberries had a higher C^* value than dried strawberries. Furthermore, no statistical difference was observed among dried strawberries.

Fig. 6c shows the $^{\circ}h$ values for the samples. Dried strawberries had lower $^{\circ}h$ values than fresh strawberries, indicating a greater red hue (0° or 360°). This is due to the higher concentration of pigments in the dried strawberry, making them redder than the fresh strawberry cube. Among the dried strawberries, the non-pre-treated dried by IMD had the lowest $^{\circ}h$ value, indicating a greater red hue. This can also be seen in Fig. 1.

The ΔE was not significantly influenced by the drying method or the osmotic process. The ΔE values were between 8.11 ± 0.74 and 10.53 ± 1.35 , that is, $\Delta E > 6.0$, which indicates that the dried strawberries showed a great color difference from the fresh strawberry (Pathare et al., 2013). Similar results were observed by Amami et al. (2017).

The color of the food is the first quality attribute that the consumer evaluates. Therefore, it is a characteristic that must be monitored in dried foods, as drying can cause reactions that change colors, such as pigment degradation and the formation of dark pigments (Pathare et al., 2013).

3.2.5 Total anthocyanins content (TAC)

The TAC of fresh and dried strawberries is shown in Fig. 7a. Fresh strawberry proved to be a good source of anthocyanins, with 15.02 ± 1.07 mg of cyanidin 3-glycoside per 100g, on a dry basis, being one of the fruits with the highest TAC, along with others, such as berry fruits (such as chokeberry, blackberry, blueberry), grape and currant (Tsuda, 2012).

Fresh strawberries had a higher TAC than dried ones, as both osmotic dehydration and drying processes are responsible for causing some losses of these important compounds (González-Pérez et al., 2021).

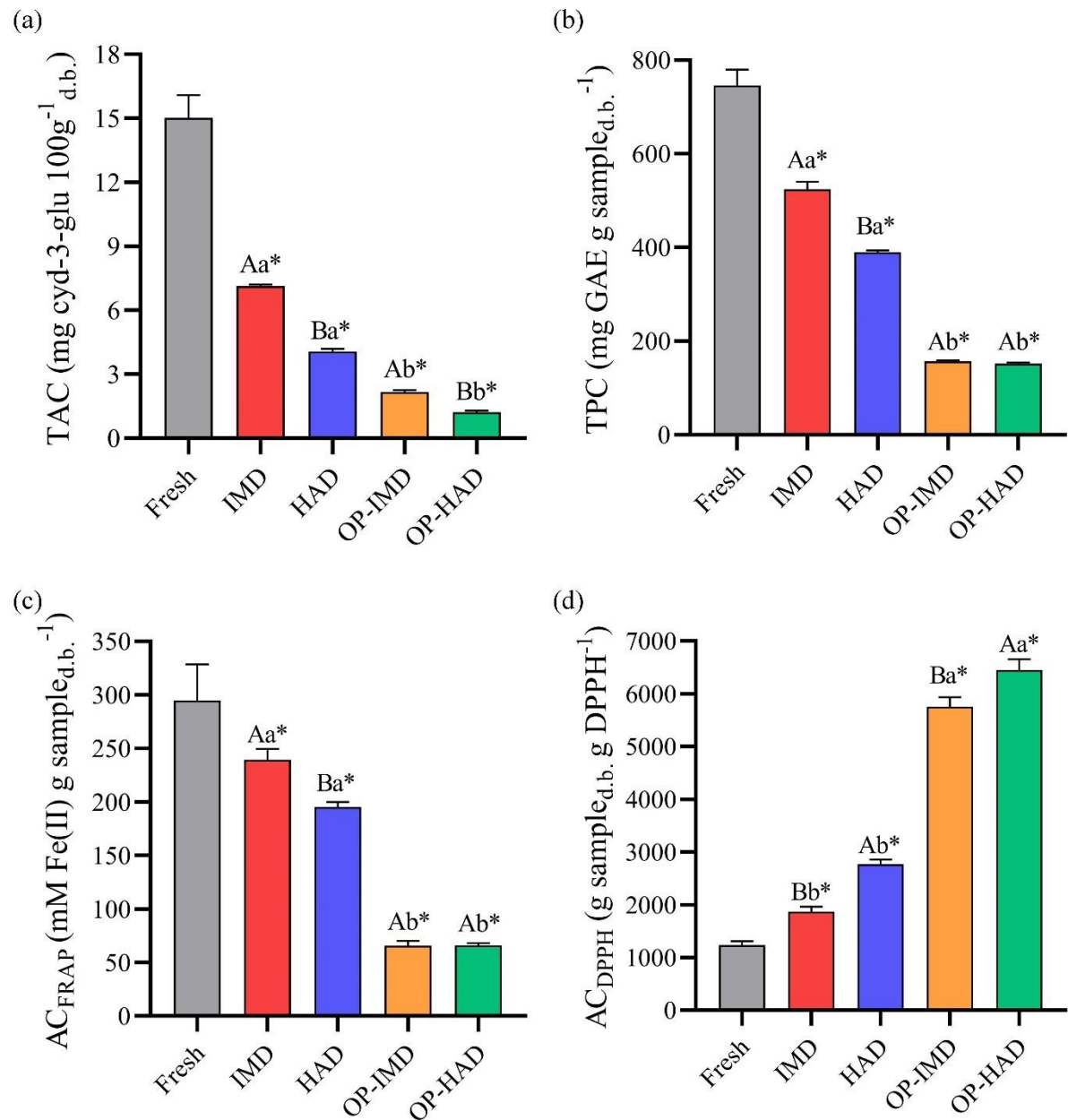


Fig. 7 – Total anthocyanins content (a), total phenolics content (b), and antioxidant capacity by FRAP (c) and DPPH (d) methods of fresh and dried strawberries.

Values are mean±standard deviation, n=3. The same uppercase letter indicates no significant difference between IMD and HAD samples. The same lowercase letter indicates no significant difference between non- and osmotically pre-treated samples by the Tukey test ($p < 0.05$). The asterisk indicates a significant difference between each treatment and the fresh sample by the Dunnett test ($p < 0.05$).

IMD produced dried strawberries with higher TAC than the HAD method for non- and pre-treated samples (Fig. 7a). As seen in Fig. 2b, the IMD required shorter drying times, which reduces the exposure time of the strawberry to high temperatures, reducing the degradation of anthocyanins.

The non-pre-treated dried strawberry had a higher TAC than the osmotically pre-treated one (Fig. 7a). In addition to the degradation of anthocyanins during drying, compounds from the food were leached to the osmotic solution during osmotic dehydration (González-Pérez et al., 2021), mainly water-soluble compounds such as anthocyanins.

Strawberry processing, transforming them into other more stable products, is necessary to ensure supply in all seasons of the year and several regions, including non-strawberry producers, and making different available products, according to the consumer preference. However, quality losses inevitably occur during strawberry processing, especially bioactive compounds such as anthocyanins (Basu et al., 2014; Bhat & Stamminger, 2015).

Anthocyanins are the most prominent bioactive compounds in strawberries. They confer the attractive red color of strawberries and are associated with several beneficial health effects, such as preventing cancer, inflammation, and cardiovascular and neurodegenerative diseases. However, they are susceptible compounds easily degraded by oxidative processes, especially when exposed to high temperatures, such as in drying (Méndez-Lagunas et al., 2017). Retentions of up to 47.46% were obtained in the present study. Although nutritional losses have occurred, dried fruits can be considered essential sources of vitamins, minerals, fiber, and bioactive components or phytochemicals, due to the concentration of compounds concerning fresh fruit (Omolola et al., 2017).

3.2.6 Total phenolics content (TPC)

The TPC of fresh and dried strawberries is shown in Fig. 7b. Fresh strawberry had an important TPC (745.88 ± 33.67 mg of gallic acid equivalent per g of strawberry, on a dry basis), as also reported in other studies (Aaby et al., 2012). Fresh strawberries had higher TPC than dried ones. This is explained by the fact that the osmotic process and drying cause the loss of these compounds. However, good TPC retention capacities were obtained in dried strawberries. Dried strawberry, non-pre-treated, showed retention of 70.26% by the IMD method. Retention of 52.23% was obtained by the HAD method. A similar result was observed by Amami et al. (2017).

Osmotically pre-treated strawberries had lower TPC than non-pre-treated dried strawberries. Furthermore, a difference in TPC of pre-treated strawberries was not observed between drying methods (Fig. 7b).

The group of phenolics present in strawberry is rich, formed by several compounds; there are anthocyanins, tannins, esters of hydroxycinnamic acids, especially p-coumaric acid, and ellagic acid and ellagic acid glycosides (Aaby et al., 2012). These compounds perform

several beneficial functions to the organism. Therefore, the preservation of these compounds must be sought.

3.2.7 Antioxidant capacity (AC)

AC by FRAP and DPPH methods are shown in Fig. 7c and 7d, respectively. The FRAP method expresses AC per sample mass. However, the determination of AC by the DPPH method expresses the sample mass required to reduce the DPPH mass. Therefore, low response values by the DPPH method indicate high sample AC (Vimercati et al., 2020).

Fresh strawberries showed high antioxidant capacity by both methods (FRAP and DPPH), with higher values than dried strawberries.

Strawberry dried by the IMD method, non-pre-treated, presented the highest AC, having an AC, by the FRAP method, of 81.18% concerning fresh strawberry, showing that this drying method is quite efficient in preserving antioxidant compounds. A possible explanation for this occurrence is the short drying time, which reduces the time the material is exposed to heating. Keser et al. (2020) also observed a good ability to preserve the antioxidant capacity of carrots when performing the IMD.

By the FRAP method, the AC of pre-treated strawberries dried by IMD and HAD were statistically equal. On the other hand, the AC by the DPPH method showed a higher value by the HAD; that is, the IMD resulted in dried strawberries with higher AC.

The AC of the osmotically pre-treated dried strawberries was lower than that of the non-pre-treated ones by both methods of AC determination. This may be associated with compound leaching during the osmotic process, as discussed above.

4 CONCLUSION

IMD and HAD were efficient methods for producing non- and osmotically pre-treated dried strawberries with good nutritional quality and low moisture and water activity, providing stability to the dried product. The principal component analysis efficiently helped the distinction of treatments and association of the evaluated responses.

IMD required less drying time than HAD. The magnetron was off for a good percentage of time during IMD, resulting in low power consumption. In addition, IMD contributed to the preservation of anthocyanin, phenolic and antioxidant compounds.

The osmotic process could be used for impregnation in strawberries with significant values. It also leads to low shrinkage. However, increased drying time, some nutritional losses,

increased hardness, and color change in dried strawberries were attributed to osmotic dehydration.

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