UNIVERSIDADE FEDERAL DE LAVRAS

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VALORIZATION OF PINEAPPLE POMACE FOR FOOD AND FEED: EFFECTS OF PRE-TREATMENT WITH ETHANOL ON CONVECTIVE DRYING AND QUALITY PROPERTIES

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Dissertação apresentada à Universidade Federal de Lavras, como parte das exigências do Programa de Pós-Graduação em Ciência dos Alimentos, para a obtenção do título de Mestre.

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VALORIZAÇÃO DO BAGAÇO DE ABACAXI PARA ALIMENTOS E RAÇÃO: EFEITOS DO PRÉ-TRATAMENTO COM ETANOL NA SECAGEM CONVECTIVA E PROPRIEDADES DE QUALIDADE

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Aprovada em 07 de julho de 2021.

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LAVRAS - MG

2021

A todos aqueles que, de alguma forma estiveram comigo, fazendo esta vida valer cada vez mais a pena.

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“Somewhere, something incredible is waiting to be known.”

(Carl Sagan)

ABSTRACT

There is a rising trend of valorizing fruit by-products for different industries. The pineapple pomace is an interesting ingredient for improving different food properties, or for application as animal feed. However, the majority of the works use fresh fruit by-products, which are high-moist, high perishable and unstable, being this approach questionable from an industrial perspective. Therefore, this work evaluated the drying process of pineapple pomace as an alternative to increase its stability. The emergent pre-treatment with ethanol was studied, evaluating both the process parameters and the quality of the final products. The pineapple pomace was obtained as a residue of juice extraction, being characterized as high moisture and fibrous nature, with little structural organization. The pre-treatment consisted of spraying ethanol on the samples in concentrations of 5 and 10%, and convective drying was carried out at 40 and 60 °C and 1 m · s^{-1}. Thermal images were obtained during processing, and the different products were evaluated through total phenolic content, antioxidant capacity, water absorption capacity and oil absorption capacity. The temperature had a great influence on the drying kinetics. On the other hand, ethanol did not affect drying time, but reduced the product temperature during processing. A possible limitation of ethanol as dryer accelerator was discussed in relation to the structure of the food. In addition, drying did not affect the bioactive compounds nor technological properties, demonstrating to be a good alternative to increase the stability of pineapple pomace, without compromising its quality, being thus an interesting ingredient for application in food formulations or as animal feed.

Keywords: By-product. Preservation. Drying. Ethanol. Quality.

RESUMO

Há uma tendência crescente de valorização de subprodutos de frutas para diferentes indústrias. O bagaço de abacaxi é um ingrediente interessante para melhorar diferentes propriedades dos alimentos, ou para aplicação como ração animal. No entanto, a maioria dos trabalhos utiliza os subprodutos de frutas frescas, que são altamente úmidos, altamente perecíveis e pouco estáveis, sendo esta abordagem questionável do ponto de vista industrial. Portanto, este trabalho avaliou o processo de secagem do bagaço de abacaxi como uma alternativa para aumentar sua estabilidade. Foi estudado o pré-tratamento emergente com etanol, avaliando-se tanto os parâmetros do processo quanto a qualidade dos produtos finais. O bagaço de abacaxi foi obtido como resíduo da extração do suco, caracterizando-se por apresentar alta umidade e natureza fibrosa, com pouca organização estrutural. Os pré-tratamentos consistiram em pulverizar as amostras com etanol nas concentrações de 5 e 10% (m/m), e a secagem convectiva foi realizada a 40 e 60 °C e 1 $m \cdot s^{-1}$. Imagens térmicas foram obtidas durante o processamento, e os diferentes produtos foram avaliados quanto ao conteúdo fenólico total, capacidade antioxidante, capacidade de absorção de água e capacidade de absorção de óleo. A temperatura apresentou grande influência na cinética de secagem. Por outro lado, o etanol não afetou o tempo de secagem, mas reduziu a temperatura do produto durante o processamento. Uma possível limitação do etanol como acelerador de secagem foi discutida em relação à estrutura do alimento. Além disso, a secagem não afetou os compostos bioativos nem as propriedades tecnológicas, demonstrando ser uma boa alternativa para aumentar a estabilidade do bagaço de abacaxi, sem comprometer sua qualidade, sendo assim um ingrediente interessante para aplicação em formulações alimentícias ou como ração animal.

Palavras-chave: Resíduo. Preservação. Secagem. Etanol. Qualidade.

LISTA DE ILUSTRAÇÕES

LISTA DE ABREVIATURAS

AC	Antioxidant capacity
C-40 °C	Control sample dried at 40 °C
C-60 °C	Control sample dried at 60 °C
E5%-40 °C	Sample pre-treated with 5% ethanol and dried at 40 °C
E5%-60 °C	Sample pre-treated with 5% ethanol and dried at 60 °C
E10%-40 °C	Sample pre-treated with 10% ethanol and dried at 40 °C
E10%-60 °C	Sample pre-treated with 10% ethanol and dried at 60 °C
F	Fresh sample
OAC	Oil absorption capacity
TPC	Total phenolic content
WAC	Water absorption capacity

SUMÁRIO

1. INTRODUCTION

There is a rising and urgent trend of valorizing agro-industrial by-products through industrial applications. In this way, fruit by-products are interesting sources for different industries, from food (GALANAKIS, 2012) and feed (KOWALSKA et al., 2017), to biorefinery (KOUTINAS et al., 2014), the production of specific compounds (such as poly-3-hydroxybutyrate (SIROHI et al., 2021) or bio-fuels / bioethanol (GIL; MAUPOEY, 2018; GUPTA; VERMA, 2015)).

For instance, pineapple (*Ananas comosus*) pomace is a by-product obtained after pulping this fruit during the production of pulp and juices. It represents about 50% of the total weight (KETNAWA; CHAIWUT; RAWDKUEN, 2012), being composed by the peel, the core (central part) and bagasse (fibrous material which is retained in the sieves) of the fruit.

This pomace can be an ingredient for different purposes, being previously proposed in the food industry, for improve different technological or bioactive properties of extruded products (RALENG et al., 2019; SELANI et al., 2014), bakery products (DARSHINI; TERDAL; JAGADEESH, 2021; JEDDOU et al., 2017; TOLEDO et al., 2017), meat products (MONTALVO-GONZÁLEZ et al., 2018; SELANI et al., 2016b; 2016c), and gluten-free pie (CHAROENPHUN, 2019). In addition, they can also be applied as animal feed (BAIDHE et al., 2021) to improve nutrient intake, energy status and growth performance (KYAWT et al., 2020) or as a feed restriction program (VASCONCELOS et al., 2020), among others. However, pineapple pomace is in general studied directly fresh or after freeze drying (BADJONA et al., 2019; TOLEDO et al., 2019), both limited alternatives from a real industrial perspective.

In fact, the pineapple pomace has high moisture content and, consequently, is a very perishable material. This fact impairs its actual industrial application, due to storage and transport constraints. Therefore, there is the need to apply processes to increase stability, without compromising its quality.

Drying is an interesting alternative to reach those goals. However, the traditional drying approaches require long processing time and energy (DENG et al., 2019; MUJUMDAR; LAW, 2010), which are undesirable from an industrial perspective and also can jeopardizing the product quality. To avoid it, some alternatives have been used to enhance food drying, being the pre-treatment with ethanol an emerging approach (LLAVATA et al., 2020). However, few studies address the use of ethanol as pre-treatment in unstructured foods, whose drying behavior can be different. It was applied only to two unstructured products: acerola residues (SILVA et al., 2020, SILVA, DUARTE; BARROZO, 2016) and pitayas pulp foam (ARAÚJO et al., 2020; MACEDO et al., 2021). Once food structure affects processing characteristics, and *vice versa*, it is important to advance the knowledge by studying different microstructures.

Consequently, the objective of this work was to evaluate the effect of pre-treatment with ethanol on the convective drying of pineapple pomace, also analyzing the microstructure and quality properties of the final product, which was proposed as an alternative for different industrial applications. Therefore, this work was focused on two important aspects: the application of techniques for pineapple pomace preservation, promoting stability and maintaining the quality properties; and studying the effects of ethanol pre-treatment for the convective drying of a product with a different structure, in order to better understanding the mechanisms involved in this process.

2. LITERATURE REVIEW

2.1 Pineapple: fruit and by-products

Pineapple is a tropical fruit much appreciated not only for its sensory characteristics such as taste, aroma, and juiciness (ALI et al., 2020) but also for being rich in antioxidant compounds, vitamins, minerals, and other essential nutrients (MOHAMMED; EDNA; SIRAJ, 2020; ZZAMAN; BISWAS; HOSSAIN, 2021). Due to the high moisture content, pineapple requires immediate consumption or further processing, resulting in several food products with a longer shelf life.

Pineapple processing generates large amounts of by-products, consisting mainly of peel (29-42%), core (central part) (9-20%), and small amounts of crown and stem (RICO et al., 2020). The pineapple pomace is obtained after the pulp and juice production process, consisting of peel, core and bagasse (fibrous material that is retained in the sieves).

The use of pineapple pomace is extremely interesting industrially, whether in food formulations, animal feed (LOBO; DORTA, 2019) or in biorefinery (GIL; MAUPOEY, 2018; SIROHI et al., 2021).

The inclusion of pineapple pomace in food formulations can be very beneficial, for example: in meat products such as sausage, the inclusion of pineapple pomace in the formulation can improve the physicochemical properties, resulting in greater water holding capacity, greater yield, and less oxidative rancidity during storage (DÍAZ-VELA; TOTOSAUS; PÉREZ-CHABELA, 2015). On the other hand, in bakery products such as cookies, it positively influences nutritional aspects, increasing the fiber content, antioxidant capacity, and phenolic compounds (TOLEDO et al., 2019b), changes in color, higher yield, and better sensory acceptance (TOLEDO et al., 2017). In addition, the use of pineapple pomace in cookies results in lower levels of antinutrients (phytate and oxalate) and improves mineral bioavailability, especially zinc (TOLEDO et al., 2019a).

Feeding is an essential factor for animal development, and the production of feed from pineapple pomace has great benefits such as the inclusion of fibers and compounds of high nutritional value (LOBO; DORTA, 2019; STRUCK; ROHM, 2020). Yuangsoi et al. (2018) noted that supplementation of pineapple pomace extract in the Nile tilapia diet results in improved protein digestibility and accelerates fish growth. In addition, Kyawt et al. (2020) demonstrated in their study that the inclusion of pineapple pomace in the bovine diet improved nutrient intake, energy balance, and body weight gain, indicating the potential for use to improve the performance of the animals.

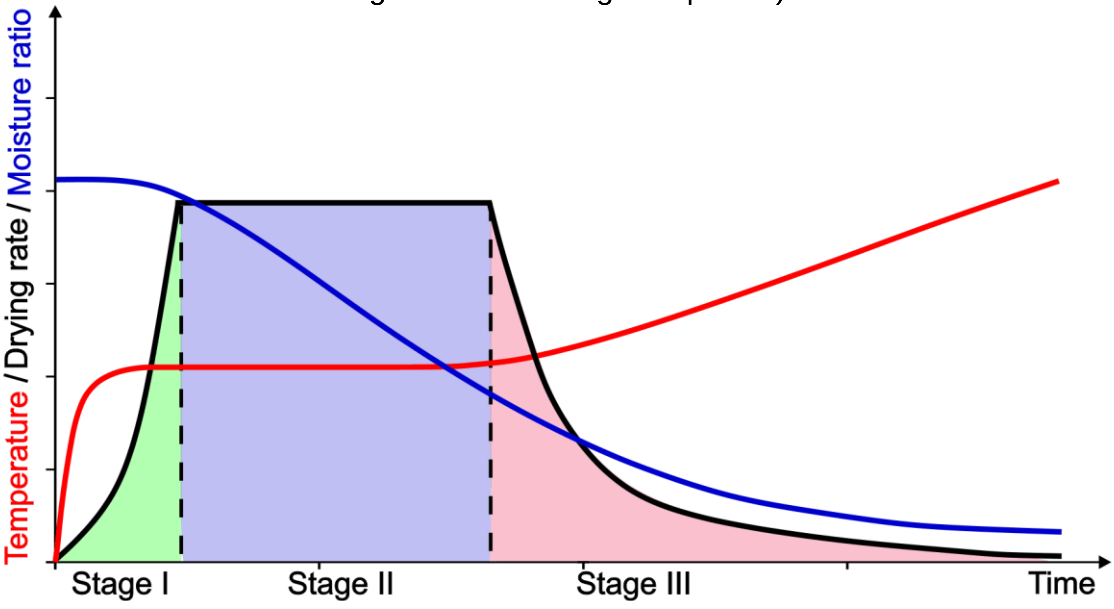
Therefore, pineapple pomace is promising for use in industry. However, the high moisture content makes it necessary to apply suitable processes to stabilize and store this pomace, and drying is an interesting alternative.

2.2 Drying

The drying process is an old preservation method and widely used in food, in most cases it consists of the application of heat and consequent removal of moisture. In addition to increasing stability and extending product life, drying can be applied to obtain foods with different characteristics (powder, flakes, granules), reduce the volume to optimize transport and storage and for the development of new products (MUJUMDAR, 1997). This process can be influenced by several factors, for example, the temperature and air velocity (CLEMENTE et al., 2014; POHNDORF et al., 2019), the type of drying (LI; ZHANG; BHANDARI, 2019; MINAEI et al., 2011) and the structure of the food (BARAT; GRAU, 2015; VALLESPIR et al., 2019).

Generally, the drying process has three distinct periods, determined by the heat and mass transfer coefficients. Figure 1 shows a typical curve for this process.

Figure 1 - Typical drying curve (Stage I: initial period; Stage II: constant rate period; Stage III: decreasing rate period).



In the first stage of the drying curve, the free water present on the food surface evaporates and, consequently, the surface temperature increases. This period is short and has an increased drying rate. The second stage has a constant drying rate, being characterized by the transfer of mass from the inside of the food to the surface by capillary forces. As the product dries, the transport of water from the inside of the food to the surface decreases, initiating the third stage of the drying curve. The period of decreasing rate depends on the diffusion of moisture from the interior to the surface and then by the transfer of mass from the surface. The drying rate decreases until the moisture content reach equilibrium, ending the drying process (BARAT; GRAU, 2015; EKECHUKWU, 1999; MUJUMDAR, 1997).

Among the traditional methods of drying foods, convective drying stands out Convective drying is a complex operation in which two different transport mechanisms occur in opposite directions simultaneously, involving the transfer of heat from the drying air to the food material and the transport of water from the interior of the product to the surface where the water evaporates to drying air. The phenomena of heat and mass transfer are often influenced by gradients of temperature and water concentration, air velocity field, and material properties (SABAREZ, 2016).

Hot air is the most common drying medium and performs two main functions: the function of providing sensitive and latent heat to cause the evaporation of moisture, and acts as a carrier gas, moving the formed water vapor away from the drying surface, allowing thus evaporation. Mass transfer during drying can be controlled by the rate of diffusion of moisture (liquid or vapor) in the food matrix (internal transfer) or by the rate of evaporation of moisture from the product surface to the drying medium (external transfer) (BRENNAN, 2003; SABAREZ, 2016).

Even so, this drying process consumes a high amount of energy due to long processing times, in addition, to directly affecting the technological and nutritional properties of foods. Because of this, many pre-treatment methods, such as ethanol, are being applied to optimize the convective drying of food, with the main objectives of reducing the drying time and energy consumption, maintaining the quality of the final product.

2.3 Ethanol as a pre-treatment for drying

In recent years, many studies have focused on the application of emerging technologies such as pre-treatments to improve the drying of food (LLAVATA et al., 2020). The application of drying accelerators (such as ethanol) is promising, as it does not involve high temperatures, and promotes a higher drying rate without impairing the final quality of the products. In fact, different studies have demonstrated the effectiveness of using ethanol to improve the drying processes of apple (AMANOR-ATIEMOH et al., 2020; FUNEBO et al., 2002; ROJAS; AUGUSTO; CÁRCEL, 2020, 2021; ZUBERNIK et al., 2019), carrots (SANTOS et al., 2021), scallion (WANG et al., 2019) and scallion stalk (ZHOU et al., 2020, 2021), melon (CUNHA et al., 2020), pitaya foam (ARAÚJO et al., 2020; MACEDO et al., 2021), potato (ROJAS; AUGUSTO, 2018c; ROJAS; SILVEIRA; AUGUSTO, 2019), eggplant (ZHAO et al., 2016), pumpkin (ROJAS; AUGUSTO, 2018a, 2018b), banana (CORRÊA et al., 2012), pineapple (BRAGA et al., 2009; BRAGA; SILVA, 2009).

Different mechanisms are associated with the effects of ethanol in food. Firstly, Funebo et al. (2002) showed that pre-treatment with ethanol promotes the dissolution of cell wall compounds, increasing the permeability of the samples. Rojas and Augusto (2018b, 2018c) confirmed that ethanol pre-treatment causes changes in the composition and structure of the cell wall and removes air from intercellular spaces. Moreover, ethanol promotes osmotic dehydration (WANG et al., 2019) and structural changes in the food matrix (SANTOS et al., 2021), impacting the reduction of the subsequent drying time.

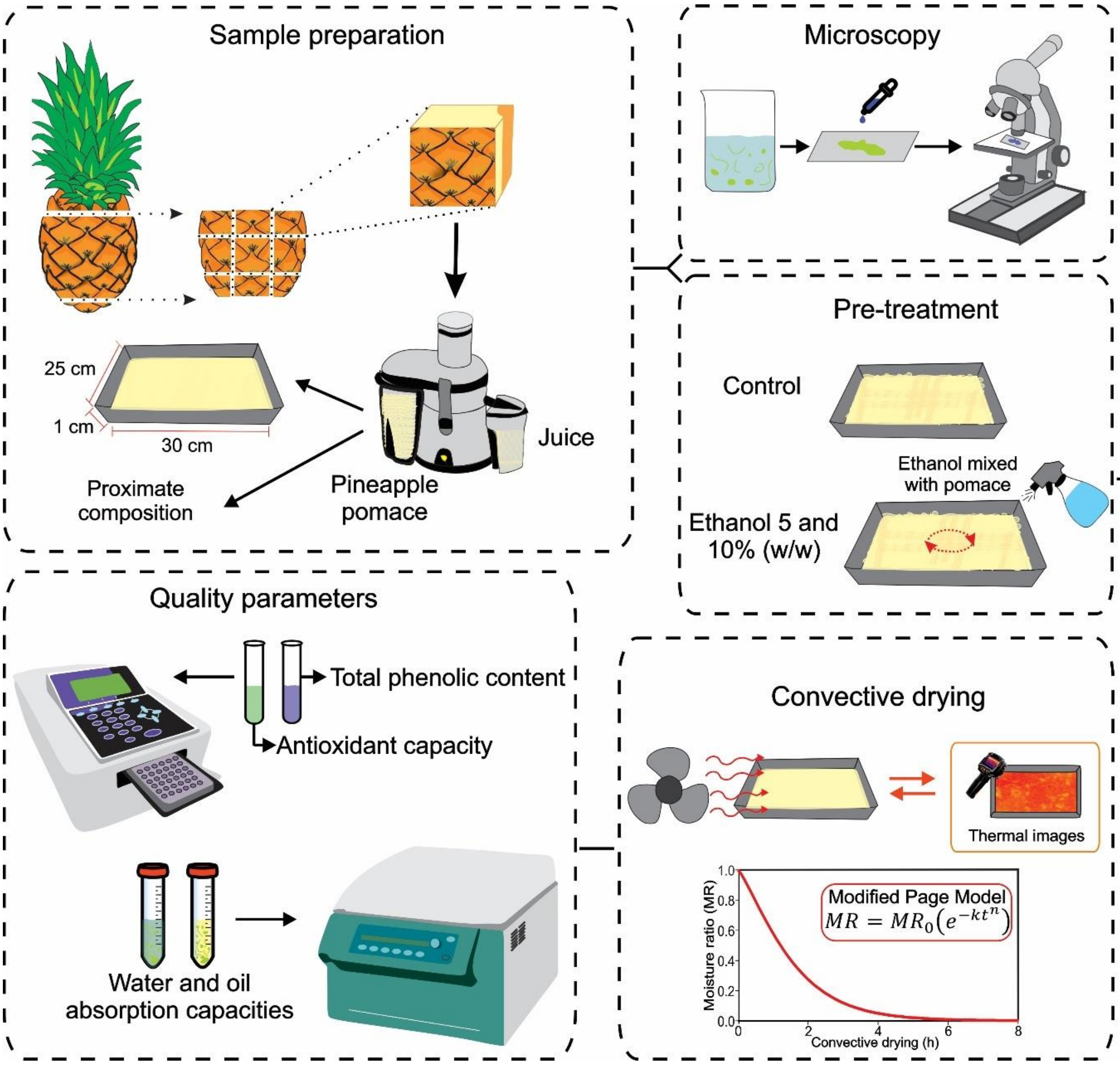
In addition, Silva, Braga and Santos (2012) demonstrated that the effects of ethanol pretreatment are due to the difference in surface tension between ethanol and water, promoting the Marangoni Effect. The Marangoni Effect, is also known as the Gibbs-Marangoni Effect, and refers to the transfer of mass along an interface between two fluids due to the difference in the surface tension of water and the organic compound, which causes a gradient of water concentration (BIRD, 2002; SILVA; BRAGA; SANTOS, 2012). During pre-treatment with ethanol, an ethanol-water mixture is formed within the food matrix. Then, during drying, the ethanol vaporizes faster, generating a gradient of surface tension within the sample. As a result, the region with the highest surface tension strongly pulls water from the inside of the food to the surface, promoting the flow of water from inside the sample until the surface tension balance is reached (ROJAS; AUGUSTO, 2018b).

However, the mechanisms of action of ethanol in drying food are based on the microstructure of the food matrix. In fact, few studies have addressed the effects of ethanol pre-treatment on unstructured products, making further studies on foods with different microstructures necessary.

3. MATERIALS AND METHODS

The experiments were conducted both at the Department of Food Science, Federal University of Lavras (UFLA) and Department of Agri-food Industry, Food and Nutrition, University of São Paulo (USP) - "Luiz de Queiroz" College (ESALQ). The experimental flowchart of sample preparation, pre-treatments, convective drying, and the analysis of the quality properties of pineapple pomace are shown in Figure 2.

Figure 2 - Illustrative flowchart of sample preparation, pre-treatment, convective drying and analysis of quality properties.



3.1 Pineapple pomace

Fruit by-products are often referred to as co-products, by-products, waste, residues, bagasse or pomace. In this work, we use the term “pomace” to refer to the pineapple residue after juice extraction, excluding the crown. Therefore, it is a fibrous high moisture by-product of pineapple juice extraction.

Samples of *in natura Pérola* pineapple (*Ananas comosus*) were obtained in the local market (Piracicaba, São Paulo, Brazil). Fresh fruits were selected to be homogeneous in ripening level, presenting a soluble solids content of 12 ± 1 °Brix (Refractometer Shibuya mod. A.C.R 121A 0-32%).

The fruits were washed with water, superficially dried and the crown was removed. The fruit, with peel, was then cut into pieces, which were introduced in a juice extractor with a 0.5 mm sieve (Juicer Centrifuge, Philips Walita). The juice was dispensed, and the pomace (peel and bagasse) was collected to be studied. This procedure was similar to the reported by Selani et al. (2014), studying the application of pineapple pomace in an extruded product for fiber enhancement.

The pineapple pomace was immediately dried, with and without pre-treatments. The dried samples were stored at -18 °C in hermetically sealed plastic bags for further analyses.

3.2 Proximate composition

The proximate composition was carried out according to the methods described by AOAC (2006): the moisture was determined in an oven at 105 °C, the nitrogen content was determined by the micro-Kjeldahl method using the factor 6.25 for the crude protein content, the lipids were determined by the Soxhlet method using hexane as a solvent, the total dietary fibers were determined by the acid-base method, the ash content was determined in muffle furnace at 550 °C until complete calcination and the carbohydrates were determined by difference. The analyses were performed at least in three repetitions.

The pineapple pomace had a moisture content of 84.93 ± 0.38 $g \cdot 100\ g^{-1}$ on a wet basis (w.b.), and the following composition (on dry matter): proteins 4.82 ± 0.01 $g \cdot 100\ g^{-1}$ d.m., lipids 0.70 ± 0.02 $g \cdot 100\ g^{-1}$ d.m., ash 3.21 ± 0.09 $g \cdot 100\ g^{-1}$ d.m., total dietary fibers 68.92 ± 1.24 $g \cdot 100\ g^{-1}$ d.m. and other carbohydrates 22.34 $g \cdot 100$

g^{-1} d.m. These results were similar to those obtained by Martínez et al. (2012) for pineapple dietary fiber concentrate and for pineapple co-products reported by Selani et al. (2016a).

3.3 Structural analysis

The fresh pineapple pomace was dispersed in water and deposited on a glass slide for structural analysis. Toluidine blue solution (0.1% in water) was used to enhance the images. Microscopies were performed using optical microscopy (model L1000, Bioval, Brazil) with a 10x magnification objective and a microscopy stereoscopic (model XTD-30-LED, NOVA, Brazil) with 6.3, 20 and 30x magnifications. The images were captured with a portable camera of 1.3 megapixels, after ensuring a representative field.

3.4 Pre-treatments and convective drying

Pre-treatments with the addition of ethanol to the pineapple pomace (5% or 10%, w/w) were carried out before convective drying. The control treatment did not contain ethanol addition. The ethanol (99.5% v/v) was sprayed in the pineapple pomace surface and then mixed to guarantee a homogeneous distribution.

The convective drying process was carried out in an oven with air circulation and renewal (MA 035, Marconi, São Paulo, Brazil) using two different temperatures (40 and 60 °C) and air velocity of $1 \pm 0.1\ m \cdot s^{-1}$ (measured parallel to the sample surface) (Testo 405i, Testo).

The pineapple pomace was placed in metal trays, achieving a thickness of 5 ± 0.5 mm. The trays were then placed in the oven for drying. The sample mass was recorded every 30 min in the first hour of drying and every 1 h until constant weight, using a precision scale (Mark, TECNAL, Piracicaba, Brazil).

The initial moisture content (fresh samples and after ethanol treatments) and final moisture content (after drying) were measured at 105 °C using a moisture analyzer (MX-50, A&D Company, Tokyo, Japan). The moisture was calculated by mass balance, and the moisture of the samples with pre-treatment include water and ethanol. During drying, the sample surface temperature was recorded by a thermal camera (Testo 865 - Testo).

The drying curves were obtained as a function of moisture ratio (MR) over the drying time, according to Equation (1), where M indicates the moisture content (% d.m.) (that is, g water · 100 g^{-1} d.m.) at a given time (t) during drying, M_e is the equilibrium moisture and M_0 is the sample moisture (% d.m.) before drying (the same approach proposed by Ricce et al. (2016) and applied by La Fuente, Zabalaga and Tadini (2017)).

Therefore, for the control samples, M_0 was the moisture of the fresh sample, while for the samples treated with ethanol, M_0 was the moisture of the samples after the pre-treatments, i.e., the moisture of the fresh sample + the added ethanol. Consequently, MR started from 1.0 for the control treatment, being higher than 1.0 for the treatments with ethanol addition.

$$MR(t) = \frac{M - M_e}{M_0 - M_e} \qquad (1)$$

The drying kinetics of pineapple pomace was evaluated using the Page Model (Eq. (2)) (PAGE, 1949), modified to present an initial moisture value different from one for the treatments with ethanol pre-treatment: therefore, MR_0 is the initial dimensionless moisture, *k* is the drying rate parameter and *n* is the dimensionless drying parameter .

$$MR(t) = MR_0\left(e^{-kt^n}\right) \qquad (2)$$

The model parameters were adjusted by interaction with the experimental data, minimizing the sum of the quadratic errors (Eq. (3)) between the experimental and predicted values. For this, we used a generalized reduced gradient algorithm, implemented in the 'Solver' tool of the Excel 365 software (Microsoft, USA). The initial assumptions of the model parameters were selected from previous work carried out by our study group (CARVALHO et al., 2020a).

$$SSE = \sum_{i=1}^{x} \left((predicted) - (experimental)\right)_i^2 \qquad (3)$$

3.5 Nutritional and bioactive aspects

3.5.1 Obtaining sample extracts

Methanolic extracts from fresh and dried samples with the different pre-treatments were obtained for the analysis of total phenolic content (TPC) and antioxidant capacity (AC). The extracts were prepared by mixing 10 mL of methanol:water solution (80:20 v/v) and the sample (~1 g of fresh samples or ~0.2 g of ground (analytical mill, 160 W, IKA A11, Brazil) dried samples). The mixture was homogenized using a rotor-stator homogenizer (Superohm, Brazil) for 30 s. The samples were shaken in an orbital shaker (20 °C, DUBNOFF MA 095/CFRE, Marconi, Brazil) for 30 min (after evaluating the conditions 15, 30, 45 and 60 min). Then, centrifugation was carried out for 20 min at 4500 *g* at 20 °C. The supernatant was collected in Falcon tubes protected from light and stored under refrigeration until analysis.

3.5.2 Total phenolic content (TPC)

The total phenolic content (TPC) was determined using the Folin-Ciocalteu reagent reduction method according to the methodology proposed by Singleton, Orthofer and Lamuela-Raventós (1999), with modifications. The reactions were performed by mixing 200 µL of extract, 1500 µL of distilled water, 100 µL of reagent Folin-Ciocalteu (Sigma-Aldrich, USA). After 5 min of rest in the dark, 200 µL of sodium carbonate (Synth, Brazil) 20% w/v were added to the reaction and maintained for 30 min in the dark at room temperature. After the reaction time, the absorbances were read in a microplate reader (Biochrom Asys Expert Plus Microplate Reader, United Kingdom) at 765 nm. The calibration curve was constructed with different concentrations of gallic acid (GA) (Vetec, Brazil) (3,4,5-trihydroxybenzoic acid) (0, 0.02, 0.04, 0.06, 0.08 and 0.10 $mg \cdot mL^{-1}$). A linear regression ($R^2 = 0.995$) was used to calculate the TPC content of the samples and the results were expressed in GA equivalents (mg GAE $\cdot$ g^{-1} d.m.).

3.5.3 Antioxidant capacity (AC)

The antioxidant capacity (AC) was performed using the ABTS method, described by Al-Duais et al. (2009) with modifications.

Firstly, the ABTS•$^{+}$ radical was generated from the oxidation of ABTS (2,2′-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) diammonium salt (Roche, Germany) (7 mM), and potassium persulfate (Dinâmica LTDA, Brazil) (140 mM), the final concentration of 2.45 mM. The mixture was remained in the dark for 16 h to complete the formation of the ABTS•$^{+}$ radical.

Then, the reaction of sample with the ABTS•$^{+}$ radical was conducted in microplates. The ABTS•$^{+}$ radical was diluted with methanol:water (80:20 v/v) until an absorbance of 0.71 ± 0.01 at 734 nm was reached using a reader microplate (Biochrom Asys Expert Plus Microplate Reader, United Kingdom). 220 µL of the ABTS•$^{+}$ radical solution and 20 µL of the extract were poured in the microplate for reaction. The absorbance was read in the microplate reader at 734 nm every 1 min until stabilization. The reaction stabilized after 10 min, which was the time chosen to determine the antioxidant capacity.

A calibration curve was constructed with different concentrations of Trolox (0, 12.5, 50, 100, 150, 200 and 250 µM). A linear regression (R^2 = 0.997) was used to calculate the antioxidant capacity of the samples and the results were expressed in Trolox equivalents (µM Trolox · g^{-1} d.m.).

3.6 Technological aspects: water absorption capacity (WAC) and oil absorption capacity (OAC)

The water (WAC) and oil (OAC) absorption capacities were carried out according to the methodology described by Beuchat (1977), with modifications. 1 g of sample was mixed with 10 mL of distilled water (for WAC) or canola oil (for OAC), left to stand for 30 min, and then centrifuged at 4500 *g* for 20 min (all at room temperature). Then, the supernatant was discarded, and the sample was weighed. The WAC was calculated according to Equation (4).

$$WAC = \frac{M_{s+w} - M_s}{DM} \tag{4}$$

Where M_{s+w} is the mass of the sample + water absorbed after centrifugation, M_s is the mass of the initial sample and DM is the dry matter of the sample; the water absorption capacity was presented as the water mass absorbed by 1 g of dry matter in the sample.

The OAC was calculated according to Equation (5).

$$OAC = \frac{M_{s+o} - M_s}{DM} \tag{5}$$

Where M_{s+o} is the mass of the sample + oil absorbed after centrifugation, M_s is the mass of the initial sample and DM is the dry matter of the sample; the oil absorption capacity was presented as the oil mass absorbed by 1 g of dry matter in the sample.

3.7 Experimental design and statistical evaluation

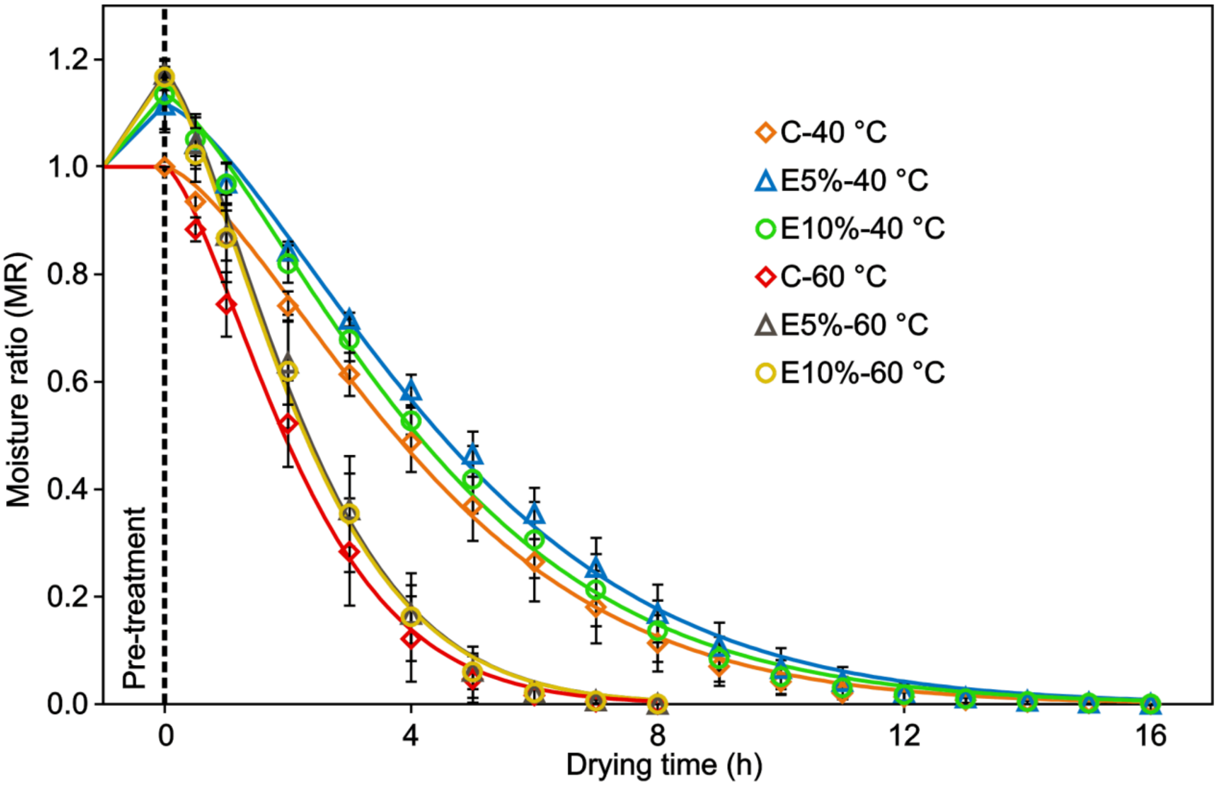
Three different lots were used, comprising three fruits each. For each lot, the pomace was obtained, dried and evaluated in three replicates. Analysis of variance (ANOVA) and Tukey test at 5% significance were performed to determine statistical differences between treatments. The statistical analyzes were determined using the Minitab® software (version 19.2, USA).

4. RESULTS AND DISCUSSION

4.1 Effect of temperature and ethanol pre-treatment on pineapple pomace convective drying

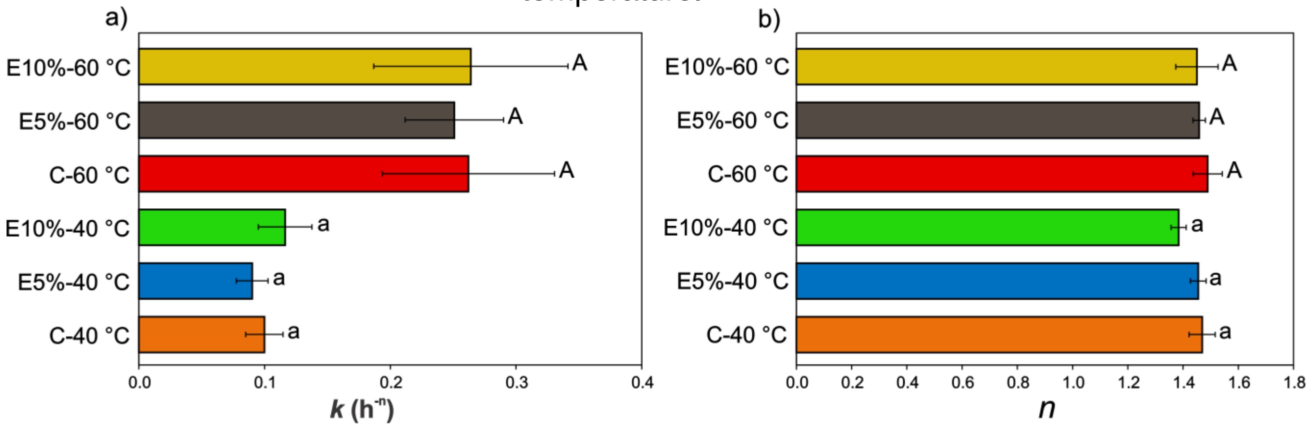
The fresh pineapple pomace had a high-water content (moisture > 84% w.b.), requiring a long drying time. At the end of the drying kinetics, the moisture content of the samples was 8.6 g · 100 g^{-1} ± 1.5 w.b. for drying at 40 °C and 5.8 g · 100 g^{-1} ± 1.0 w.b. for drying at 60 °C. The drying kinetics of pineapple pomace after different pre-treatments: Control (C), Ethanol 5% (E5%, w/w) and Ethanol 10% (E10%, w/w) are shown in Figure 3, with the data adjusted to the Modified Page Model. As can be seen, the drying process started with dimensionless moisture higher than one ($MR_0 > 1$) for the treatments using ethanol, due to the increase in sample moisture.

Figure 3 - Convective drying kinetics (40 and 60 ºC, 1 ± 0.1 m·s^{-1}) of pineapple pomace without (C) and with ethanol pre-treatment (E5% and E10%, w/w). Points are experimental data; continuous lines are the Modified Page Model (Equation 2). Vertical bars are the standard deviation.



In Modified Page Model, the parameter *k* can be associated to the diffusion coefficient and geometry of the sample (Figure 4a), while the *n* parameter indicates the type of diffusion and food microstructure ($n = 1$ pure diffusion, $n > 1$ super diffusion and $n < 1$ sub diffusion) (Figure 4b) (SIMPSON et al., 2017).

Figure 4 - Parameters: a) *k* and b) *n*, of the Modified Page Model (Equation 2) of pineapple pomace samples without (C) and with ethanol pre-treatment (E5% and E10%, w/w). Horizontal bars indicate the standard deviation. Equal letters indicate that there were no significant differences ($p > 0.05$) among treatments in each temperature.



As expected, the temperature had a great influence on the pineapple pomace drying rate, where drying at 40 °C (16 h) took twice as long as drying at 60 °C (8 h) (Figure 3). This was confirmed by the parameter *k*, which was significantly higher for drying at 60 °C (Figure 4a). In fact, the increase in the drying temperature promotes greater mobility of the water molecule within the food and the increase in the water pressure gradient between the phases, causing a decrease in the internal and external resistances to moisture transport (CORRÊA et al., 2017). In addition, contrary to structured vegetables, the pre-treatments with ethanol did not affect the pineapple pomace drying rate in any of the evaluated temperatures (Figure 3a).

The pre-treatment with ethanol has shown great efficiency in accelerating convective drying processes of different vegetables (LLAVATA et al., 2020) such as pineapple (BRAGA et al., 2009; BRAGA; SILVA, 2009), banana (CORRÊA et al., 2012), pumpkin (ROJAS; AUGUSTO, 2018), apple (ROJAS; AUGUSTO; CÁRCEL, 2020; ZUBERNIK et al., 2019), melon (CUNHA et al., 2020) and carrot (SANTOS et al., 2021). Moreover, the effect of ethanol can be dependent on the food structure.

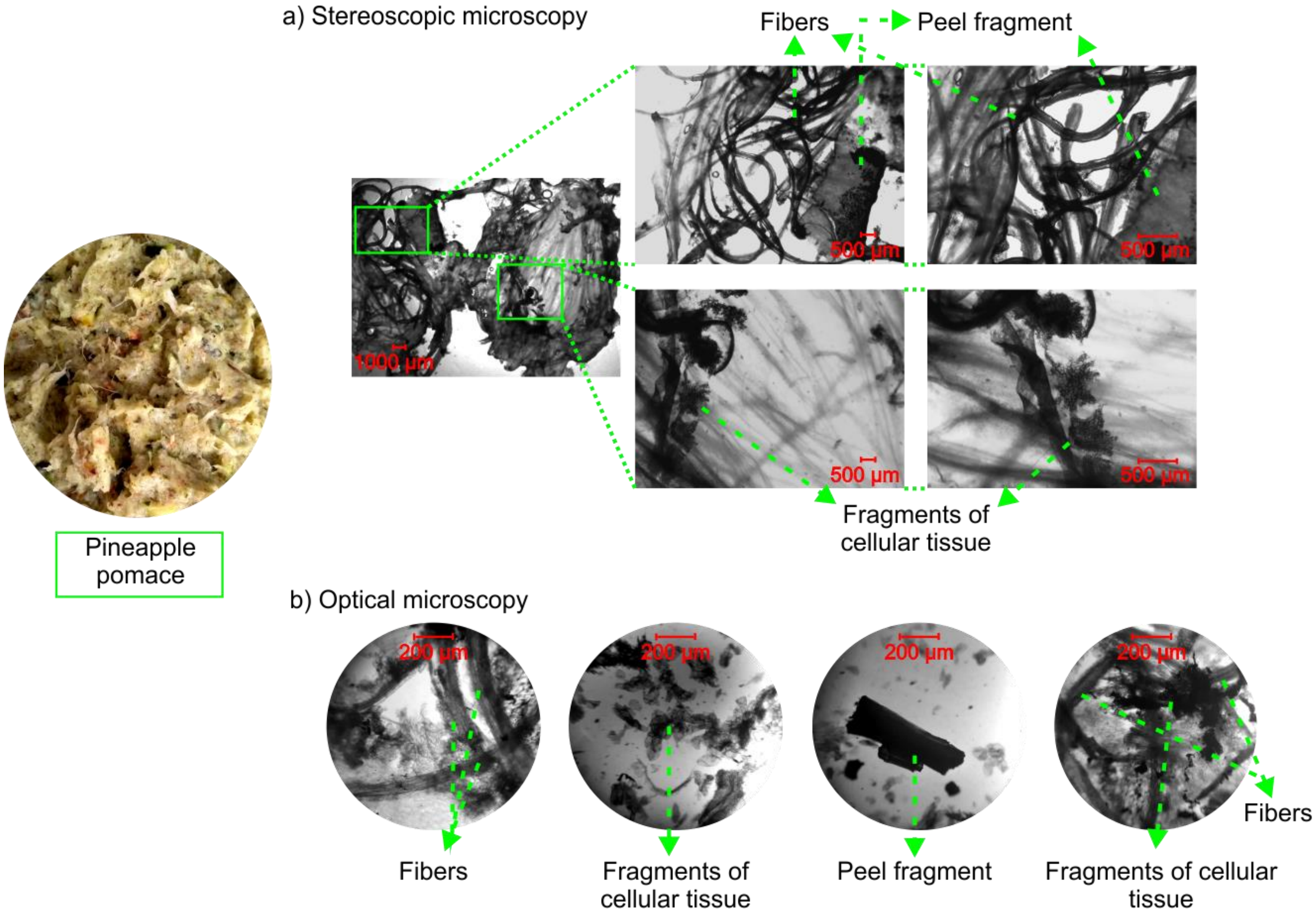
For example, Silva, Braga and Santos (2012) and Carvalho et al. (2020b) demonstrated the effect of ethanol on vegetables drying can be associated with the Marangoni Effect (SILVA; BRAGA; SANTOS, 2012). This mechanism was confirmed by Rojas and Augusto (2018) who showed the ethanol travels through xylem vessels in pumpkim pulpe, which can improve the Marangoni Effect during the drying process.

In addition, ethanol promotes osmotic dehydration (WANG et al., 2019b) and also structural changes in cell wall and membrane (SANTOS et al., 2021), which can influence the subsequent drying process.

In fact, the effect of ethanol on drying food is more pronounced at the beginning of the process, where there is a greater amount of water. As the amount of water decreases and ethanol is vaporized, the effects are reduced (ROJAS; SILVEIRA; AUGUSTO, 2019). Therefore, the behavior observed in this work can be mainly explained by the structural differences between structured pineapple and its pomace (fibrous material, with few soluble solids and high free water content).

Figure 5 shows the structure of pineapple pomace using both stereoscopic microscopy (Figure 5a) and optical microscopy (Figure 5b).

Figure 5 - Microscopic evaluation of pineapple pomace: a) images obtained by stereoscopic microscopy; b) images obtained by optical microscopy.



The pineapple fruit microstructure is composed mainly by xylem, phloem, parenchyma, and sclerenchyma of fibrous constitution (LUENGWILAI; BECKLES; SIRIPHANICH, 2016). However, during pineapple processing, the tissues and cells were disrupted, and the further centrifugation and filtration result in a particular microstructure: the pineapple pomace is composed of fibers, fragments of peel, and cell walls (Figure 5), being a material with a high content of free water and small organization. In addition, the content of soluble solids was reduced along with the removal of the juice, contributing to the availability of free water in the food. Consequently, the internal resistance to mass transport is smaller than the structured fruit, favoring the outflow of water during drying.

In fact, this can be verified in the drying curves, that present a long period of constant rate, which is associated with the external resistance to the mass flow: in the period of constant rate the free moisture vaporization of the food occurs and the rate of mass transfer within the food is equal to the rate at which water evaporates from the surface. The drying during this period is totally governed by the external heat and mass transfer rates, since there is always the presence of free water on the food surface (BARAT; GRAU, 2015; MUJUMDAR; OSMAN, 2006). In the pineapple pomace, this period extended to a certain extent, until the moisture had been reduced, decreasing the rate of water vaporization and starting the period of falling rate. Moreover, all treatments showed a super diffusive behavior ($n > 1$; Figure 4b), including the control. The parameter n is related to the material's microstructure, and when $n \neq 1$, other mechanisms besides diffusion are important (SIMPSON et al., 2017). For example, Rojas and Augusto (2018) associated the phenomenon of super diffusion with capillarity.

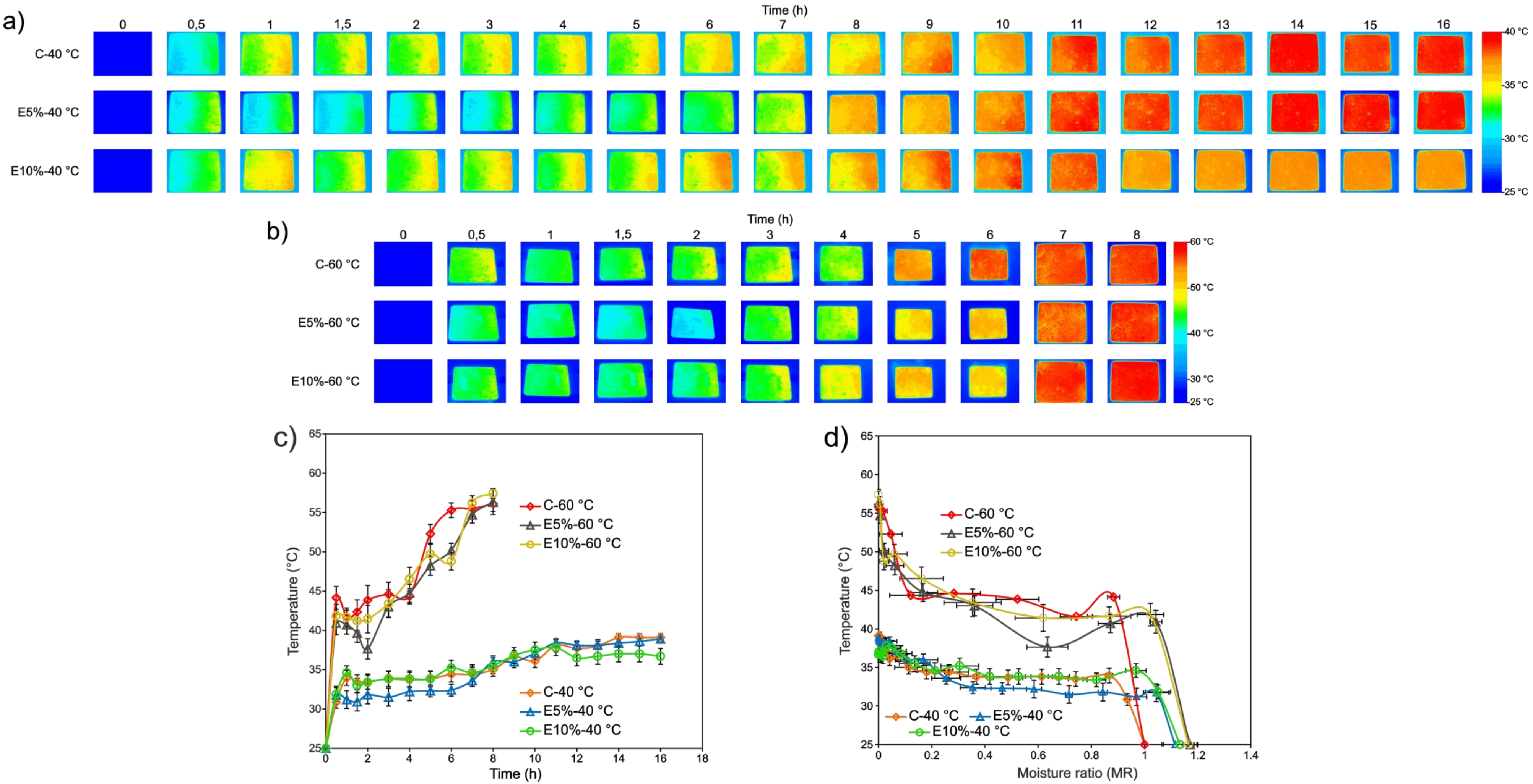
Therefore, in unstructured products, drying kinetics is more influenced by external resistance to mass transfer. The external resistance is influenced by the boundary layer that develops around the particle. Therefore, the factors that affect the boundary layer will also affect this resistance, for example, air velocity, particle shape and size (MULET, 1994). In addition, Rojas, Augusto and Cárcel (2020) observed that ethanol also influences external resistance to mass transfer, as the air-product interaction is different for products pre-treated with ethanol. However, in an unstructured sample, ethanol tends to vaporize much faster than in structured samples, minimizing the action of ethanol in these products. Moreover, the water flow through the pomace already experience less resistance than in structured vegetables. Consequently, no advantage of using ethanol is observed.

There are only two studies in the literature addressing the effects of drying accelerators on fruit residues, an unstructured product. Silva, Duarte and Barrozo (2016) and Silva et al. (2020) applied ethanol to acerola residues (a tropical small stone fruit) by spraying onto the residue surface as pre-treatment before drying in a Designed Rotary Dryer and Rotary Dryer Assisted by Infrared Radiation. The authors noted that ethanol pre-treatment increased the product drying. However, those works differ from the study described here in some aspects. For example, the particle size of the acerola residue (11.5x19.70 mm) was much larger than the pineapple pomace, which was crushed in a juice extractor with a 0.50 mm sieve. Consequently, not only the composition of both products are different, but also their microstructures. In fact, although the authors did not present microscopies, it is interesting to notice the acelora residue particle size was within the dimensions of small pieces of tissues, still containing intact cells. In a by-product with larger particles, ethanol pre-treatment can behave similarly to those used for structured products, as described before - thus increasing the drying rate. In addition, the drying processes are different: in the works of Silva, Duarte and Barrozo (2016) and Silva et al. (2020), a rotary dryer was applied, promoting a constant and great aggitation during convective or infrared assisted convective drying. In the present work, a regular convective drying was applied, where the sample was kept static.

In addition, Macedo et al. (2021) applied ethanol as a pre-treatment for drying the pitaya foam pulp, also an unstructured food, and observed a reduction in the drying time. In this work, the foaming process of pitaya pulp results in a food with broken cell structure, however, there is no loss of juice and/or soluble solids during the process. On the other hand, during the pineapple processing, the juice is separated from the pomace, thus reducing the content of soluble solids in the final pomace. The reduction of soluble solids in pineapple pomace contributes to the increase of free water content, favoring the flow of water and limiting the effect of ethanol in this product.

The surface temperature distribution during pineapple pomace drying is shown in Figure 6: Figures 6a and 6b show the thermal images at different drying temperatures, with the scale varying from blue (less hot) to red (hotter); while Figures 6c and 6d show the average surface temperature profiles as a function of time and moisture ratio (MR), respectively.

Figure 6 - Temperature history during the convective drying of pineapple pomace without (C) and with ethanol pre-treatment (E5% and E10%, w/w): a) thermal images of drying at 40 °C; b) thermal images of drying at 60 °C; c) history of temperatures as a function of time; d) history of temperatures as a function of moisture ratio. Vertical and horizontal bars indicate the standard deviation.



Ethanol has a higher vapor pressure than pure water, as well as ethanol-water mixtures. Silva, Braga and Santos (2012) demonstrated the effect of ethanol in improving drying cannot be related to vapor pressure. In fact, Carvalho et al. (2020b) showed the ethanol vapor pressure has a greater influence on the sample temperature than on its drying rate. This is confirmed inthis work by the thermal images (Figure 6a and 6b): Samples with ethanol pre-treatment showed lower temperatures than the control at some points during drying (Figure 6c and 6d). Even so, the drying rate is not altered (Figure 3). It demonstrated just adding a compound with greater volatility and higher vapor pressure is not enough to improve the drying process, which reinforces the main mechanisms of ethanol as drying acellerator are related with the Marangoni Effect and structural changes.

In the last years, several studies have shown that ethanol promotes many advantages for food drying, promoting faster drying, ensuring less energy consumption and avoiding nutrient degradation (CUNHA et al., 2020; ROJAS; AUGUSTO, 2018; SANTOS et al., 2021; WANG et al., 2019b). However, the effect of ethanol depends on several factors, such as the internal resistance to mass transfer – which is associated with structure.

Therefore, this work described a possible limitation of using ethanol as drying accelerator, once its efficiency can be restricted to structured products. Even so, further studies are needed to improve the unstructured products drying. Moreover, the quality of the obtained dried pineapple pomace must evaluate, as described as follows.

4.2 Quality properties

Fruit pomaces are considered good sources of fiber, phenolics and antioxidants compounds, and their application in the development of new products is interesting to increase the nutritional and bioactive values. In addition to improving the nutritional value, the presence of fibers can improve the technological (O’SHEA; ARENDT; GALLAGHER, 2012) and sensory (PUVANENTHIRAN et al., 2014) attributes of food products, such as in bakery products. Moreover, the fruit pomaces have been also used for animal feed as a source of nutrients and bioactive compounds. For example, Vasconcelos et al. (2020) observed a number of benefits in the application of pineapple by-products in pig diet, such as an increase in the percentage of lean meat in the animals.

Although drying can promote preservation, conventional methods that use hot air for long periods can result in undesirable changes in the final product quality. Therefore, it is important to evaluate the quality properties before and after drying, as described as follows.

4.2.1 Nutritional and bioactive aspects: total phenolic content (TPC) and antioxidant capacity (AC)

The phenolic compounds are important due to their antioxidant capacity and consequent inactivation of free radicals, chelation of metal ions, among others (OLIVEIRA et al., 2009). In fact, antioxidant compounds are essential for producing food with greater stability and quality, in addition to being directly linked to consumer health (BREWER, 2011).

Agro-industrial residues can be rich in bioactive compounds (LEYVA-LÓPEZ et al., 2020), and so their application can be interesting in many sectors. The main application for those residues is in food products, with different objectives. For instance, improving the nutritional quality, such as cakes (JEDDOU et al., 2017) and cookies, or promoting other technological properties, such as retaining water, oil or substituting fats, such as hamburgers (SELANI et al., 2016a, 2016b). Moreover, the agro-industrial residues have potential application as animal feed, bringing benefits in terms of meat quality and safety (HERNÁNDEZ-LÓPEZ et al., 2016).

Consequently, it is interesting to evaluate nutritional aspects during processing.

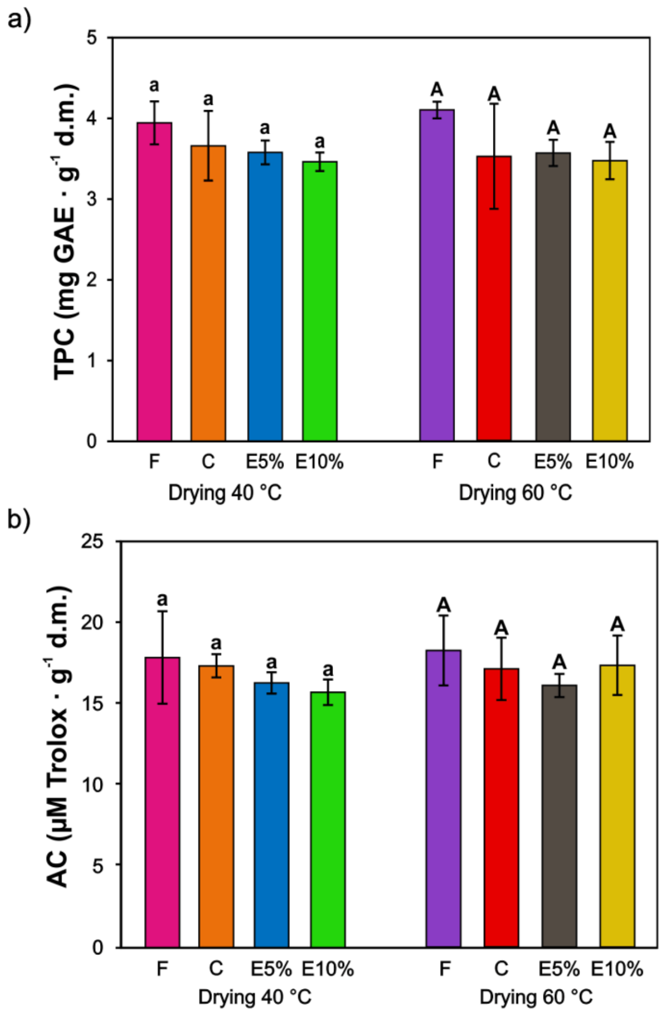
The fresh pineapple pomace showed TPC values of 4.02 ± 0.19 mg GAE · g^{-1} d.m. and AC of 18.05 ± 0.27 µM Trolox · g^{-1} d.m. These values are similar to the levels previously reported by the central pineapple axis (~3 mg GAE · g^{-1} d.m. for TEAC and ~7, 6 µM Trolox · g^{-1} d.m. for AC (using ethanolic extract) - Toledo et al. (2019)), pineapple byproducts (~4 mg GAE · g^{-1} d.m. for TEAC and ~14 µM Trolox · g^{-1} d.m. for AC (using ethanolic extract) - Selani et al. (2016a)) and pineapple residue fiber concentrate (1.29 mg GAE · g^{-1} dm for TEAC and 7.7 µM Trolox · g^{-1} dm for AC (using methanol: acetone extract) - Martínez et al. (2012)).

In the present study, drying and pre-treatments with ethanol did not affect the levels of phenolic compounds and antioxidants in pineapple pomace. As can be seen in Figure 7, there was no degradation of TPC and AC during drying for all treatments

($p > 0.05$) compared to fresh samples. That is, the application of the drying process is efficient to reduce the moisture content of pineapple pomace, improving its stability and logistics, without affecting its nutritional quality. It is worth to mention this is an interesting result from an application perspective.

This result is different from those reported by Izli, Izli and Taskin (2018) with convective drying of pineapple. The authors observed a decrease in both TPC and AC the contents, but when dried at higher temperatures: 60, 70, 80 and 90 °C. The combination of high temperatures and the presence of oxygen can lead to degradation of phenolic compounds and antioxidants, decreasing the product nutritional value (FANG; BHANDARI, 2011). Therefore, the drying process must preserve these compounds as much as possible, maximizing the quality of the product (LLAVATA et al., 2020).

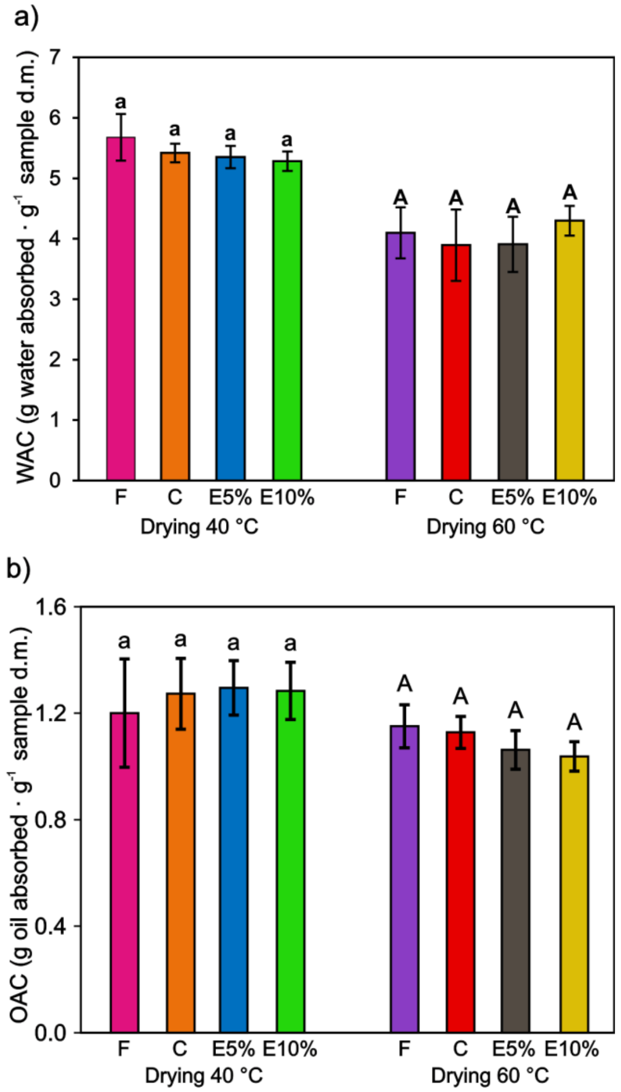
Figure 7 - a) Total phenolic content (TPC) and b) antioxidant capacity (AC) measured in fresh samples (F) and after drying without (C) and with pre-treatment with ethanol (E5% and E10%, w/w) of pineapple pomace. Vertical bars indicate the standard deviation. Equal letters indicate that there were no significant differences (p>0.05) among treatments in each temperature.



4.2.2 Technological aspects: water (WAC) and oil (OAC) absorption capacities

The water (WAC) and oil (OAC) absorption capacities were evaluated in the fresh and dry pineapple pomace, being presented on Figure 8. The pineapple pomace showed WAC values of 5.68 ± 0.38 g of water · g^{-1} of sample d.m. and OAC of 1.20 ± 0.20 g of oil · g^{-1} of sample d.m. corresponding to the fresh pomace used in drying at 40 °C. And it presented WAC of 4.10 ± 0.42 g of water · g^{-1} of sample d.m. and OAC of 1.15 ± 0.08 g of oil · g-1 of sample d.m. corresponding to the fresh pomace used in drying at 60 °C. The WAC and OAC values were statistically equal for all the treatments, demonstrating drying did not compromises these properties.

Figure 8 - a) water absorption capacity (WAC) and b) oil absorption capacity (OAC) in fresh pineapple pomace (F) and after convective drying without (C) and with pre-treatment with ethanol (E5% and E10%, w/w). Vertical bars indicate the standard deviation. Equal letters indicate that there were no significant differences (p>0.05) between treatments in each temperature.



The obtained values are similar to previous studies using similar products. For instance, Selani et al. (2014, 2016c) reported values of approximately 5.3 g water · g^{-1} sample d.m. and 2.0 g oil · g^{-1} sample d.m. for freeze-dried pineapple pomace. In addition, this result was similar to the pineapple pomace fiber, which presented 4.44 g of water · g^{-1} of sample d.m. and 1.65 g oil · g^{-1} sample d.m. (CHAREONTHAIKIJ; UAN-ON; PRINYAWIWATKUL, 2016).

Pineapple pomace showed similar WAC to pomegranate pomace (VIUDA-MARTOS et al., 2012) and higher than flours prepared from pequi fruit by-products (LEÃO et al., 2017), tamarind fiber (TRIL et al., 2014) and chickpea protein isolate (SOHAIMY et al., 2021). On the other hand, the pineapple pomace OAC was lower than pomegranate pomace powder co-product (VIUDA-MARTOS et al., 2012) and chickpea protein isolate (SOHAIMY et al., 2021), but similar to the flours prepared from pequi fruit by-products (LEÃO et al., 2017) and tamarind fiber (TRIL et al., 2014).

High indices of water and oil absorption capacities are desirable characteristics for fiber-rich co-products being applied as an ingredient in different products.

A pomace with a high water absorption capacity can be used to modify the viscosity and texture of formulated foods (GRIGELMO-MIGUEL; MARTÍN-BELLOSO, 1998; GUILLON; CHAMP, 2000). For example, in bakery products, water plays an important role throughout the production process (CAPPELLI et al., 2018), in addition, co-products with high WAC are an option for animal feed, promoting satiety and well-being (SILVA et al., 2012).

The oil absorption capacity (OAC) of the fibers is important for greater stability and flavor retention of foods with a high percentage of fat and emulsions (GRIGELMO-MIGUEL; MARTÍN-BELLOSO, 1998; GUILLON; CHAMP, 2000), as well as substituting fat in products such as beef burger (SELANI et al., 2016b, 2016c), buffalo meat sausages and patties (YOUNIS; YOUNIS; AHMAD, 2017).

In summary, pineapple pomace showed a high water absorption capacity and the oil absorption capacity was similar to other pomaces. Therefore, combining the technological and nutritional properties, the large generation of pineapple pomace worldwide, we can describe its application as interesting compared to other products. For example, this pomace can be used in products where water retention is important, reducing the oily feeling in fried products, and even for animal feed.

In addition, drying did not affect the quality properties of the pineapple pomace. This result is important from the point of view of the processing and application of this product, since drying promoted a significant decrease in the amount of water in the pineapple pomace, increasing stability without impairing quality.

5. CONCLUSIONS

This work studied the effects of pre-treatments with ethanol and convective drying on pineapple pomace. The internal resistance to mass transfer were smaller than the external resistance, reinforcing the particularity of pineapple pomace structure: it is composed by fibers, fragments of peel, and cell walls, being a material with a high content of free water and small organization. Consequently, pre-treatments with ethanol did not affect the drying kinetics of pineapple pomace, highlighting a limitation of this strategy to enhance drying. This result is interesting as it helps to understand the mechanisms associated with the ethanol pre-treatment, that are different in structured and unstructured products. On the other hand, ethanol promoted a lower temperature during drying, which was attributed to the higher vapor pressure of ethanol in comparison with water.

All the evaluated drying strategies maintained the nutritional and technological quality properties of pineapple pomace, namely the total phenolic content (TPC), antioxidant capacity (AC), water (WAC) and oil (OAC) absorption capacities.

Consequently, convective drying was shown as an adequate strategy to enhance pineapple pomace preservation without affecting its quality properties, being an interesting ingredient for applications in food formulations or as animal feed.

Future studies are needed to investigate methods for optimizing the drying process of unstructured products.

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