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CONDUCTIVE THIN-FILM DRYING METHODS FOR PRODUCING AÇAÍ (*Euterpe oleracea* Mart.) POWDER

Florianópolis
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Thesis submitted to the Graduate Program in Food Engineering of the Federal University of Santa Catarina in fulfillment of the requirements for the degree of doctor.
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Prof. João Borges Laurindo, Dr.
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Florianópolis, 2022.

This work is dedicated to my family and friends.

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“Nothing in life is to be feared, it is only to be understood. Now is the time to understand more, so that we may fear less.”

Marie Curie

RESUMO

Açaí (*Euterpe oleracea* Mart.) apresenta um alto valor nutricional, sendo uma importante fonte de antocianinas e ácidos graxos insaturados. No entanto, a fruta é altamente perecível. A secagem do suco de açaí é uma alternativa para aumentar sua vida útil, agregar valor, reduzir perdas pós-colheita, bem como, diversificar as opções de consumo e diminuir os custos de transporte e armazenamento. A liofilização e a secagem por atomização são os métodos mais empregados na literatura e na indústria de alimentos para produção de açaí em pó. Contudo, o elevado custo da liofilização e a baixa eficiência energética e a necessidade de uma prévia filtração do suco de açaí para a secagem por atomização tornam esses métodos de secagem desvantajosos. Assim, o objetivo deste trabalho foi aplicar tecnologias de secagem alternativas para produção de açaí em pó. O efeito dos processos de secagem condutivos de filmes finos, tais como, a secagem em tambor e *cast-tape drying*, na qualidade dos pós de açaí foi investigado. A qualidade do produto em pó foi baseada na retenção de antocianinas, preservação da atividade antioxidante, teor de lipídeos e oxidação lipídica. O trabalho foi dividido em 3 etapas. Na primeira etapa, avaliou-se o efeito da pressão da câmara de secagem (pressões a vácuo e atmosférica) nas propriedades físicas e químicas dos pós de açaí. Esse estudo foi realizado em escala laboratorial e piloto. Experimentos utilizando secagem com ar quente e liofilização foram realizados como casos de referência. Os resultados demonstraram que a secagem condutiva em baixas pressões contribuiu para a preservação da qualidade do açaí em pó, e que a seleção da pressão da câmara de secagem deve ser realizada de maneira cuidadosa a fim de obter um produto de alta qualidade. A segunda parte do trabalho consistiu em analisar o impacto da adição de hidrocolóides (pectina de alta metoxilação, goma arábica e maltodextrina 10DE), em uma concentração de 3% (3 g hidrocolóide 100 g^{-1} suco de açaí), nas propriedades físico-químicas dos pós obtidos por *cast-tape drying*. A qualidade desses produtos foi comparada com a do pó de açaí sem hidrocolóides obtido por liofilização. A adição de pectina ao suco de açaí reduziu a separação do óleo durante o processo de secagem e contribuiu para a remoção do produto seco na forma de um filme contínuo do suporte de secagem. Além disso, a adição desse hidrocolóide resultou em um pó de açaí com cor, teor de antocianinas e atividade antioxidante similares ao do pó de açaí liofilizado. Na última etapa do trabalho, a estabilidade dos pós de açaí produzidos por *cast-tape drying*, com e sem adição de pectina, foi avaliada durante o armazenamento em diferentes umidades relativas, por meio da construção de isotermas de sorção de umidade, teor de antocianinas e medidas dos parâmetros de cor. Os teores de umidade da monocamada, determinados a partir das isotermas de sorção, não foram suficientes para predizer a estabilidade das antocianinas nos pós de açaí. Além disso, a degradação de antocianinas não foi o fator principal responsável pela mudança de cor nos pós de açaí armazenados nas condições estudadas. Os resultados deste trabalho sugerem, assim, que a aplicação de pressões reduzidas ou adição de hidrocolóides ao suco de açaí nos processos de secagem condutivos são estratégias promissoras para produzir pós de açaí de alta qualidade em curtos tempos de secagem.

Palavras-chave: Secagem condutiva. Antocianinas. Oxidação lipídica.

RESUMO EXPANDIDO

Introdução

O açaí (*Euterpe oleracea* Mart.) é uma fruta nativa da região amazônica, sendo o seu plantio uma importante atividade econômica que gera empregos e garante a renda de populações ribeirinhas. Essa fruta tem atraído a atenção das indústrias e cientistas devido ao seu alto valor nutricional, sendo considerado uma superfruta. O açaí é rico em fibras, proteínas e ácidos graxos insaturados, bem como é uma importante fonte de antocianinas com alta atividade antioxidante. Porém, apresenta elevada perecibilidade, necessitando, assim, de processos que aumentem sua vida útil, agreguem valor, reduzam as perdas pós-colheita e diversifiquem as suas opções de consumo. A secagem do suco de açaí para obtenção de um produto em pó representa uma alternativa adequada de conservação da fruta, diversificação da oferta do produto e facilitação da exportação. Os métodos de secagem mais empregados para produção de açaí em pó são a liofilização e a secagem por atomização. Apesar da liofilização resultar em produtos desidratados de alta qualidade, essa tecnologia apresenta elevado custo devido ao longo tempo de processo e ao alto consumo de energia. Por sua vez, a secagem por atomização é um processo de secagem rápido, que previne o aquecimento excessivo do produto. No entanto, essa técnica apresenta baixa eficiência energética e não é adequada para secagem de líquidos viscosos, como por exemplo, o suco de açaí, requerendo, desta forma, uma prévia filtração para evitar o entupimento do bico atomizador devido aos sólidos em suspensão. Essa etapa, entretanto, resulta em perdas nutricionais significativas para o produto. Logo, métodos de secagem alternativos que permitam preservar a qualidade do produto e reduzir os custos de processo são de grande interesse. Dentre esses, pode-se destacar as técnicas de secagem condutivas, tais como, a secagem em tambor e o *cast-tape drying*, que possibilitam a aplicação de líquidos viscosos e são processos energeticamente eficientes. No entanto, os biocompostos presentes no suco de açaí, tais como, as antocianinas e ácidos graxos insaturados, são altamente sensíveis ao oxigênio, temperatura, e umidade, podendo ser facilmente degradados durante os processos de secagem e armazenamento. Assim, a redução da pressão de secagem ou adição de hidrocolóides ao suco de açaí podem ser boas alternativas para obter pós de açaí de alta qualidade.

Objetivos

O objetivo central da tese foi produzir pós de açaí por métodos de secagem condutivos em forma de filmes finos e avaliar a qualidade do produto em termos do teor de antocianinas, atividade antioxidante e produtos da oxidação lipídica. Especificamente, os objetivos da tese foram: (i) avaliar o efeito das pressões de secagem na qualidade de açaí em pó produzido por secagem condutiva em escala laboratorial e piloto; (ii) investigar a influência da adição de diferentes hidrocolóides na qualidade dos pós de açaí produzidos por *cast-tape drying*; (iii) comparar a qualidade dos pós de açaí obtidos pelas secagens condutivas com aquela produzida pela liofilização; e (iv) estudar a estabilidade das antocianinas nos pós de açaí obtidos por *cast-tape drying* armazenados em diferentes umidades relativas.

Metodologia

A tese foi dividida em três etapas. Na primeira etapa, os pós de açaí foram, primeiramente, produzidos em um secador condutivo em escala laboratorial, que simula a secagem em tambor (a vácuo). Avaliou-se o efeito das diferentes pressões da câmara de secagem (415, 700 e 1013 mbar) no processo de secagem e nas propriedades físico-químicas dos produtos secos. A qualidade do produto foi comparada com aquelas dos pós obtidos por liofilização e secagem com ar quente. A caracterização do produto foi baseada nas propriedades físicas (teor de umidade, atividade de água, teor de lipídeos e cor) e químicas (teor total de antocianinas

monoméricas, atividade antioxidante pelo ensaio DPPH e produtos da oxidação lipídica quantificado por ressonância magnética nuclear). Em seguida, os experimentos foram realizados em escala piloto, utilizando-se um secador de tambor a vácuo com variação da pressão de secagem e da velocidade do tambor. A qualidade dos pós de açaí também foi avaliada. A segunda parte do trabalho consistiu em analisar o impacto da adição de hidrocolóides (pectina de alta metoxilação, goma arábica e maltodextrina 10DE) nas propriedades físico-químicas dos pós de açaí obtidos por *cast-tape drying*. As propriedades viscoelásticas das suspensões, a liberação do óleo a partir das suspensões para o suporte de secagem durante o processo de desidratação, bem como a distribuição do óleo nas amostras após a secagem foram investigadas. A qualidade dos pós de açaí obtidos por *cast-tape drying* foi comparada com aquela do pó de açaí puro liofilizado (sem hidrocolóides). Por fim, na terceira etapa da tese, a estabilidade dos pós de açaí produzidos por *cast-tape drying* com e sem adição de pectina foi avaliada durante o armazenamento em diferentes umidades relativas, por meio da construção de isotermas de sorção de umidade, teor total de antocianinas monoméricas e medidas instrumentais de cor.

Resultados e Discussão

O suco de açaí original (sem diluição ou filtração) foi uniformemente espalhado nos suportes dos secadores condutivos como um filme fino. Os processos de secagem condutivos possibilitaram a produção de pós de açaí com baixos teores de umidade e atividades de água em curtos tempos de secagem (poucos minutos). A redução da pressão da câmara de secagem resultou em menores temperaturas de ebulição e curtos tempos de processo, mas também na formação mais vigorosa de bolhas de vapor. Um alto teor de antocianinas e atividade antioxidante foram obtidos por meio da secagem em baixa pressões devido às baixas temperaturas de ebulição e concentração de oxigênio e aos curtos tempos de processo. Esses resultados foram comparáveis aos do pó de açaí liofilizado e significativamente maiores do que aqueles secos com ar quente. No entanto, a secagem condutiva apresentou menor estabilidade à oxidação lipídica do que a liofilização e secagem com ar quente, sugerindo o efeito dominante da temperatura durante o processo de secagem do suco de açaí para aceleração da oxidação lipídica. Além disso, a liberação do óleo pode ser favorecida durante a secagem em tambor a vácuo dependendo das condições de pressão da câmara e tempo de retenção empregados. A adição de hidrocolóides ao suco de açaí resultou em maiores tempos de secagem por *cast-tape drying* do que o suco de açaí puro, devido às propriedades de ligação dos hidrocolóides com as moléculas de água. No entanto, os tempos de processo do *cast-tape drying* foram mais curtos do que o da liofilização. Embora a adição de pectina tenha resultado no maior tempo de secagem em comparação com as outras suspensões secas por *cast-tape drying*, esse hidrocolóide dificultou a liberação de óleo a partir da amostra para o suporte de secagem e facilitou a remoção do material seco devido à forte rede formada no interior da amostra. A adição de pectina também resultou em um pó de açaí com cor, teor de antocianinas e atividade antioxidante semelhantes ao do pó de açaí liofilizado (sem adição de hidrocolóides), indicando o efeito protetor desse hidrocolóide durante o processo de secagem do suco de açaí por *cast-tape drying*. No entanto, o processo de secagem do suco de açaí, com ou sem hidrocolóides, por *cast-tape drying* também apresentou uma menor estabilidade à oxidação lipídica do que a liofilização. Isto é, uma maior formação de produtos secundários da oxidação lipídica, os quais são responsáveis por alterações negativas da qualidade, como o desenvolvimento de *off-flavors* e/ou redução da qualidade nutricional, foi observada para os pós de açaí produzidos por *cast-tape drying*. Por fim, os pós de açaí com e sem adição de pectina (hidrocolóide que apresentou melhores resultados, como discutidos anteriormente), obtidos por *cast-tape drying*, foram armazenados em diferentes umidades relativas a 25 °C. Os modelos de GAB e BET foram

ajustados aos dados experimentais das isotermas de sorção de umidade. Apesar do modelo de GAB descrever bem matematicamente os dados experimentais, o modelo de BET apresentou mais significado físico. A adição de pectina resultou em um maior teor de umidade da monocamada. Supôs-se que os valores de atividade de água crítica correspondentes aos valores de umidade da monocamada eram as condições para uma boa estabilidade de armazenamento dos pós. Entretanto, uma degradação das antocianinas nos pós de açaí foi observada em atividade de água de armazenamento abaixo das atividades de água críticas. Em contrapartida, pós de açaí com pectina armazenados em maiores umidades relativas apresentaram uma coloração mais avermelhada e atraente. Ao correlacionar os parâmetros de cor ao teor total de antocianinas monoméricas, observou-se uma fraca correlação, sugerindo que as medidas de cor não podem ser indicadoras do nível de antocianinas nas amostras de pós de açaí produzidas por *cast-tape drying* nas condições avaliadas.

Considerações Finais

Esta tese propôs técnicas alternativas de secagem para a obtenção de açaí em pó, objetivando a produção de pós de alta qualidade em curtos tempos de processo. A aplicação de pressões reduzidas ou adição de hidrocolóides nos processos de secagem condutivos (secagem em tambor e *cast-tape drying*, respectivamente) resultaram em pós de açaí obtidos em, aproximadamente, 30 min de secagem com alta retenção de antocianinas e atividade antioxidante. Contudo, a estabilidade dos pós de açaí à oxidação lipídica durante os processos de secagem condutivos ainda é uma preocupação do ponto de vista sensorial e nutricional, uma vez que essas técnicas alternativas favoreceram a formação de produtos secundários da oxidação lipídica (aldeídos) em maior concentração do que os métodos convencionais (secagem com ar quente e liofilização). Assim, estudos futuros mais detalhados sobre a aceitabilidade dos pós de açaí pelos consumidores, bem como a avaliação dos benefícios/riscos do consumo dos pós para a saúde humana são recomendados.

Palavras-chave: Secagem condutiva. Antocianinas. Oxidação lipídica.

ABSTRACT

Açaí (*Euterpe oleracea* Mart.) contains a high nutritional value, being an important source of anthocyanins with high antioxidant activity and unsaturated fatty acids. However, it is a highly perishable fruit. Drying açaí juice is an interesting alternative to increase its shelf-life, add value, reduce losses, diversify consumption options, and decrease transport and storage costs as well. Freeze-drying and spray drying are the most common drying methods applied in the literature and food industry to produce açaí powder. Nevertheless, the main disadvantages of these drying techniques for dehydration of açaí juice are the high cost of the freeze-drying process, the low energy efficiency, and the requirement of a previous filtration of açaí juice for spray drying. Therefore, this thesis aimed to apply alternative drying technologies to produce açaí powder. The effect of the conductive thin-film drying processes, namely drum drying and cast-tape drying, on the quality of açaí powders was investigated. The quality of the final product was based on anthocyanin retention, antioxidant activity preservation, lipid content, and lipid oxidation. The thesis was divided into three sections. The first one was to assess the effect of drying pressure (vacuum and atmospheric pressures) on the physicochemical properties of açaí powders. The study was performed at the laboratory and pilot scale. Hot air drying and freeze drying were carried out as reference processes. The results showed that conductive drying at low pressures contributed to the quality preservation of açaí powder. In addition, the chamber pressure during conductive thin film drying should be carefully selected to obtain high-quality açaí powder. In the second section of the thesis, the impact of the addition of hydrocolloids (high methoxyl pectin, gum Arabic, and maltodextrin 10DE), at a concentration of 3% (w/w juice), on the physicochemical properties of açaí powders produced by cast-tape drying was investigated. The quality parameters were compared to those of freeze-dried açaí powders (without adding hydrocolloids). The addition of pectin to açaí juice hindered the oil separation during the drying process and helped remove the dried material like a continuous film from the drying support. Moreover, the incorporation of pectin resulted in a powdered product with color, anthocyanin content, and antioxidant activity similar to those of freeze-dried açaí powder. Finally, in the third section of the thesis, the stability of açaí powders, with and without the addition of pectin, produced by cast-tape drying was analyzed during storage at different relative humidities. Moisture sorption isotherms, total monomeric anthocyanin content, and instrumental color parameters were determined. The monolayer moisture contents estimated from moisture sorption isotherms were not enough to predict anthocyanin stability in the açaí powder. Furthermore, anthocyanin degradation was not the main factor responsible for color changes in the açaí powders stored under studied conditions. The results of this thesis suggest that the application of low pressures or the addition of hydrocolloids to açaí juice in the conductive drying processes are promising strategies for producing açaí powders in short drying times with excellent quality.

Keywords: Conductive drying. Anthocyanins. Lipid oxidation.

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LIST OF ABBREVIATIONS

ANOVA	Analysis of variance
AOAC	Association of Official Analytical Chemists
AOCS	American Oil Chemists' Society
CLSM	Confocal laser scanning microscopy
CTD	Cast-tape drying
CTFD	Conductive thin-film drying
d.b.	Dry basis
DE	Dextrose equivalent
DM	Degree of methoxylation
FD	Freeze-drying
FPE	Laboratory of Food Process Engineering
FTIR	Fourier transform infrared
HAD	Hot air drying
IBGE	Brazilian Institute of Geography and Statistics
LCME	Central Laboratory of Electron Microscopy
MAPA	Ministry of Agriculture, Livestock and Food Supply
NMR	Nuclear magnetic resonance
RH	Relative humidity
RWD	Refractance window drying
SD	Spray drying
TE	Trolox Equivalent
VDD	Vacuum drum drying
w.b.	Wet basis
^{1}H NMR	Proton nuclear magnetic resonance

LIST OF SYMBOLS

α	Significant level	
a^*	a^* chromaticity	
a_w	Water activity	
b^*	b^* chromaticity	
C_{GAB}	Guggenheim constant	
C_{BET}	BET constant	
δ	NMR chemical shift	ppm
ΔE^*	Total color difference	
ε	Molar absorptivity	
G′	Elastic or storage modulus	Pa
G″	Viscous or loss modulus	Pa
k	GAB constant	
L^*	Luminosity	
P	p-value	
r	Coefficient of correlation	
R^2	Coefficient of determination	
X_{eq}	Equilibrium moisture content	g g^{-1}
X_m	Monolayer moisture content	g g^{-1}

CONTENTS

THESIS CONCEPTUAL SCHEME

CONDUCTIVE THIN-FILM DRYING METHODS FOR PRODUCING AÇAÍ (*Euterpe oleracea* Mart.) POWDER
Why? - Açaí is considered a superfruit, rich in fibers, proteins, unsaturated lipids, and anthocyanins with high antioxidant activity. - Anthocyanins in açaí juice can readily degrade when exposed to high temperature, water, light, and oxygen during processing and storage. - Lipids in açaí powder are liquid at room temperature, making them prone to oxidation. - Conductive thin-film drying of açaí juice is a feasible alternative for several production scales.
State of the art - Most studies in the literature use freeze-drying and spray-drying to produce açaí powder. - Few reports on applying conductive thin-film drying methods to produce dried açaí. - The literature is scarce on the evaluation of the impact of storage relative humidity on the stability of açaí powder from conductive drying.
Hypothesis - It is possible to produce açaí powder by conductive thin-film drying methods at short process times and with quality comparable to the freeze-dried açaí powder.
Experimental steps - Production of açaí powder by conductive thin-film drying under different drying pressures at laboratory and pilot-scale. - Addition of different hydrocolloids to açaí juice for further processing by cast-tape drying. - Evaluation of the açaí powder quality after the drying processes based on anthocyanin content, antioxidant activity, and lipid fraction. - Comparison of the quality of açaí powders produced by conductive thin-film drying with that from freeze-dried açaí powders. - Assessment of açaí powder stability dehydrated by cast-tape drying at different storage relative humidity.
Response - Production of açaí powder using conductive thin-film drying methods competitively to other drying processes remaining anthocyanins and lipid quality as much as possible.

GENERAL INTRODUCTION

Açaí (*Euterpe oleracea* Mart.) has attracted the attention of industries and scientists due to its high nutritional value, being considered a superfruit. Açaí is rich in fibers, proteins, and unsaturated fatty acids (e.g., omega-6 and omega-9). It is also an important source of anthocyanins, with high antioxidant activity (SCHAUSS et al., 2006; YAMAGUCHI et al., 2015). Nevertheless, fresh açaí is highly perishable, showing a short shelf life, even under refrigeration. Moreover, anthocyanins and lipid fraction stability in açaí depends on the processing and storage conditions. Anthocyanins are sensitive compounds to high temperatures, humidity, light, and oxygen, among others (TONON, BRABET, and HUBINGER, 2010). The high monounsaturated and polyunsaturated fatty acids content in açaí increases its susceptibility to lipid oxidation (OLIVEIRA, NETO, and PENA, 2007). Therefore, processes that increase the shelf-life of açaí and preserve the fruit biocoumpounds as much as possible are required.

The production of açaí powder is an interesting alternative for prolonging the fruit shelf-life, as well as diversifying the product's consumption options. Freeze-drying and spray drying are the most cited drying processes in the literature for producing powdered açaí (LUCAS, ZAMBIAZI, and COSTA, 2018; PAVAN, 2010; TONON, 2009). Freeze-drying results in high-quality dried products. However, this technique has elevated costs because of the long operation time and high energy consumption (RATTI, 2008). Conversely, spray drying shows fast water evaporation, preventing excessive heating of the product. However, it has low energy efficiency and is not adequate to dry viscous liquids, such as açaí juice, which requires a previous filtration to facilitate the material's passage through the nozzle atomizer (QIU, 2019; TONON, BRABET, and HUBINGER, 2010). Thus, other drying techniques for obtaining açaí powder have been investigated to overcome these limitations.

Conductive thin-film drying may be an appropriate option to convert açaí juice into a powder, as it allows the use of viscous liquids and is energy efficient. Drum drying and cast-tape drying (or refractance window drying) are examples of conductive drying methods. During drying, thermal energy is transferred from steam condensation or hot water to a thin product film spread on a steel wall or flexible support. In drum drying, the material is exposed to high drying temperatures at atmospheric pressure, which can result in a loss of product quality. The application of reduced pressures (vacuum drum drying) can alleviate the undesired damage to thermal-sensitive and oxygen-sensitive compounds (e.g., anthocyanins and lipids) due to low boiling temperature and oxygen concentration (QIU, 2019). On the other hand, cast-tape drying or refractance window drying employs mild drying conditions (moderate drying temperatures

and relatively short drying times), which can be suitable for heat-sensitive foods (SIMÃO et al., 2020; ZOTARELLI et al., 2017). Nevertheless, Souza (2015) reported that the anthocyanin retention in the açaí powder produced by cast-tape drying was lower than that in freeze-dried açaí powder. Furthermore, the author observed that the conductive drying promoted the oil separation on the powder surface, which may become the product prone to lipid oxidation. The addition of hydrocolloids to açaí juice can be a strategy for protecting these biocompounds in açaí dried by cast-tape drying.

Therefore, this thesis focused on exploring strategies to develop açaí powders with high-quality produced by conductive thin-film drying techniques. The effects of drying pressures and the addition of hydrocolloids on quality parameters of the conductive thin-film dried products were investigated.

LITERATURE REVIEW

An overview of the main aspects of açaí fruit and its physicochemical properties are presented in this chapter. The impact of conventional and alternative drying methods on the quality of the dried açaí is also discussed. Moreover, important information about the quality parameters of açaí powders that support this research is described.

1.1 AÇAÍ

Euterpe oleracea Martius is a palm tree native to the Amazon region, abundantly found in the northern and northeastern regions of Brazil, as well as in some countries in South America (e.g., Venezuela, Colombia, Ecuador, Suriname, and Guyana) and Central America (e.g., Panama). In the northern region of Brazil, the açaí production is an important source of employment and income for the Amazonian population. According to the Brazilian Institute of Geography and Statistics (IBGE, 2022), the production of açaí in Brazil was 1,478,168 tons in 2020, concentrated in the State of Pará with a production of 1,389,941 tons, representing approximately 94% of national production. Most Brazilian açaí production is destined for the domestic market, but it is also consumed in many countries in Europe, North America, and Asia. The American and Japanese markets are responsible for 90% of açaí export (MAPA, 2022).

The growing interest in the consumption of açaí has been generated by its exotic flavor and potential benefits for human health, such as antioxidant, anti-proliferative, anti-inflammatory, and cardioprotective properties. These health effects are attributed to its chemical composition (Table 1), which is rich in fibers, proteins, unsaturated fatty acids, and anthocyanins with high antioxidant activity (NOGUEIRA, FIGUEIRÊDO, and MÜLLER, 2005; PACHECO-PALENCIA, DUNCAN, and TALCOTT, 2009; YAMAGUCHI et al., 2015). The content of unsaturated fatty acids in açaí represents approximately 74% of all fatty acids in the fruit. Oleic acid (monounsaturated fatty acid) and linoleic acid (polyunsaturated fatty acid) are the predominant unsaturated fatty acids, constituting around 60% and 13%, respectively, of the total fatty acids in açaí (SCHAUSS et al., 2006). The high content of anthocyanins accounts for the high antioxidant activity of açaí juice, as well as the violet color of the fruit (BICHARA and ROGEZ, 2011). Açaí presents higher antioxidant activity than other anthocyanin-rich fruits such as blueberry, blackberry, raspberry, and strawberry (DEL POZO-INSFRAN, BRENES, and TALCOTT, 2004). Cyanidin-3-glycoside

and cyanidin-3-rutinoside are the major anthocyanins in açaí (GORDON et al., 2012; PACHECO-PALENCIA, HAWKEN e TALCOTT, 2007; SCHAUSS et al., 2006).

Table 1 - Chemical composition of açaí juice.

Component	Tonon (2009)	Costa et al. (2015)	Souza (2015)
Moisture (g 100 g^{-1} w.b.)	85.96	83.82	80.97
Proteins (g 100 g^{-1} d.b.)	10.19	9.83	9.58
Lipids (g 100 g^{-1} d.b.)	46.51	37.82	46.79
Carbohydrate (g 100 g^{-1} d.b.)	3.42	48.02	3.74
Total fiber (g 100 g^{-1} d.b.)	32.19	11.12	36.84
Ash (g 100 g^{-1} d.b.)	3.13	4.20	3.53
Anthocyanins (mg 100 g^{-1} d.b.)	1265.14	341.10	706.90

Note: w.b.: wet basis; d.b.: dry basis; Anthocyanins content expressed in terms of cyanidin-3-glycoside.

Even though all benefits of açaí, the fruit is highly perishable, showing a short shelf-life. After harvesting, the fruit must be pulped within 24 h. The fresh açaí juice, prepared by adding water while the pulp is mechanically extracted, has a shelf-life of 12 h, even under refrigeration (BICHARA and ROGEZ, 2011; NOGUEIRA, FIGUEIRÊDO, and MÜLLER, 2005). Hence, açaí juice should be frozen or dried to increase its shelf-life. Frozen juice is the traditional commercialization way of açaí, despite the large volumes of storage and high transport costs. In turn, the drying of açaí juice is an attractive alternative because it adds value, diversifies consumption options, and facilitates storage and transport. Nevertheless, the drying techniques and conditions should be carefully selected to obtain high-quality dried açaí.

1.2 DRYING OF AÇAÍ

Drying is one of the widely applied processing methods in the food industry to extend the shelf life of products since the water activity is reduced at very low levels, hindering, thereby, microbial spoilage and many chemical reactions. Drying also facilitates the storage and transportation of foods by decreasing product weight and volume. In addition, it provides more product options for consumers (SINGH and HELDMAN, 2014).

The açaí juice can be dried into açaí powder to increase its shelf-life, add value, diversify consumption options, reduce losses, and facilitate commercialization. The powdered product can be used as an ingredient in healthy drinks, yogurt, ice cream, baked goods, baby foods, desserts, and cereal bars. Table 2 shows the studies reported in the literature on the drying of açaí juice for producing açaí powder. Freeze-drying and spray drying are the most applied

techniques to convert açaí juice into powder (LUCAS, ZAMBIAZI, and COSTA, 2018; OLIVEIRA et al., 2021; PAVAN, 2010; TONON, 2009).

Freeze-drying has been considered an adequate technique for obtaining high-quality dried products because of the low temperature and low oxygen concentration during the drying process. It is based on drying by directly sublimating the frozen product's ice fraction under reduced pressures (RATTI, 2008). Lucas, Zambiazi, and Costa (2018) compared the açaí powder produced by freeze-drying to those from spouted bed drying and spray drying. The authors reported that the freeze-drying resulted in higher anthocyanin and carotenoid retention. Moreover, the lipid fraction (oil) in the freeze-dried açaí powder did not undergo oxidation during the drying process. Although many advantages, freeze-drying is a costly process due to the long operation time and high energy consumption, restricting its use on an industrial scale (RATTI, 2008).

Spray drying is a widely applied process in the food industry to dry fruit pulps or juices into powder. It is a convective drying process, in which the hot air contacts the wet sprayed droplets for moisture removal. This process shows fast water evaporation, thus preventing excessive heating of the product. However, spray drying has low energy efficiency due to energy loss by the warm and moist exhaust gas and is not suitable for drying viscous liquids, such as açaí juice (QIU, 2019). Tonon (2009) filtered the açaí juice before entering the spray dryer to eliminate solids in suspension, facilitating the passage of the material through the nozzle atomizer. However, this operation resulted in a significant nutritional loss for the product, as it reduced the fiber and lipid content. Another disadvantage of spray drying is the addition of a high amount of hydrocolloids, which are high molecular weight compounds, to reduce the stickiness of fruit products on the metallic wall of the spray dryer (FRABETTI et al., 2021).

Hence, alternative drying methods have been proposed to reduce process cost, increase energy efficiency, and still retain product quality as much as possible. Conductive thin-film drying can be a good option for achieving those goals, as discussed in the following section.

Table 2 - Studies reported in the literature on the drying of açaí juice (*Euterpe oleracea* Mart.) to produce açaí powder.

Raw material	**Additives**	**Drying technique**	**Main results**	**References**
Filtered açaí juice: total solid content of 3%, lipid content of 0.21%, and no fibers	Maltodextrin 10DE, maltodextrin 20DE, gum Arabic, and tapioca starch 6% (w/w)	Spray drying	- Açaí powders added with tapioca starch showed the highest anthocyanin loss. - Adverse effect of temperature and water activity on anthocyanin stability. - Maltodextrin 10DE displayed the highest anthocyanins protection during storage at different temperatures (25 °C and 35 °C) and water activities (32.8% and 52.9%) conditions. - Açaí powders added with carrier agents showed higher anthocyanin retention than pure açaí powder from freeze-drying.	TONON (2009)
Pasteurized açaí juice: total solid content of 11-13%, total soluble solids of 2-5 °Brix, pH < 4.6	-	Freeze-drying, hot air drying, and refractance window drying	- Anthocyanin retention: Freeze-drying > refractance window drying > hot air drying. - Lipids present in açaí powders were liquid at room temperature. - Dried samples with water activity ranging between 0.119 and 0.240 showed a significant increase in the concentration of lipid oxidation-derived compounds after 3 months of storage at 25 °C in nitrogen flush amber glass bottles.	PAVAN (2010)
Açaí juice: total solid content of 11%, lipid content of 4.2%, and total fibers content of 1.2%	Maltodextrin 10DE 15, 20, and 25% (w/w)	Spouted bed drying	- Anthocyanin loss in the açai powders obtained at different conditions ranged from 21% to 76%. - Airflow rate was the variable that most influenced the degradation of anthocyanins.	COSTA et al. (2015)
Açaí juice: total solid content of 19%, lipid content of 8.9%, fibers content of 7.0%, total soluble solids of 10 °Brix, and pH 5.0	-	Cast-tape drying	- Formation of a superficial layer on the spread açaí juice during drying, hindering the moisture removal. - Açaí powders obtained from higher drying temperatures showed a higher distribution of oil molecules on the surface of the particles. - The drying of açaí powders resulted in an anthocyanin degradation of 46-62%.	SOUZA (2015)

Table 2 (continued)

Raw material	Additives	Drying technique	Main results	References
Açaí juice: total solid content of 19% and lipid content of 10% (For spray drying, the total solid content of açaí juice was 3.9%)	-	Spouted bed drying, freeze-drying, and spray drying	- Freeze-drying resulted in the highest bioactive compounds (carotenoids and anthocyanins) retention. - Spray-dried açaí powder showed the lowest anthocyanin content. - Freeze-drying did not result in the lipid oxidation of açaí oil.	LUCAS, ZAMBIAZI, and COSTA (2018)
Açaí juice: total solid content of 5.4% and lipid content of 2.4%	-	Infrared-assisted freeze-drying	- Infrared-assisted freeze-drying showed a shorter drying time than traditional freeze-drying. - Açaí powder produced by infrared-assisted freeze-drying was darker and showed higher antioxidant activity than traditional freeze-dried samples due to the Maillard reaction. - Infrared-assisted freeze-drying did not influence the phenolic content. - Far infrared-assisted freeze-drying resulted in an açaí powder with lower anthocyanin content than traditional freeze-drying.	OLIVEIRA et al. (2021)

Note: The percentage data of the raw material section are expressed on a wet basis.

1.2.1 Conductive thin-film drying

Conductive thin-film drying is suitable for converting viscous food products, such as fruit and vegetable purees, into sheets, flakes, or powders. During the drying process, the thermal energy is provided by steam condensation or hot water and transferred mainly by conduction to a thin layer of product spread on a steel wall or flexible support. Conductive drying has been applied to obtain high-quality dried products in short drying times (ABONYI et al., 2001; DESOBRY, NETTO and LABUZA, 1997; GALAZ et al., 2017; QIU et al., 2019a; SIMÃO et al., 2021). Furthermore, it is an energy-efficient process, consuming around half energy of freeze-drying and having lower energy loss by exhaust gas than convective drying (BAZYMA et al., 2006; DEVAHASTIN and MUJUMDAR, 2015). Drum drying and cast-tape drying or refractance window drying are examples of conductive thin-film drying technologies.

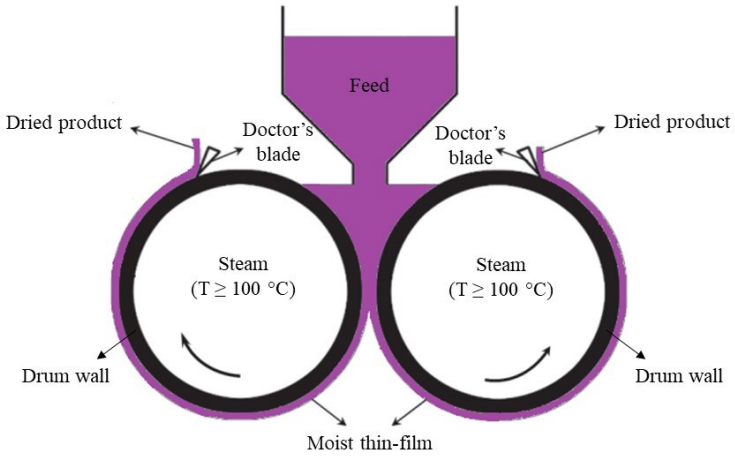
1.2.1.1 Drum drying

Atmospheric drum drying is commonly used in the food industry to dry high-viscosity liquids and pureed foods, such as starch slurries, milk products, and fruit purees. A drum dryer mainly consists of one or two horizontal hollow drums made of cast iron or stainless steel, a product feeding, a heating system, and a scraper (TANG, FENG, and SHEN, 2003). A schematic representation of the atmospheric double drum dryer is displayed in Figure 1. During operation, the material is spread as a thin layer on the outer surface of the rotating drum, which is heated internally by steam condensing. The adhering material is rapidly dried (a few seconds) and then scraped off from the drum surface by a doctor's blade. Most of the moisture in the material is removed at the water boiling temperature. The dried product can be ground into flakes or powder (DAUD, 2015; TANG, FENG, and SHEN, 2003). The major drawback of this drying method is the severe quality loss in heat-sensitive products due to the high-temperature exposure of material (commonly at 120-170 °C) during the drying process. Enclosing the drums in a vacuum chamber to dry the material under reduced pressure (vacuum drum drying) can alleviate undesired damage to thermal-sensitive and oxygen-sensitive compounds due to the lower boiling temperature and oxygen concentration. However, the high capital cost of vacuum drum dryers is still a limitation for several industrial applications (NINDO and TANG, 2007).

Qiu et al. (2019a) compared the organoleptic quality of tomato powders produced by atmospheric drum drying and vacuum drum drying. The authors observed that the drying

methods influenced the volatile and non-volatile profiles in the tomato powders, which might be related to aroma retention and taste perception, respectively. Volatile compounds were more abundant in vacuum drum-dried powders, whereas atmospheric drum drying resulted in tomato powders with a higher concentration of non-volatile compounds.

Figure 1 - Schematic diagram of the atmospheric double drum dryer.



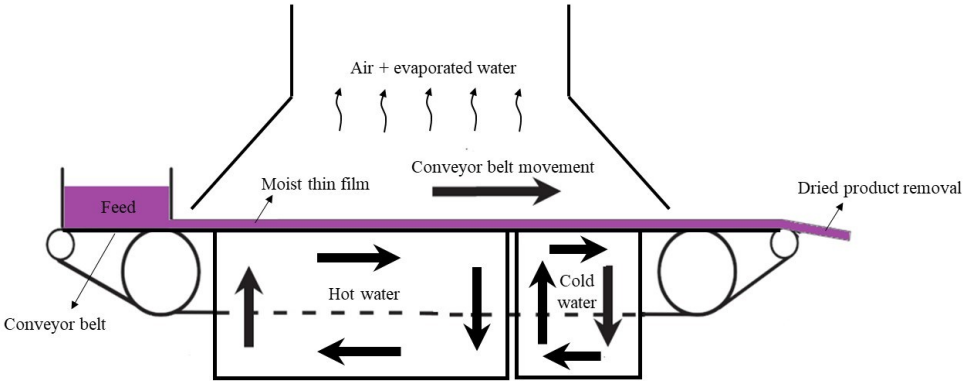
Source: adapted from Qiu (2019)

1.2.1.2 Cast-tape drying

Cast-tape drying or refractance window drying (a particular case, as discussed in the following) is a relatively novel drying technique that allows dehydration of thermosensitive foods due to mild drying conditions. It is operated below boiling temperature (usually at 90-98 °C) at atmospheric pressure and relatively short process times (a few minutes) (RAGHAVI, MOSES, and ANANDHARAMAKRISHNAN, 2018; SIMÃO et al., 2020; WAGHMARE, 2021). During this process, a viscous solution or suspension is spread as a thin layer and dried over flexible support, which is heated on its bottom face by hot water or steam (Figure 2). The flexible support can be a polyester film, commercially known as Mylar®, or a fiberglass film coated with Teflon®, which are semitransparent or not transparent to infrared radiation, respectively. The Teflon-based film has low surface energy resulting in a low adhesion of the dried product to the support (DURIGON et al., 2018). At the end of the drying process, the dried product passes over a colling section to decrease its temperature below the glass transition temperature, reducing, thereby, product stickiness and facilitating its scraping-off (NINDO and TANG, 2007; SIMÃO et al., 2020). Nevertheless, Frabetti et al. (2021)

reported difficulty in completely removing strawberry leathers from the Teflon-based support after moving over the colling section. Hence, the colling zone of the equipment was switched off, and the products were detached from the support at the end of the heating section, where they were more pliable than the cooled films.

Figure 2 - Schematic diagram of the cast-tape drying.



Source: adapted from Qiu (2019)

The drying process of fruits and vegetables reported in the literature as refractance window drying is a variant of cast-tape drying. During the refractance window drying process, hot water and polyester film (partially transparent to infrared radiation) are used as the heating medium and the drying support, respectively. However, Durigon et al. (2018) reported that steam as the heating medium simplifies temperature control and equipment construction. Moreover, Ortiz-Jerez et al. (2015) and Zotarelli et al. (2015) showed that the heat transfer by radiation accounts for less than 5% of the total heat supplied to the material during drying. This result implies that most heat is transferred by conduction through the drying support. Consequently, non-transparent drying supports, such as fiberglass film coated with Teflon®, may replace the polyester film. Therefore, cast-tape drying is a general and more appropriate denomination for this drying process.

Pavan (2010) and Souza (2015) studied the production of açaí powder using refractance window drying. Pavan (2010) evaluated the effect of refractance window drying, hot air drying, and freeze-drying on the açaí powder quality. The author reported that freeze-drying resulted in the highest anthocyanin retention. The anthocyanin losses in the açaí samples dried by freeze-drying, refractance window drying, and hot air drying were 5%, 15%, and 51%, respectively. In addition, the author observed that lipids present in açaí powders, regardless of the drying process applied, were liquid form at room temperature, thus having higher mobility

to participate in degradation reactions, such as lipid oxidation. Souza (2015) also found that açaí powder obtained by refractance window drying showed lower anthocyanin retention than freeze-dried powder. The refractance window drying process also contributed to the oil separation on the powder surface, which can become the product susceptible to lipid oxidation. In this context, the addition of hydrocolloids, such as polymers and gums, to açaí juice before drying can protect these sensitive compounds, as well as improve their stability (TONON et al., 2009c).

1.3 ADDITION OF HYDROCOLLOIDS

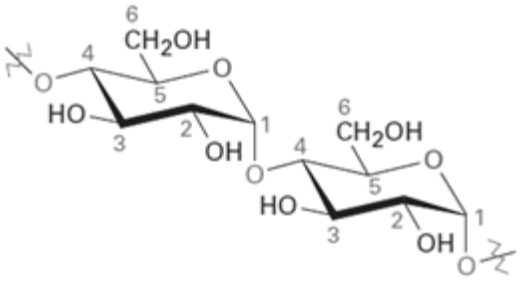
Hydrocolloids can be added to fruit pulp or juice before drying to protect sensitive foods or ingredients from oxygen, moisture, temperature, and/or light during processing and storage. These additives are composed of hydrophilic and/or hydrophobic groups that create a polymer network (TURCHIULI et al., 2005). Tonon et al. (2009c) reported the protective effect of the hydrocolloids (maltodextrin 10DE, maltodextrin 20DE, and gum Arabic) on polyphenolics' retention and antioxidant activity preservation of spray-dried açaí during storage at 40 °C at two different water activities for 15 days. The authors observed that the freeze-dried pure açaí (produced without any hydrocolloids) showed a more pronounced decrease in those properties than spray-dried açaí samples, thus affirming the protective effect of the hydrocolloids on the açaí powders. The hydrocolloids used in this present work are discussed below.

1.3.1 Maltodextrin

Maltodextrin is one of the most applied hydrocolloids in the drying of fruit pulps or juices, mainly due to its bland flavor and low viscosity at high concentrations (TELIS and MARTÍNEZ-NAVARRETE, 2009). Maltodextrins are starch hydrolysates produced by partial hydrolyzing starches using acids or enzymes. Its chemical structure (Figure 3) consists of D-glucose units liked by α (1→4), with a dextrose equivalent (DE) less than 20 (KENNEDY, KNILL, and TAYLOR, 1995). The DE value is related to the degree of starch hydrolysis, i.e., the lower the DE value, the more similar the hydrolysate properties to starch ones, whereas the higher the DE value, the more similar the hydrolysate properties to dextrose ones. Higher DE maltodextrins create a dense and less oxygen permeable network, providing a better protective

effect to oxygen-sensitive compounds (CAI and CORKE, 2000; SHAHIDI and HAN, 1993; WAGNER and WARTHESEN, 1995). However, these compounds are more prone to physical changes during storage (for example, caking) since they are high hygroscopic. On the other hand, lower DE maltodextrins have the biding properties of starch are thus may more effectively bind to lipids than higher DE maltodextrins (KENNEDY, KNILL, and TAYLOR, 1995).

Figure 3 - Structure of maltodextrin.

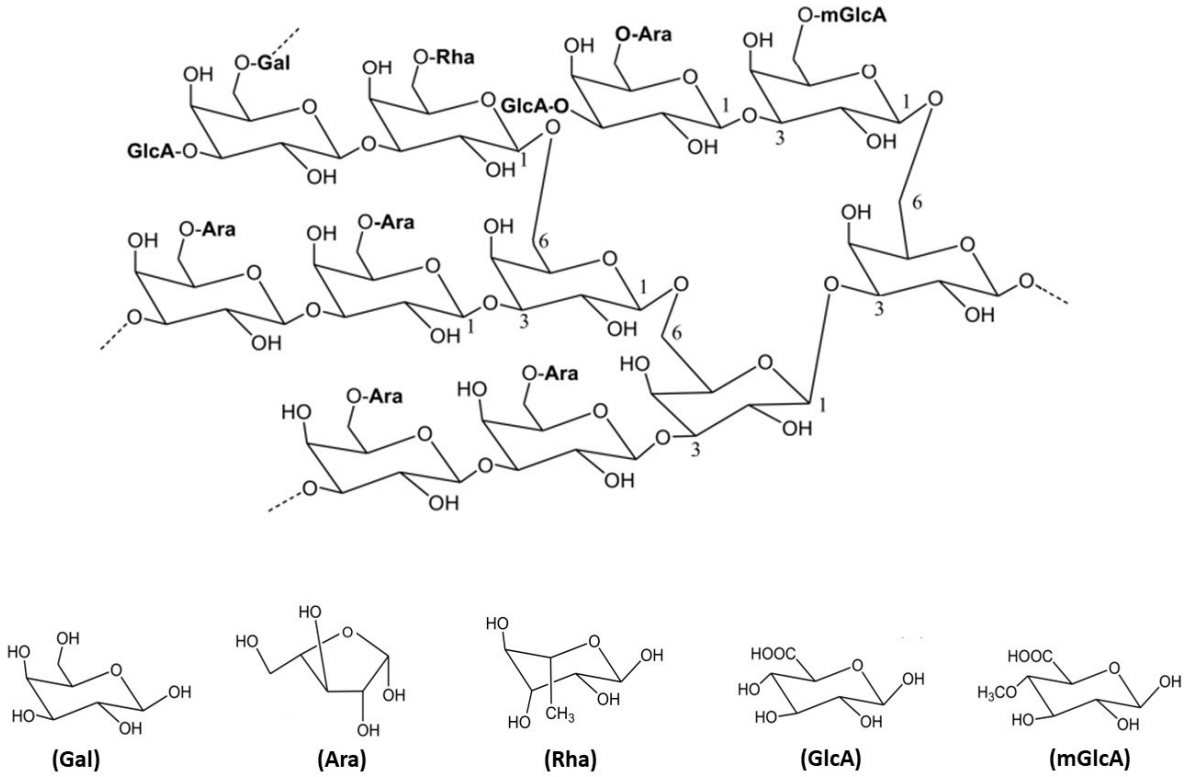


Source: adapted from McMurry (2018)

1.3.2 Gum Arabic

Gum Arabic or acacia gum is the most commonly used gum in drying fruit pulps or juices because of its high solubility, low viscosity, and emulsification properties (TELIS and MARTÍNEZ-NAVARRETE, 2009). This hydrocolloid is a complex heteropolysaccharide produced by the natural exudation of Acacia trees. It comprises a highly branched structure (Figure 4) with the main chain of (1→3)-linked β-D-galactopyranosyl units and side chains of two to five (1→3)-linked β-D-galactopyranosyl units joined to the main chain by (1→6)-linked. Both main and side chains are composed of α-L-arabinofuranosyl, α-L-rhamnopyranosyl, β-D-glucuronopyranosyl, and 4-*O*-methyl-β-D-glucuronopyranosyl units (HUBER and BEMILLER, 2017). Gum Arabic also contains a protein fraction of approximately 2% (w/w) covalently bound within its molecular arrangement, which plays a crucial role in the functional properties of Arabic gum (TURCHIULI et al., 2005).

Figure 4 - Structure of carbohydrates fraction of gum Arabic. β-D-galactopyranose (Gal), α-L-arabinofuranose (Ara), α-L-rhamnopyranose (Rha), β-D-glucuronic acid (GlcA), and 4-*O*-methyl-β-D-glucuronic acid (mGlcA).



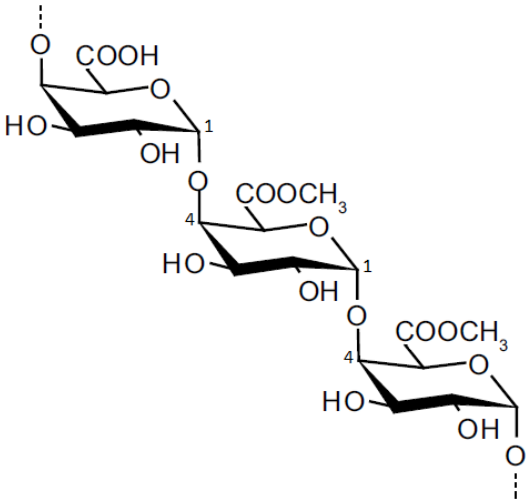
Source: adapted from Ashour et al. (2022)

1.3.3 Pectin

Pectin is another potential hydrocolloid for protecting sensitive compounds present in açaí juice since it has been used for protecting various bioactive compounds, such as polyphenols, vitamins, and essential oils (REHMAN et al., 2019). Moreover, pectin is commonly used in the food industry as a gelling agent, thickener, emulsifier, and stabilizer (ESPITIA et al., 2014). This hydrocolloid is commercially obtained from apple pomace and citrus peel (e.g., orange, lemon, and lime) (HUBER and BEMILLER, 2017). Pectin is a complex polysaccharide that mostly contains a linear chain of (1→4)-linked α-D-galacturonic acid units with varying contents of methyl ester groups (Figure 5). Some of the carboxyl groups (-COOH) of the galacturonic acids are esterified with methanol, and the percentage of carboxyl groups in the methyl ester form (-$COOCH_3$) is defined as the degree of methoxylation (DM). Pectin is classified according to the DM as high-methoxyl pectin (DM ≥ 50%) and low-methoxyl pectin (DM < 50%) (HUBER and BEMILLER, 2017; MESBAHI, JAMALIAN and

FARAHNAKY, 2005). The functional properties of pectin depend on its DM, e.g., high methoxyl pectin is highly hydrophobic and thus may interact with hydrophobic molecules (REHMAN et al., 2019).

Figure 5 - Structure of pectin.



Source: adapted from Ali, El-Regal, and Saeed (2015)

1.4 QUALITY PARAMETERS OF AÇAÍ POWDERS

The quality of powders is directly related to their production process, which influences their stability during storage. The knowledge of factors that affect the quality of food powders, their microstructure, and their chemical composition is essential to optimize processes and minimize the rates of undesirable reactions. The açaí powder quality was in this thesis based on the anthocyanin content, antioxidant activity, and lipid oxidation.

1.4.1 Anthocyanins and antioxidant activity

Anthocyanins, which are phenolic compounds belonging to the flavonoid group, are water-soluble pigments responsible for various colors in plants, including blue, purple, red, and orange. Furthermore, these compounds have a significant antioxidant activity, which benefits human health (CASTAÑEDA-OVANDO et al., 2009). According to Pacheco-Palencia, Duncan, and Talcott (2009), anthocyanins account for approximately 90% of the antioxidant activity of açaí. Nevertheless, anthocyanins are highly unstable and susceptible to degradation during the processing and storage of foods, resulting in undesirable changes in colors and

antioxidant activity. Their stability depends on many factors, such as temperature, water activity, light, oxygen, pH, and ascorbic acid, among others (CASTAÑEDA-OVANDO et al., 2009; WROLSTAD, DURST, and LEE, 2005). Studies on the effect of these factors on the anthocyanin stability and antioxidant activity of açaí products during drying and storage have been reported in the literature. For example, Tonon, Brabet, and Hubinger (2008) evaluated the influence of inlet air temperature, feed flow rate, and maltodextrin concentration on the anthocyanin retention of açaí juice dried by spray drying. Costa et al. (2015) dried açaí juice by spouted bed drying and assessed the effect of the drying air temperature, airflow rate, and maltodextrin concentration on anthocyanin degradation. Souza (2015) reported on the effect of the spreading thickness and hot water temperature on the anthocyanin concentration and antioxidant activity of the refractance window dried-açaí juice. Tonon, Brabet, and Hubinger (2010) stored açaí powders at two different temperatures and water activities, and the authors verified the effect of these conditions on the anthocyanin degradation and antioxidant activity. In general, the authors observed that increasing temperatures, oxygen availability and/or water activity negatively affected the stability of anthocyanins and antioxidant activity.

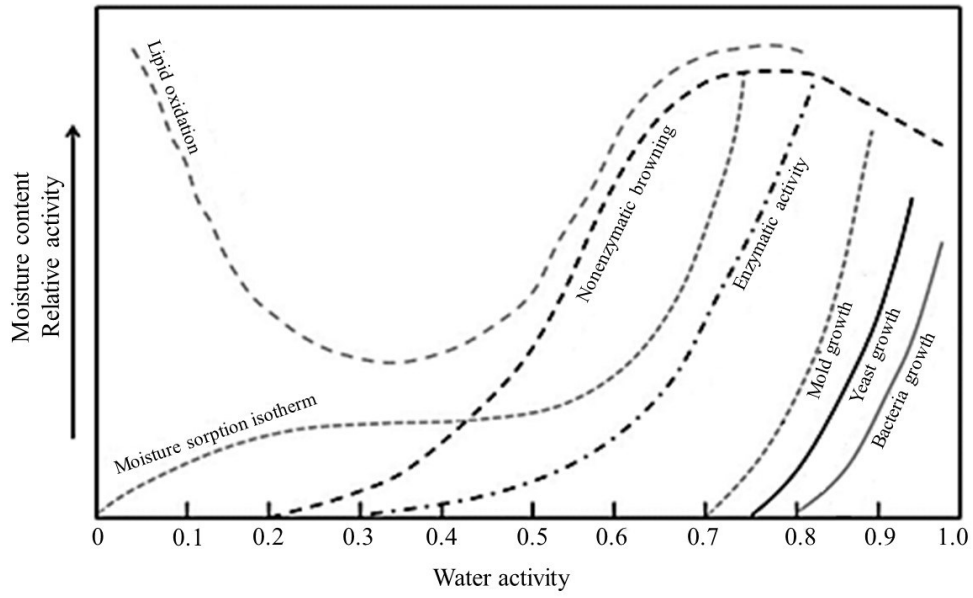
Several *in vitro* assays have been applied to determine the antioxidant activity of food products. These methods may be based on organic radical scavenging capacity (ABTS, DPPH), metal-reducing power (FRAP), and peroxyl radical scavenging capacity (ORAC, TRAP), among others (GONÇALVES et al., 2018; RUFINO et al., 2010). ORAC, ABTS, DPPH, and FRAP assays are the most commonly used methods to evaluate the antioxidant activity of plant extracts. However, ORAC shows a high variability of results caused by the temperature variation in the plate readers used in this assay (SEERAM et al., 2008). ABTS and FRAP assays are generally indicated for hydrophilic compounds (RUFINO et al., 2010). In contrast, the DPPH method can be applied for aqueous-organic extracts containing hydrophilic and hydrophobic compounds, such as extracts from açaí juice or powder. Moreover, the DPPH method is accurate, provides relevant information about the general activity of the antioxidant compounds, and employs a stable radical (GONÇALVES et al., 2018).

1.4.2 Lipid oxidation

Lipids can undergo oxidation during food processing and storage. Lipid oxidation is a complex process of reactions between unsaturated fatty acids and active oxygen, in which are firstly formed the primary products (peroxides, especially hydroperoxides) and once exposed

to extended oxidation conditions, give rise to secondary oxidation products (e.g., aldehydes, ketones, acids, and alcohols). Lipid oxidation is responsible for negative quality changes in foods, for example, the development of off-flavors, loss of nutrients, and the formation of compounds with possible risks to human health (BARRIUSO, ASTIASARÁN, and ANSORENA, 2013; VERCELLOTTI et al., 1992). Lipids can be oxidized by three main pathways: (1) the free radical mechanism or autoxidation; (2) the singlet oxygen mechanism or photooxidation; and (3) the enzyme-catalyzed oxidation (BARRIUSO, ASTIASARÁN, and ANSORENA, 2013). The degree of the unsaturation of fatty acids, the exposure to light and/or heat, and the presence of oxygen, enzymes, metals, and antioxidants influence lipid oxidation rate (AMARAL, SILVA, and LANNES, 2018). Moreover, water activity plays a crucial role in lipid oxidation. Lipid oxidation shows a peculiar behavior with a minimal rate at the monolayer moisture, which commonly occurs at water activity between 0.3 and 0.5, and an increase in the reaction rate below and above the monolayer, as illustrated in Figure 6 (RAHMAN and LABUZA, 2007; TAOUKIS, LABUZA, and SAGUY, 1997). At high water activity, the lipid oxidation rate increases due to the increase in the mobility of reactants (such as oxygen and metal ions). An acceleration of lipid oxidation also occurs at low water activity because of the loss of the protective effect of water surrounding lipids (BELL, 2007; MCCLEMENTS and DECKER, 2017; RAHMAN and LABUZA, 2007).

Figure 6 - Effect of water activity on relative rates of deterioration reactions in foods.



Source: adapted from Labuza et al. (1972)

The determination of lipid oxidation can be performed by conventional methods, including peroxide value, *p*-anisidine value, TBA (thiobarbituric acid) assay, and gas chromatography, as well as by alternative techniques, such as nuclear magnetic resonance. The primary lipid oxidation products (peroxides analysis) have been commonly and widely measured by iodometry titration assay (AOCS, 2009) mainly due to the procedure's simplicity (BARRIUSO, ASTIASARÁN, and ANSORENA, 2013). However, this official method is not able to determine the peroxide value in colored lipids, such as açaí oil, which has a dark green color (SILVA and ROGEZ, 2013), because the color change of the indicator solution cannot be identified (GOTOH et al., 2011). Many secondary lipid oxidation products are volatile; thus, headspace gas chromatography can be used to identify and quantify these compounds. The hexanal, an aldehyde derived from the oxidation of linoleic acid, is often used as an indicator of lipid oxidation in foods since its formation is usually higher than that of the other secondary oxidation products (BARRIUSO, ASTIASARÁN, and ANSORENA, 2013). On the other hand, nuclear magnetic resonance is an accurate method that allows the simultaneous determination of both primary and secondary lipid oxidation products in foods (MERKX et al., 2018).

The evaluation of lipid oxidation in açaí powders has been little published in the literature. Lucas, Zambiazi, and Costa (2018) analyzed the primary and secondary lipid oxidation products in freeze-dried açaí. Pavan (2010) monitored the concentration of volatile compounds indicative of lipid oxidation in açaí powders produced by freeze-drying, refractance window drying, and hot air drying during storage.

RESEARCH OBJECTIVES

Based on the theoretical background discussed previously, the overall objective of this project was to evaluate the quality of açaí powders produced by conductive thin-film drying methods, considering the anthocyanin content, antioxidant activity, and lipid oxidation during drying and storage. The primary hypothesis was that conductive thin-film drying methods would result in açaí powders with quality comparable to the freeze-dried açaí powder at short process times.

The specific objectives were:

a) Evaluate the effect of drying pressure on the quality of açaí powder produced by conductive thin-film drying at laboratory and pilot scales.

b) Investigate the influence of hydrocolloids' addition on the quality of açaí powders obtained by cast-tape drying.

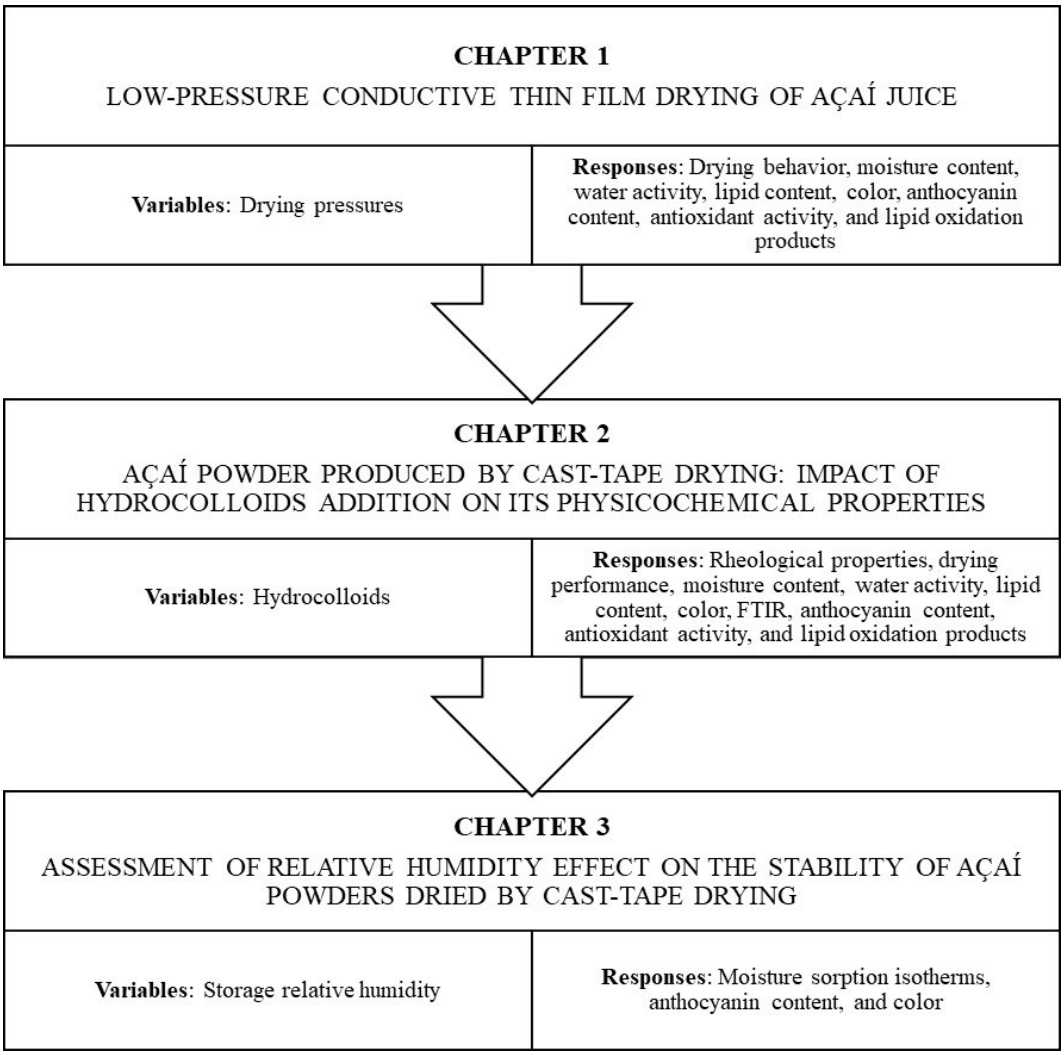
c) Compare the quality of açaí powders produced by conductive thin-film drying methods to that of freeze-dried açaí powder.

d) Study the anthocyanin stability in açaí powders from cast-tape drying stored at different relative humidities.

OUTLINE OF THIS THESIS

The research results of this thesis were divided into chapters, as presented in Figure 7, in order to facilitate the presentation and comprehension of them.

Figure 7 – Schematic overview of the content in this thesis.



CHAPTER 1. LOW-PRESSURE CONDUCTIVE THIN FILM DRYING OF AÇAÍ JUICE

The study performed in this chapter used a conductive thin-film dryer at a laboratory scale to mimic the (vacuum) drum drying of açaí juice. The effect of drying chamber pressure on the drying kinetics and powdered product characteristics was investigated. Hot air drying and freeze-drying experiments were performed as references. A pilot-scale conductive dryer, namely a vacuum drum dryer, was used as well to dry açaí juice. Conductive drying at low pressures led to lower boiling temperatures and shorter drying times but also more vigorous bubble formation. The anthocyanin content and antioxidant activity of contact-dried samples at low pressures were comparable to those of freeze-dried and significantly higher than those of hot air-dried. However, conductive drying caused greater lipid oxidation than freeze-drying and hot air drying. Oil loss was observed during vacuum drum drying except for the lowest pressure and the shortest drying time. Therefore, the results suggest that chamber pressure during conductive thin film drying should be carefully selected to obtain high-quality açaí powder. This study was carried out at the Laboratory of Food Process Engineering (FPE) at Wageningen University & Research (the Netherlands).

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1.1 INTRODUCTION

Drying is one of the widely applied processing methods in the food industry to reduce losses and extend the shelf life of products. Drying also facilitates the storage and transportation of foods by decreasing product weight and volume. In addition, it provides more product options for consumers. The quality of the dried product and the energy efficiency of the drying method are the two main concerns in the food drying process (KUMAR, KARIM, and JOARDDER, 2014). Hot air drying (HAD) is the most used technique for dehydrating fruits and vegetables because of its simplicity and low energy consumption. Nevertheless, this drying process results in nutritional and sensorial damage to the product due to the high air temperatures and long drying times. In contrast, freeze-drying (FD) has been found a very suitable drying method for producing high-quality dried fruits and vegetables since the process is conducted at low temperatures under a vacuum. Unfortunately, FD is a high-cost process because of the long drying time and high energy consumption, restricting its use on the industrial scale (RATTI, 2001; XU et al., 2006). Hence, alternative drying techniques have been investigated to decrease drying time, increase energy efficiency, and retain product quality as much as possible.

Conductive thin-film drying (CTFD) can potentially be used to produce high-quality dried products of fruits and vegetables with short drying times (QIU et al., 2019a; SIMÃO et al., 2021). It is an energy-efficient process, with an energy consumption comparable to HAD and around half of FD (BAZYMA et al., 2006). During CTFD, thermal energy is transferred mainly by conduction from hot water or steam condensation to a thin product film spread over a flexible support or steel wall. Conductive drying may be done by refractance window drying or cast-tape drying employing mild drying conditions, i.e., heating medium with a temperature just below boiling point (usually at 90-98 °C) at atmospheric pressure and relatively short drying times (few minutes), which can be adequate for thermosensitive foods (SIMÃO et al., 2020). Another option is atmospheric drum drying. One disadvantage of this drying method is that the product is exposed to high drying temperatures (commonly at 120-170 °C), resulting in quality loss. Therefore, drum drying is sometimes carried out under reduced pressure (vacuum drum drying), alleviating the thermal damage thanks to the lower boiling temperature, while lower oxygen concentration also can be an advantage (QIU et al., 2019a).

Açaí (*Euterpe oleracea* Mart.) is a Brazilian fruit with a high nutritional value, being rich in fibers, unsaturated fatty acids, and anthocyanins with high antioxidant capacity

(SCHAUSS et al., 2006). The interest in the consumption of açaí has grown worldwide due to the exotic flavor and benefits for human health, such as antioxidant, anti-proliferative, anti-inflammatory, and cardioprotective properties (PACHECO-PALENCIA, DUNCAN, and TALCOTT, 2009; YAMAGUCHI et al., 2015). However, fresh açaí is highly perishable. Therefore, the drying of açaí juice to produce açaí powder is an attractive alternative to prolong its shelf life. Furthermore, açaí powder may extend the consumption and commercialization of the fruit. The powdered product can be used as an ingredient in healthy drinks, yogurt, ice cream, baked goods, baby foods, desserts, and cereal bars. Pavan (2010) compared the influence of refractance window drying, hot air drying, and freeze-drying on the quality attributes of açaí powder. Nevertheless, no systematic studies have been conducted on the CTFD of açaí juice under reduced pressures to the best of our knowledge.

In this context, this study aimed to evaluate the effect of drying pressure on the quality of açaí powder produced by CTFD. The hypothesis was that drying under lower pressures would produce a higher quality product. For comparison, reference açaí powders were produced by HAD and FD. The product characterization was based on physical properties (moisture content, water activity, lipid content, and color) and quality markers (total monomeric anthocyanin content, antioxidant activity measured by DPPH assay, and lipid oxidation products quantified by ^{1}H NMR). Contact drying of açaí juice was conducted at the laboratory and pilot scale.

1.2 MATERIAL AND METHODS

1.2.1 Açaí juice

Frozen açaí juice bars (Açaí Diamante Negro, Pará, Brazil) were purchased from a local market in Amsterdam, the Netherlands. The total solids content of the juice was 14.1 ± 0.3 g·100 g^{-1}, total soluble solids of 5.1 ± 0.2 °Brix, and pH 4.9 ± 0.1. Açaí juice bars were kept frozen at -20 °C. For each test, a required amount of sample was taken from the frozen bar and thawed at room temperature until its temperature reached 10 °C.

1.2.2 Drying experiments

Three different drying methods were used to dry açaí juice: conductive thin-film drying at the laboratory- and pilot-scale, hot air drying, and freeze-drying. Except for the contact drying at the pilot-scale (vacuum drum drying), the samples were spread with a thickness of 0.5 mm over the drying supports, which was the minimum thickness possible to apply. The drying processes were stopped when the moisture content of dried açaí samples was below 5% (w.b.), following Brazilian legislation (MAPA, 2018). Then, the dried samples were collected and stored in the freezer for further analysis.

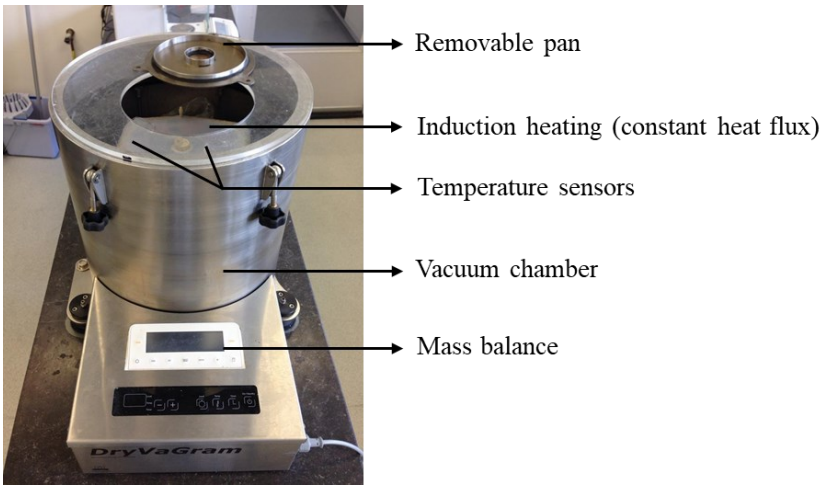
1.2.2.1 Conductive thin-film drying (CTFD) at lab-scale

A custom-built experimental conductive dryer (Figure 8) was used to mimic the (vacuum) drum drying at a laboratory scale, as described in Qiu et al. (2019b). This device consists of a drying chamber equipped with an analytical balance (Sartorius, Cubis MSE, Germany) and two temperature sensors (National Instruments^{TM}, K-Type, the Netherlands), allowing simultaneous online monitoring of the product mass and temperatures of the product and the pan during the drying process. The sample is uniformly spread on a steel plate (with a surface area of 0.0163 m²), which is heated with an induction coil. The induction heating was used to avoid any interaction between the heating system and the balance or pan. Even though this system does not precisely mimic the conditions during the drum drying due to the difference in heat supply (our system operates at constant heat flux and drum drying at a constant temperature), it allows a better understanding of the conductive drying behavior and the effect of drying on product quality.

Films of açaí juice were formed by carefully spreading 8.2 mL of the sample over the plate surface that was first coated with a vegetable oil-based spray (Dr. Oetker, Baking spray, Germany). The spray was applied to prevent juice browning due to contact with the steel plate (SANTOS et al., 2016) and adhesion of the dehydrated juice layer to the plate. Films were dried at two constant power inputs of 1300 and 1500 W (heat flux of 80 and 92 kW m^{-2}, respectively). The lower power stages of the equipment were not possible to apply because they did not provide a constant energy input. Three different chamber pressures (415, 700, and 1013 mbar) were used, for which the pure water boiling temperatures are 77, 90, and 100 °C, respectively.

Mass and temperatures were recorded every second. The obtained data were used to calculate the evaporation rate (g_{water} s^{-1} m^{-2}). The experiments were performed in triplicate.

Figure 8 - Laboratory-scale conductive thin-film dryer (DryVaGram).



Source: adapted from Wolbert (2019)

1.2.2.2 Hot air drying (HAD) and freeze-drying (FD)

Açaí juice was spread on polystyrene trays and dried by HAD and FD. These drying processes were considered referential cases for the study. The HAD was performed in a convective oven (Heraeus, Function Line T6, Germany) at 60 °C, with a relative humidity of 8%, for 2.5 h. The FD was conducted according to the following steps: first, the spread açaí juice was frozen at -20 °C and then transferred into the freeze-dryer (Martin Christ, Epsilon 2-10D LSCplus, Germany). The chamber pressure was reduced to 1 mbar, at a shelf temperature of -20 °C, for 2 h. After this, the shelf temperature was increased to -10 °C and maintained at this temperature for 4 h. Subsequently, the shelf temperature was increased to 0 °C, which was maintained for 4 h. Finally, the shelf temperature was increased and kept at 20 °C for 1.5 h, the pressure was decreased to 0.01 mbar and maintained at this level for 1.5 h. This last drying step lasted for 11 h, and the total drying time was 29 h. All drying experiments were conducted in duplicate.

1.2.2.3 Vacuum drum drying (VDD)

A pilot-scale vacuum drum dryer (ANDRITZ Gouda, Waddinxveen, the Netherlands), as shown in Figure 9, was also used for drying açaí juice.

Figure 9 - Pilot-scale conductive thin-film dryer.

The device consisted of two hollow steel chromium-plated drums with 0.20 m external diameter and 0.48 m length enclosed in a vacuum chamber. The chamber was maintained at 65 °C to avoid vapor condensation and was operated under two reduced pressures (140 and 200 mbar), resulting in pure water boiling temperatures of 52 and 60 °C, respectively. The drums were internally heated by steam with a pressure of 1.0 bar, which provided a hot wall temperature of 100 °C. The rotation speeds of drums were fixed at 0.5 and 1 rpm, giving residence times of 30 and 60 s, respectively. The sample was poured manually and fed evenly between the two drums into a hot feeding pool. The clearance between the two drums was fixed at 0.15 mm, allowing the formation of a thin layer adhered to the drum surface as the sample flowed through the gap of the drums. The dried product was scraped from the drum surface by doctor blades on the opposite side of the drum. The dried sample collected was mixed together for further analysis from the same drying conditions. Table 3 shows the drying conditions applied in the VDD.

Table 3 - Drying conditions used for dehydration of açaí juice in the vacuum drum dryer.

Parameters	Drying conditions		
	1	2	3
Chamber pressure (mbar)	140	140	200
Wall temperature (°C)	100	100	100
Residence time (s)	30	60	60
Chamber temperature (°C)	65		
Film thickness (μm)	≈ 75		

Note: Chamber temperature and film thickness were kept the same for all three drying conditions. Film thickness was estimated from the gap between the two drums.

1.2.3 Physical analysis of açaí powder

1.2.3.1 Moisture content and water activity

The moisture content of the powder samples was determined gravimetrically using a vacuum oven (BINDER, VD 53, Germany) at a pressure of 310 mbar and a temperature of 70 °C for 24 h. The water activity was measured using a water activity meter (Decagon Devices Inc., Aqualab TDL, USA) at 25 °C.

1.2.3.2 Lipid content

For lipid content determination, açaí samples were extracted with petroleum ether using an automatic Soxhlet extractor (Gerhardt, Soxtherm®, Germany). Results were expressed as g of lipid per 100 g of dry solids.

1.2.3.3 Color

The color parameters of samples were measured using a colorimeter (Konica Minolta, CR-400, Japan). The color values were expressed according to the CIELab scale as L^* ($L^* = 0$: black; $L^* = 100$: white), a^* ($-a^*$ = green; $+a^*$ = red) and b^* ($-b^*$ = blue; $+b^*$ = yellow). The total color differences (ΔE^*) between contact dried and reference samples were calculated using the Equation 1:

$$\Delta E^* = \sqrt{\left(L^*_{sample} - L^*_{ref}\right)^2 + \left(a^*_{sample} - a^*_{ref}\right)^2 + \left(b^*_{sample} - b^*_{ref}\right)^2} \qquad (1)$$

1.2.4 Chemical analysis of açaí powder

1.2.4.1 Total monomeric anthocyanin content

The total monomeric anthocyanin content in the juice and dried samples was quantified by the pH-differential method (GIUSTI and WROLSTAD, 2001). About 0.3 g of açaí powder was extracted with 10 mL of HCl/water/ethanol solution (1:29:70, v/v/v) using an ultrasonic bath (Elma, Elmasonic P, Germany) for 5 min at room temperature. The material was centrifuged at 3,400 rpm for 10 min, and the supernatant was filtered. The pellets were extracted twice more in the same fashion, and supernatants were combined. The extracts were diluted with two different buffer solutions (pH 1.0 and pH 4.5), and absorbances were recorded at 523 nm and 700 nm. Results were expressed as mg of cyanidin-3-rutinoside equivalent per 100 g of dry extract, using a molar absorptivity (ε) of 28,840 L mol^{-1} cm^{-1} and a molecular weight of 631 g mol^{-1}. Cyanidin-3-rutinoside is the major anthocyanin present in açaí (PACHECO-PALENCIA et al., 2009; TONON, BRABET, and HUBINGER, 2010).

1.2.4.2 Antioxidant activity

The antioxidant activity was determined using the DPPH free radical (DPPH•) scavenging method proposed by Brand-Willians, Cuvelier, and Berset (1995), with modifications. The extracts were prepared according to the method described by Larrauri, Rupérez, and Saura-Calixto (1997), with minor adaptations. In brief, 0.5 g of açaí powder was extracted with 40 mL methanol/water (50:50, v/v) at room temperature for 60 min. The material was centrifuged at 4,700 rpm for 15 min, and the supernatant was recovered. Then, the residue was re-extracted with 40 mL acetone/water (70:30, v/v) at room temperature for 60 min and centrifuged. Methanol and acetone extracts were pooled and diluted to a final volume of 100 mL with distilled water. Afterward, aliquots of 100 µL of açaí extract were added to 3.9 mL of 0.06 mmol L^{-1} DPPH methanolic solution. The absorbance was measured at 515 nm after 60 min of reaction at room temperature and in the dark. A standard Trolox curve (40-1000 µM) was prepared, and results were expressed as µmol of Trolox Equivalent (TE) per g of dry extract.

1.2.4.3 Lipid oxidation products

The proton nuclear magnetic resonance (^{1}H NMR) method, as proposed by Merkx et al. (2018), was applied to quantify hydroperoxides and aldehydes, which are primary and secondary lipid oxidation products, respectively. Firstly, the extraction of oil present in the açaí powder samples was performed as reported by Vargas-Ortiz et al. (2017). Subsequently, 150 µL of oil was collected, and 450 µL of 5:1 $CDCl_3$/DMSO-d_6 (Euriso-top, France) was added. After vortexing, the solution was transferred to a 5-mm NMR tube. ^{1}H NMR spectra were recorded on a Bruker Avance III 600 MHz NMR spectrometer (Bruker BioSpin, Switzerland) equipped with a 5-mm cryoprobe at a temperature of 295 K. A single pulse experiment was performed to record the peaks of the glycerol backbone at δ 4.4 ppm, which were used as internal calibration for the quantification of the lipid oxidation products. The peaks of hydroperoxides (δ 11.3-10.6 ppm) and aldehydes (δ 9.8-9.4 ppm) were obtained using two band-selective experiments. Data were processed using Bruker TopSpin 4.1 software. The concentrations of hydroperoxides and aldehydes were expressed as mmol per kg of oil.

1.2.5 Statistical analysis

All physicochemical analyses were performed at least in duplicate, and results are presented as mean ± standard deviation. One-way analysis of variance (ANOVA) and Tukey's test at a 95% confidence level ($\alpha = 0.05$) were applied to evaluate the experimental data. The statistical analyses were carried out using Statistica 10.0 (StatSoft, Tulsa, USA).

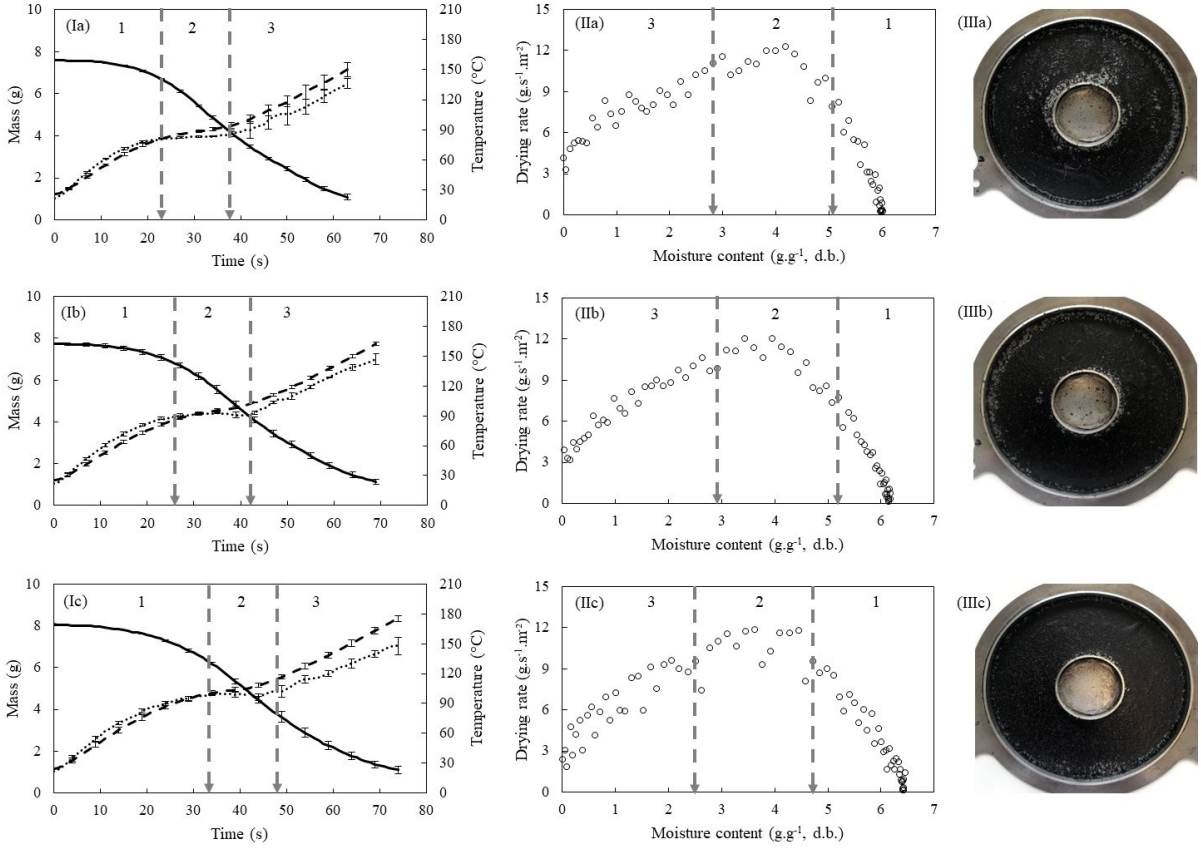
1.3 RESULTS AND DISCUSSION

1.3.1 Impact of varying pressure at lab-scale CTFD on drying behavior

Monitoring mass and temperature profiles during drum drying operation at a pilot or industrial scale is challenging. Thus, a laboratory-scale conductive thin-film dryer was used to provide relevant insights for drum drying and to study the impact of drying pressure on the drying kinetics of açaí juice and the quality of the final product. Figure 10 (I and II) and Figure 11 (I and II) show the mass and temperatures' evolutions throughout drying time and drying rate curves of açaí juice during the lab-scale CTFD process, applying three different drying

pressures and two power inputs. During drying, three distinct periods were identified: heating period (period 1), boiling or constant rate period (period 2), and conductive drying or declining rate period (period 3). Qiu et al. (2019b) also observed these three drying periods for the CTFD of maltodextrin and starch suspensions with thicknesses of 1 and 2 mm, using the same experimental system.

Figure 10 - (I) Mass (solid lines) and temperature profiles (dotted lines) of açaí juice, and temperature profile of the heated pan (dashed lines); (II) drying rates versus moisture content; and (III) images of the dried açaí juice using a power input of 1300 W at varying chamber pressure: (a) 415 mbar, (b) 700 mbar, and (c) 1013 mbar.

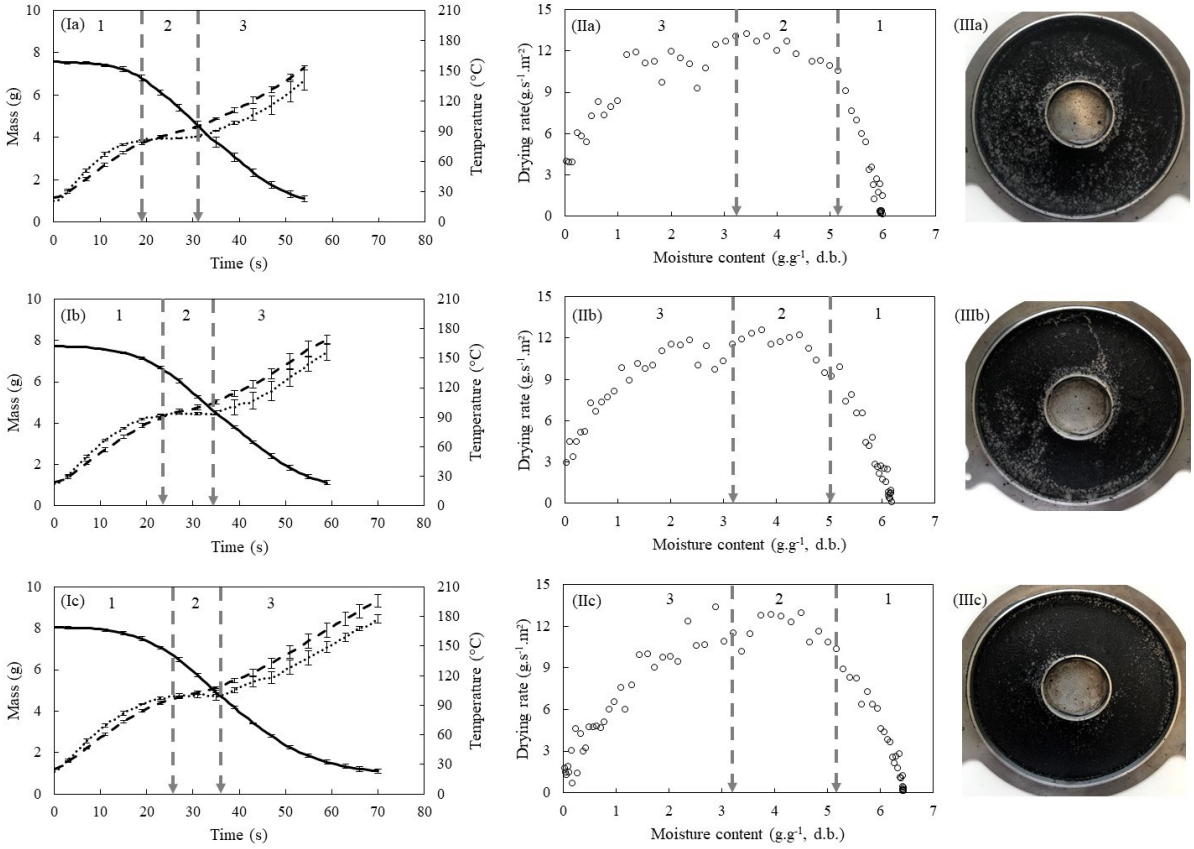


Note: The curves represent the average of three independent measurements. The error bars represent the standard deviation of the experimental data (n=3).

As expected, the decrease in the drying pressure reduced the boiling temperature of the samples. The corresponding boiling temperatures for 415, 700, and 1013 mbar were around 82, 91, and 100 °C, respectively. These temperatures were slightly above pure water boiling temperatures due to boiling point elevation. Decreasing the chamber pressure resulted as well in shorter drying times because the boiling (period 2) started earlier. The drying times were 63, 69, and 74 s for the chamber pressures of 415, 700, and 1013 mbar, respectively, applying a

power input of 1300 W. Whilst increasing the power input for 1500 W, the drying times were reduced for 54, 59, and 70 s in the corresponding drying pressures.

Figure 11 - (I) Mass (solid lines) and temperature profiles (dotted lines) of açaí juice, and temperature profile of the heated pan (dashed lines); (II) drying rates versus moisture content; and (III) images of the dried açaí juice using a power input of 1500 W at varying chamber pressure: (a) 415 mbar, (b) 700 mbar, and (c) 1013 mbar.



Note: The curves represent the average of three independent measurements. The error bars represent the standard deviation of the experimental data (n=3).

Drying at the different pressures showed a similar maximum constant drying rate of approximately 10.5 and 11.8 g_{water} s^{-1} m^{-2} for the energy inputs of 1300 and 1500 W, respectively. These values were at least 1.2 times lower than those reported by Qiu et al. (2019b) for the drying of maltodextrin and starch suspensions, which may be explained by the presence of more oil in açaí juice influencing the drying kinetics. Oil has a lower thermal conductivity than water and can be a physical barrier to moisture migration (CYPRIAN et al., 2016).

At the end of açaí juice drying, it was found that with decreasing chamber pressure, bubble formation was more vigorous (Figure 10III and Figure 11III). A higher growth rate and larger size of bubbles are related to the lower vapor density at lower pressure (SURTAEV,

SERDYUKOV, and MALAKHOV, 2020). Furthermore, the increase in power input also resulted in a more vigorous bubble formation, which can be explained by the higher heat supply for drying açaí juice, resulting in a less uniform drying process. Slightly higher product temperatures, at the end of drying, by applying the power input of 1500 W was observed as well. Thereby, from these results, the samples produced at 1300 W were chosen for further analyses.

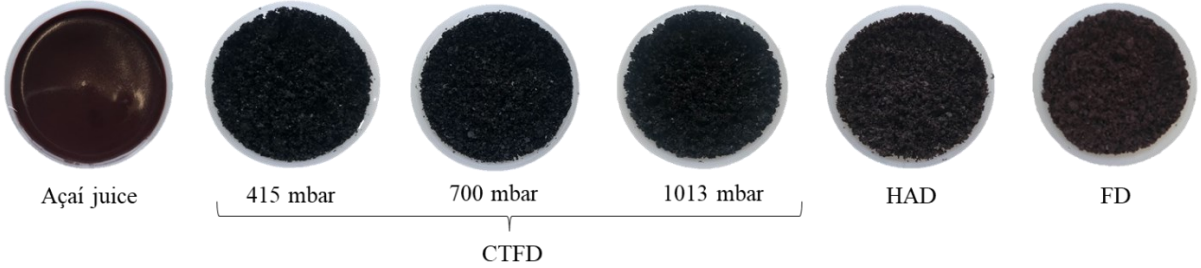
1.3.1.1 Moisture content and water activity

The moisture content and water activity of açaí powders produced by lab-scale CTFD at different drying pressures, HAD, and FD ranged from 0.015 to 0.026 g g^{-1} (d.b.) and from 0.163 to 0.500, respectively. Moisture content values were close to those reported by Pavan (2010) for açaí powder produced by refractance window drying, hot air drying, and freeze-drying. Water activity values below 0.5 prevent microbial growth and reduce rates of other deteriorative reactions, including nonenzymatic browning and enzymatic activity (TAOUKIS, LABUZA, and SAGUY, 1997).

1.3.1.2 Color

The açaí juice had a dark purple color, as shown in Figure 12, due to the high concentration of anthocyanins (BICHARA and ROGEZ, 2011). Anthocyanins are plant pigments responsible for blue, purple, red, and orange colors (SCHWARTZ et al., 2017). The different drying methods resulted in different product colors. From visual analysis (Figure 12), the contact-dried and hot air-dried samples showed a darker color than the freeze-dried açaí powders.

Figure 12 - Images of açaí juice and powders produced by lab-scale conductive thin-film drying (CTFD) at 415, 700, and 1013 mbar, hot air drying (HAD), and freeze-drying (FD).



The instrumental color parameters (L^*, a^*, and b^*) and the total color differences (ΔE^*) of the açaí powders, considering HAD and FD as references, are shown in Table 4. The chamber pressure during lab-scale CTFD did not significantly influence dried samples' luminosity (L^* value) and redness (a^* value). Nevertheless, decreasing the drying pressure decreased the b^* value, indicating a slight tendency to blue. Comparing each drying method, the L^* values of açaí juice dried by CTFD were similar to that from HAD and significantly lower than the FD samples, suggesting a darker color. Moreover, higher ΔE^* values were observed between CTFD and FD powders. These results reassert the visual analysis. The fewer color changes between CTFD and HAD samples can be attributed to the drying temperatures during CTFD and extended exposure to oxygen during HAD, which possibly accelerated nonenzymatic browning (Maillard reaction) and anthocyanin degradation, respectively.

Table 4 - Color parameters (L^*, a^*, and b^*) and total color differences (ΔE^*) of açaí powders.

Drying process	***L****	***a****	***b****	***ΔE**** (HAD ref)	***ΔE**** (FD ref)
CTFD (415 mbar)	30.15 ± 0.23^{b}	0.26 ± 0.01^{c}	0.16 ± 0.05^{d}	1.26	3.23
CTFD (700 mbar)	30.38 ± 0.16^{b}	0.30 ± 0.04^{c}	0.22 ± 0.03^{cd}	1.16	3.10
CTFD (1013 mbar)	30.19 ± 0.17^{b}	0.28 ± 0.01^{c}	0.27 ± 0.07^{c}	1.18	3.16
HAD	30.47 ± 0.14^{b}	1.33 ± 0.07^{b}	0.74 ± 0.02^{b}	-	-
FD	31.33 ± 0.34^{a}	3.09 ± 0.08^{a}	1.19 ± 0.04^{a}	-	-

Note: Drying processes: conductive thin-film drying (CTFD), hot air drying (HAD), and freeze-drying (FD). HAD ref and FD ref are hot air-dried and freeze-dried reference samples, respectively. Color parameters are expressed as average ± standard deviation (n=6). For each drying process, açaí powder was prepared in two independent experiments, and samples were evaluated in triplicate. Means followed by different letters in the same column represent significant differences ($p \leq 0.05$), according to Tukey's test.

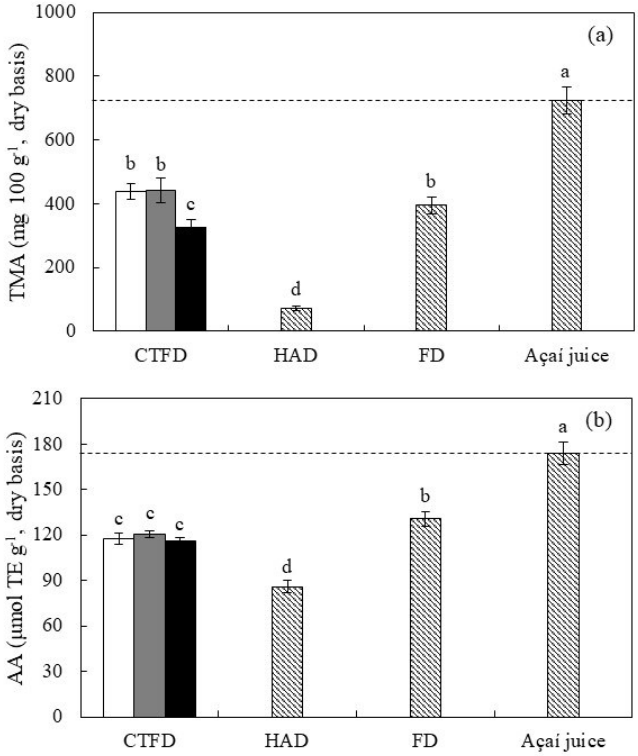
1.3.1.3 Total monomeric anthocyanin content and antioxidant activity

The anthocyanin content and antioxidant activity measured by the DPPH assay of açaí juice and CTFD, HAD, and FD powders are presented in Figure 13. Açaí powders obtained by CTFD under reduced pressures showed anthocyanin retention (61%) comparable to that of freeze-dried samples (54%) and significantly greater than that of hot air-dried powders (10%). Anthocyanins are sensitive to high temperature, oxygen, and light, among other factors (TONON, BRABET, and HUBINGER, 2010). Therefore, the high anthocyanin retention during CTFD at low pressures may be explained by the short drying times at moderate boiling temperatures and low oxygen concentrations. This result is supported by Zhou et al. (2017),

who reported the positive effect of low temperature and low oxygen atmosphere on anthocyanin retention during mulberry fruit drying.

Conversely, the antioxidant activity of CTFD samples was significantly lower than that of freeze-dried samples, with no significant difference among drying pressures applied in CTFD. Even though anthocyanins account for approximately 90% of the antioxidant activity of açaí (PACHECO-PALENCIA et al., 2009), two hypotheses can justify the antioxidant activity behavior of the samples (TONON, BRABET, and HUBINGER, 2010): (1) the freeze-drying may have increased the bioavailability of non-identified compounds, which contributed to the antioxidant activity; and (2) the increase in boiling temperature in CTFD, due to the increase in the chamber pressure, may have provided the formation of Maillard reaction products with antioxidant activity.

Figure 13 - (a) Total monomeric anthocyanin content (TMA) and (b) antioxidant activity (AA) in the açaí juice and powders produced by lab-scale conductive thin-film drying (CTFD) at 415 mbar (white column), 700 mbar (grey column), and 1013 mbar (black column), hot air drying (HAD), and freeze-drying (FD).

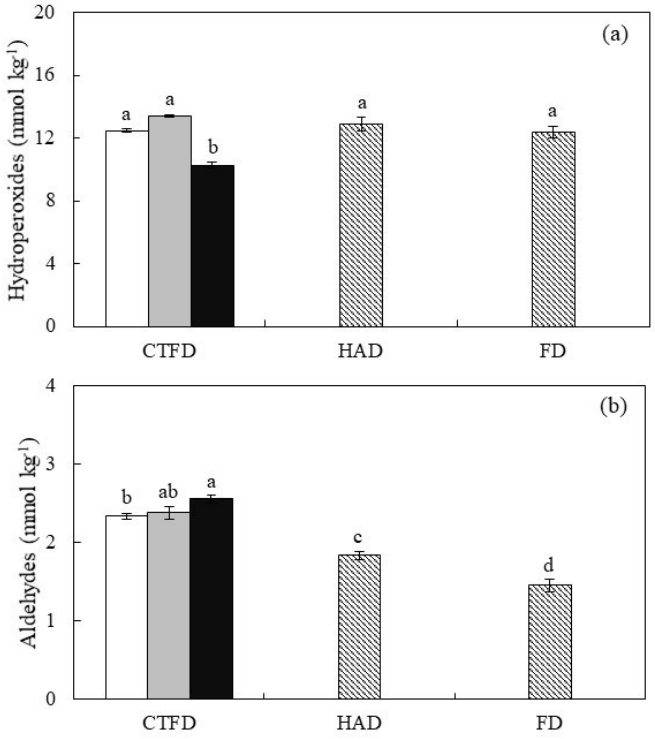


Note: For each drying process, açaí powder was prepared in independent duplicate, and the measurements were carried out in triplicate. The error bars represent the standard deviation of the experimental data (n=6). The different letters represent significant differences ($p \leq 0.05$). The dashed lines were added to guide the eyes.

1.3.1.4 Lipid oxidation products

Açaí has a high unsaturated fatty content, being susceptible to lipid oxidation during processing and storage (SCHAUSS et al., 2006). In the lipid oxidation process, primary oxidation products are formed (peroxides, especially hydroperoxides), which further decompose to secondary oxidation products (e.g., aldehydes, ketones, alcohols, and acids) that are responsible for negative quality changes in foods, such as deterioration of flavor and reduction in nutritional value (BARRIUSO, ASTIASARÁN, and ANSORENA, 2013). After the drying processes, the concentration of hydroperoxides and aldehydes in the lipid fraction (oil) of açaí powders was measured (Figure 14).

Figure 14 - The concentration of (a) hydroperoxides and (b) aldehydes in açaí powders produced from lab-scale conductive thin-film drying (CTFD) at 415 mbar (white column), 700 mbar (grey column), and 1013 mbar (black column), hot air drying (HAD), and freeze-drying (FD).



Note: For each drying process, açaí powder was prepared in two independent experiments, and the samples were analyzed in duplicate. The error bars represent the standard deviation of the experimental data (n=4). The different letters represent significant differences ($p \leq 0.05$).

The CTFD at atmospheric pressure resulted in the lowest formation of hydroperoxides, which can be due to the fastest decomposition of these compounds to secondary lipid oxidation

products, as illustrated by the highest aldehydes concentration (Figure 14b), which was caused by the highest boiling temperature during drying (HOPPENREIJS et al., 2021). The formation of aldehydes during CTFD decreased as drying pressure was reduced, which can be associated with the reduction in the boiling temperature, oxygen concentration, and drying time. Although HAD showed a longer air product exposure time than CTFD, a lower degree of aldehydes formation was observed for hot air-dried samples. This result is probably related to lower temperatures in HAD compared to CTFD, suggesting a dominant effect of temperature during the drying of açaí juice for accelerating lipid oxidation.

According to Codex Alimentarius (1999), the recommended maximum value of hydroperoxides for edible oils is 10 milli-equivalents O_2 kg^{-1}. However, for all açaí powders in this study, irrespective of the drying method used, the hydroperoxides concentrations exceeded this recommended value and varied from 10.3 to 13.4 mmol hydroperoxides kg^{-1} (or 20.6 to 26.8 milli-equivalents O_2 kg^{-1}). Lucas, Zambiazi, and Costa (2018) found lower hydroperoxides concentration in freeze-dried açaí powder (6.34 milli-equivalents O_2 kg^{-1}). Different oil extraction and determining lipid oxidation methods contribute to the differences in the concentration of primary lipid oxidation products. Extraction with petroleum ether at a temperature of 45 °C and nuclear magnetic resonance technique were used in the present study, whereas Lucas et al. (2018) used extraction with chloroform, methanol, and water at room temperature and iodometry titration assay.

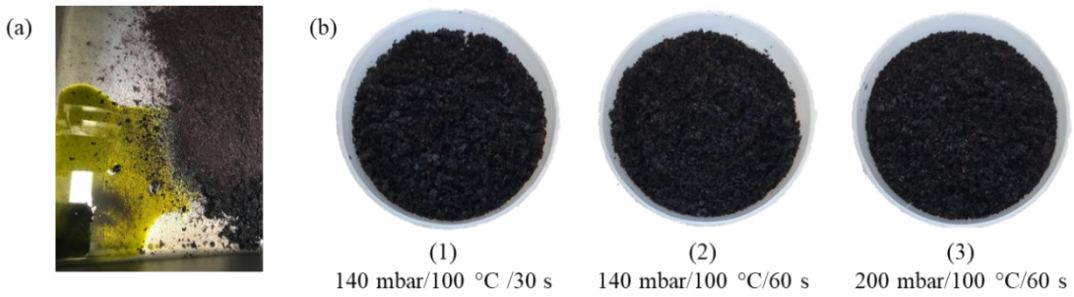
1.3.2 Performance of the VDD for production of açaí powder

A partial limitation of the experimental system (CTFD at lab-scale) is its operation at constant power, while (vacuum) drum drying operates at a constant wall temperature. In this way, the drying of açaí juice was also performed in VDD at the pilot scale. The effect of pressure in the vacuum chamber and residence time on the quality of açaí powder were assessed.

The original açaí juice (86%, wet basis) could be directly fed into the dryer. During VDD, most of the moisture in the sample was evaporated during boiling in the feeding pool between the drums (period 2), and a conductive drying (period 3) occurred on the drums after the açaí juice was cast as a thin film (QIU et al., 2019a). The spreading, drying, and collection of the dried açaí as small flakes were observed for all applied drying conditions. The drying process took between 30 and 60 s. However, oil separation was visually observed (Figure 15a), except for condition 1 (140 mbar/100 °C/30 s), which was the mildest drying condition. This

visual result was confirmed by quantitative analysis of lipid content, as discussed in the following section.

Figure 15 - (a) Representative image of oil release from the matrix during vacuum drum drying (VDD) of açaí juice; and (b) Images of vacuum drum-dried açaí powders at the different drying conditions.



1.3.3 Physicochemical properties of vacuum drum-dried açaí powder

The moisture content, water activity, lipid content, and color parameters of the vacuum drum-dried açaí powders at the different drying conditions are presented in Table 5. The VDD of açaí juice resulted in powders with moisture content and water activity close to those obtained by other drying methods that require a much longer drying time (OLIVEIRA et al., 2020; PAVAN, 2010).

Table 5 - Moisture content, water activity, lipid content, and color of vacuum drum-dried açaí powders.

Parameters	**Drying conditions**		
	(1) 140 mbar/100 °C/30 s	**(2) 140 mbar/100 °C/60 s**	**(3) 200 mbar/100 °C/60 s**
Moisture content (g g^{-1} d.b.)	0.015 ± 0.000[a]	0.010 ± 0.001[b]	0.014 ± 0.001[a]
Water activity	0.232 ± 0.002[a]	0.143 ± 0.004[c]	0.216 ± 0.003[b]
Lipid content (g 100 g^{-1} d.b.)	45.1 ± 0.6[a]	39.6 ± 0.2[b]	35.2 ± 1.2[c]
Color			
*L**	30.38 ± 0.10[a]	30.32 ± 0.15[a]	30.59 ± 0.09[a]
*a**	1.07 ± 0.06[b]	0.94 ± 0.02[c]	1.20 ± 0.06[a]
*b**	0.38 ± 0.02[a]	0.38 ± 0.03[a]	0.39 ± 0.05[a]

Note: Parameters are expressed as average ± standard deviation (n=3). For each drying condition, the measurements were performed in triplicate. Means followed by different letters in the same row represent significant differences ($p \leq 0.05$), according to Tukey's test.

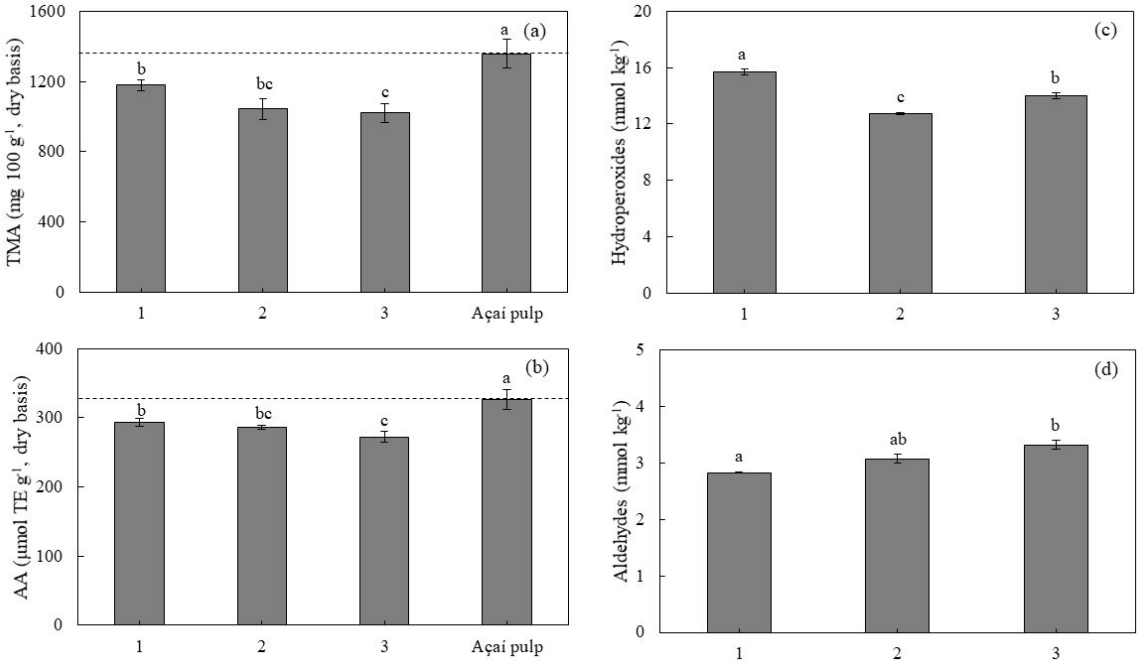
Lipids are the major compounds of açaí juice (46.7 ± 0.3 g 100 g^{-1} d.b.). During the VDD, the increase in the residence time and boiling temperature (conditions 2 and 3) resulted in a significant decrease in the lipid content of dried samples because of undesired oil release from the matrix. The oil loss during drying significantly reduces the product's nutritional value, besides operational problems, such as frequent dryer downtime for cleaning.

In contrast to what was observed in the lab-scale conductive drying, drying conditions used in VDD affected the *a** value (greenness/redness), probably due to the loss of oil during drying since the açaí oil has a dark green color (SILVA and ROGEZ, 2013). Figure 15b shows the açaí powders produced by VDD.

The anthocyanin content and antioxidant activity of samples produced by VDD (Figure 16a and b) were calculated by excluding the lipid content of samples to have the same dry matter content at all drying conditions. The VDD decreased the anthocyanin content and antioxidant activity of powders, by an average of 13-25% and 10-16%, respectively, from the anthocyanin content and antioxidant activity of açaí juice. Similar results were reported by Tonon et al. (2010) for powders produced by spray drying of the filtered açaí juice added with carrier agents. As observed in the lab-scale CTFD, increasing boiling temperature under vacuum conditions did not influence the anthocyanin content and antioxidant activity in açaí powders. However, the product's anthocyanin content and antioxidant activity were negatively affected by the simultaneous increase in boiling temperature and residence time (condition 3).

Concerning oxidative stability of açaí oil in the vacuum drum-dried samples (Figure 16c and d), the concentration of primary lipid oxidation products ranged from 12.7 to 15.7 mmol hydroperoxides kg^{-1} (or 25.5 to 31.4 milli-equivalents O_2 kg^{-1}). These high values can be again related to the oil extraction method and the technique for determining lipid oxidation. Although condition 1 showed the highest hydroperoxides concentration, the lowest aldehydes concentration was observed, indicating few changes in flavor and odor of powders. This result agrees with that from the lab-scale drying, i.e., a low boiling temperature, low oxygen concentration, and short drying time during contact drying resulted in a slow oxidative breakdown of primary products to form secondary products.

Figure 16 - (a) Total monomeric anthocyanin content (TMA), (b) antioxidant activity (AA), and concentration of (c) hydroperoxides and (d) aldehydes in vacuum drum-dried açaí powders at different drying conditions: (1) 140 mbar/100 °C/30 s; (2) 140 mbar/100 °C/60 s; and (3) 200 mbar/100 °C/60 s.



Note: For each drying condition, the measurements were performed in duplicate The error bars represent the standard deviation of the experimental data (n=2). The different letters represent significant differences ($p \leq 0.05$). The dashed lines were added to guide the eyes.

1.4 CONCLUSION

The conductive thin-film drying process at the laboratory and pilot-scale allowed producing açaí powders with low moisture contents and water activities with short drying times. High retention of anthocyanins and antioxidant activity was achieved by drying at low pressures due to low boiling temperatures, low-oxygen environments, and short process times. Moreover, decreasing the drying pressure resulted in slow decomposition of primary lipid oxidation products to form secondary lipid oxidation products, suggesting smaller undesirable sensorial and nutritional effects in açaí powders. However, contact drying resulted in less oxidative stability of açaí oil than hot air drying and freeze-drying. In addition, oil release is a risk during pilot-scale drying but could be mitigated by selecting mild low-pressure drying conditions. Therefore, vacuum conductive thin film drying is a good alternative for producing açaí powder, although further process optimization is still required. The assessment of functional (such as dispersion time and solubility) and sensorial properties is also recommended to obtain detailed information on the rehydration behavior and consumers' acceptability of açaí powders.

CHAPTER 2. AÇAÍ POWDER PRODUCED BY CAST-TAPE DRYING: IMPACT OF HYDROCOLLOIDS ADDITION ON ITS PHYSICOCHEMICAL PROPERTIES

In this chapter, açaí juice was dried by cast-tape drying (CTD) to produce powdered samples. The effect of the hydrocolloids' addition, like pectin, maltodextrin, and gum Arabic on the drying performance and final product characteristics were evaluated. The hydrocolloids were added to açaí juice at a concentration of 3% (w/w juice). Freeze-dried pure açaí juice was produced to establish a reference for the study. The rheological properties of the suspensions were investigated by dynamic shear measurements to obtain insights into the suitability of the suspensions for the CTD process. The suspensions showed different elastic properties, being the açaí-pectin suspension showed a more solid-like behavior. All suspensions were evenly spread on the drying support of the cast-tape dryer. The process times in CTD depended on the sample and ranged from 16 min to 32 min, shorter than freeze-drying (24 h). The addition of pectin to açaí juice hindered the oil separation during the CTD process and contributed to removing the dried material from the drying support. Moreover, the color, anthocyanin content, and antioxidant activity of the açaí-pectin powder were similar to those of freeze-dried açaí powder and significatively higher than those of the other powders from CTD. Nevertheless, the CTD process led to lower oxidative stability of açaí oil in the dried samples. This study was carried out at the Laboratory of Physical Properties of Food (PROFI) at the Federal University of Santa Catarina (Brazil) and the Laboratory of Food Process Engineering (FPE) at Wageningen University & Research (the Netherlands).

This chapter will be submitted as: SIMÃO, Raquel da Silva; MORAES, Jaqueline Oliveira de; ZHANG, Lu; SCHRÖDER, Anja; CARCIOFI, Bruno Augusto Mattar; SCHUTYSER, Maarten A. I.; LAURINDO, João Borges. **Açaí powder produced by cast-tape drying: Impact of hydrocolloids addition on its physicochemical properties**.

2.1 INTRODUCTION

Açaí (*Euterpe oleracea* Mart.) is a native fruit to the Amazon region with elevated production and socio-economic importance in the northern states of Brazil. This fruit has attracted the attention of industries and scientists due to its high nutritional value, being considered a superfruit. Açaí is rich in fibers, proteins, and unsaturated fatty acids. The oleic acid and linoleic acid represent approximately 56% and 13% of the lipid fraction of the açaí fruit, respectively. It is also an important source of anthocyanins, with high antioxidant activity (SCHAUSS et al., 2006; YAMAGUCHI et al., 2015). Nevertheless, açaí fruit and its fresh juice are highly perishable, showing a short shelf life, even under refrigeration. Alternatively, açaí juice can be dried into açaí powder to increase its shelf-life, add value, diversify consumption options, reduce losses, and facilitate commercialization. However, anthocyanins and lipid fraction stability depend on the drying processing parameters. Thus, the drying techniques and conditions must be carefully selected to retain the fruit's nutritional quality as much as possible.

Freeze-drying (FD) and spray drying (SD) are the most applied techniques to convert açaí juice into powder (LUCAS, ZAMBIAZI, and COSTA, 2018; OLIVEIRA et al., 2021; TONON et al., 2009c). Even though FD results in high-quality dried products, this technology is very expensive because of the long operation time and high energy consumption and may therefore not be economically feasible to apply on an industrial scale (RATTI, 2008). On the other hand, SD has required a previous filtration of the açaí juice to eliminate solids in suspension, therefore allowing the product's passage through the nozzle atomizer (TONON, BRABET, and HUBINGER, 2010). However, filtrating the açaí juice results in significant nutritional losses for the product as this operation reduces insoluble fiber and fat content.

The production of fruit powders with good nutritional properties by energy-efficient drying techniques has been challenging. Cast-tape drying (CTD), also known as refractance window drying – a particular case of CTD –, is an innovative drying method that converts whole fruit or vegetable purees into films, flakes, or powders. During the process, energy for water evaporation is supplied by a heating medium (hot water or steam), at atmospheric pressure, and transferred mainly by conduction to a thin layer of the material spread onto flexible support (transparent or not to infrared radiation) (SIMÃO et al., 2020). CTD is characterized by short process times (a few minutes) and moderate temperatures of the product (below 70 °C) during drying, allowing high retention of compounds that are heat-sensitive and prone to oxidation (vitamins and bioactive compounds) (BAEGHBALI, NIAKOUSARI, and

FARAHNAKY, 2016; SIMÃO et al., 2021). Moreover, this process requires lower energy consumption than SD and FD (BAEGHBALI, NIAKOUSARI, and FARAHNAKY, 2016). However, Pavan (2010) reported lower retention of anthocyanins in açaí powder produced by refractance window drying (84.8%) than that obtained by freeze-drying (95.1%). Furthermore, lipids in dried açaí samples were liquid form at room temperature, which have higher mobility to participate in degradation reactions, such as lipid oxidation. Hence, adding food hydrocolloids (polysaccharides and/or proteins) before drying can be a good option to improve the açaí powder's quality produced by CTD. Another advantage of this drying process compared to SD is the possibility of adding a smaller amount of biopolymers.

Hydrocolloids contain hydrophilic and/or hydrophobic groups that create a polymer network and may promote the protection of sensitive compounds (such as anthocyanins and unsaturated fatty acids) from oxygen, moisture, temperature, and/or light during processing and storage (TURCHIULI et al., 2005). Tonon et al. (2009c) reported the protective effect of hydrocolloids on the retention of polyphenolics and antioxidant activity of açaí powders produced by SD. Maltodextrin and gum Arabic are commonly used hydrocolloids to prevent the degradation of bioactive compounds during the drying of fruit juices or purees, mainly due to their high solubility, low viscosity, and good coating properties (TELIS and MARTÍNEZ-NAVARRETE, 2012; TONON et al., 2009c). Pectin has also shown great potential for the effective protection of bioactive compounds because of its gelling, stabilizing, binding, and coating properties (REHMAN et al., 2019).

The objective of this study was to investigate the influence of hydrocolloids addition on the quality of açaí powders obtained by CTD and to compare them to freeze-dried pure açaí juice (produced without any hydrocolloids). The hypothesis was that hydrocolloids addition would result in a cast-tape dried açaí powder with quality comparable to freeze-dried açaí powder. Pectin, maltodextrin, and gum Arabic were used as hydrocolloids and added to açaí açaí, which was subsequently dehydrated by CTD. The product quality was based on anthocyanin content, antioxidant activity, and lipid fraction.

2.2 MATERIAL AND METHODS

2.2.1 Materials

Frozen açaí juice, which was obtained by macerating the açaí fruit and adding water and afterward frozen, was purchased from Norfrutas Eireli (Belém, Brazil) at a local market in Florianópolis, Brazil. The hydrocolloids used in this study were: GENU® high methoxyl pectin type 106 BP (CPKelco, Limeira, Brazil) with a DM of 68%, maltodextrin MOR-REX® 1910 (Ingredion, São Paulo, Brazil) with DE10, and gum Arabic (Wifa Ingredientes, Palhoça, Brazil). Nile Red, potassium bromide (KBr), 2,2-diphenyl-1-picrylhydrazyl (DPPH), and (±)-6-Hydroxy-2,5,7,8-tetramethylchromane-2-carboxylic acid (Trolox) were purchased from Sigma-Aldrich (St. Louis, USA). Chloroform-d ($CDCl_3$) and dimethyl sulfoxide-d6 (DMSO-d_6) were purchased from Euriso-top, (Saint-Aubin, France). All the other used chemical reagents were of analytical grade.

2.2.2 Preparation of açaí suspensions

The açaí juice was kept in a freezer at -18 °C, and the required amount for each experiment was thawed at room temperature until the juice temperature reached 10 °C. The physicochemical composition of açaí juice is presented in Table 6. Proximate composition was carried out according to AOAC methods (AOAC, 2005) for moisture content (method no. 930.15), proteins (method no. 977.02), lipids (method no. 930.09), crude fiber (method no. 978.10), and ash (method no. 930.05). Carbohydrates were calculated by difference. The soluble solids concentration was determined by a refractometer (Atago, PAL-BX/RI, Japan), and pH was measured using a pH meter (Testo, testo 205, Germany).

Four suspensions were prepared: (i) Açaí juice without the addition of hydrocolloids (pure açaí juice); (ii) Açaí juice + pectin; (iii) Açaí juice + maltodextrin; and (iv) Açaí juice + gum Arabic. Hydrocolloids were used in a concentration of 3% (3 g of hydrocolloid per 100 g of açaí juice), which was selected based on preliminary tests. The homogenization was performed using a household hand blender (Oster, FPSTHB2610R-057, Brazil) for 2 min at the minimum speed.

Table 6 - Composition of açaí juice (*Euterpe oleracea* Martius).

Component	Mean ± SD
Moisture (g 100 g^{-1} w.b.)	84.97 ± 0.25
Dry matter (g 100 g^{-1} w.b.)	15.03 ± 0.25
Proteins (g 100 g^{-1} d.b.)	10.49 ± 0.20
Lipids (g 100 g^{-1} d.b.)	52.23 ± 0.83
Total fiber (g 100 g^{-1} d.b.)	7.02 ± 1.50
Carbohydrates (g 100 g^{-1} d.b.)	25.55 ± 0.27
Ash (g 100 g^{-1} d.b.)	4.97 ± 0.35
pH	5.01 ± 0.12
Total soluble solids (˚Brix)	4.36 ± 0.40

2.2.3 Rheological properties

Dynamic oscillatory measurements were performed using a rotational rheometer (Thermo Scientific™, HAAKE MARS, Germany) equipped with a parallel plate geometry with a 20 mm diameter and 2.5 mm gap. The açaí suspensions were evaluated at a temperature of 11.2 ± 0.1 °C. Oscillatory stress sweeps between 0.1 and 25 Pa were carried out at a constant frequency of 0.1 Hz. The elastic or storage modulus (G′) and viscous or loss modulus (G″) were recorded as a function of the shear stress. The critical shear stress was determined at the crossover point (G′ = G″). The measurements were performed in triplicate.

2.2.4 Drying process

2.2.4.1 Cast-tape drying (CTD)

CTD experiments were performed in a pilot-scale apparatus (Figure 17) with an effective area of 0.54 m^2 (2.00 m long x 0.27 m wide). The hot water temperature was adjusted to 98 °C to produce steam, which provided latent heat for water evaporation from the açaí suspensions during the drying process. The suspensions were uniformly spread on the continuous support of 0.25 mm-thick fiberglass film coated with Teflon® (Indaco, Lençol Armalon® Standard, Brazil), which is a hydrophobic and opaque surface and has low surface energy. The spreading of suspensions was carried out using a spreader (doctor blade) with an adjusted gap between 1 and 2 mm, and conveyor belt velocity varying from 6.25 to 12.5 cm min^{-1} in order to the thickness of spread suspensions was approximately 1.5 mm (Table 7). Thicknesses of spread samples were measured with a caliper (Mitutoyo Co., Japan)

immediately after the passage of suspensions through the spreader. The spreader gap and conveyor belt speed depended on the rheology of the suspensions, and they were selected from previous experiments.

Figure 17 - Device of the continuous cast-tape drying.

The cooling zone of the equipment was switched off, and the dried açaí suspensions were scraped off from the support at the end of the heating zone since the samples adsorbed moisture from the ambient during passage over the cooling zone. The evaporated water during drying was removed by natural convection. The ambient relative humidity (50–73%) and temperature (25–29 °C) were continuously measured. Drying was conducted until the moisture content of the product was less than 5% (w.b.), following the Brazilian legislation (MAPA, 2018).

Table 7 - Gap of the doctor blade, conveyor belt velocity, and spreading thickness for each açaí suspension dehydrated by CTD.

Suspension samples	**Spreader gap (mm)**	**Belt speed (cm min^{-1})**	**Thickness of spread suspension (mm)**
Pure açaí	1.0	12.5	1.41 ± 0.13
Açaí-pectin	2.0	6.25	1.40 ± 0.12
Açaí-maltodextrin	1.5	8.00	1.55 ± 0.11
Açaí-gum Arabic	1.5	8.00	1.50 ± 0.10

2.2.4.2 Freeze-drying (FD)

FD experiments were performed to establish a reference for the study. The pure açaí juice was freeze-dried, and the powder characteristics were compared to those produced by CTD. The FD process was conducted in a lab-scale freeze-dryer (Liotop, L101, Brazil). The sample was dried to a moisture content below 5% (w.b.), in a chamber with an internal pressure of 0.06 mbar, and an ice condenser temperature of -54 °C.

2.2.5 Characterization after CTD process

2.2.5.1 Oil release on the flexible support

Micrographs of the fiberglass support coated with Teflon® after drying açaí suspensions were captured using an optical stereoscope (OptiCam, OPT 10000, Brazil). The images were analyzed by an image processing software (TSview, Tucsen, China).

2.2.5.2 Oil distribution in the dehydrated açaí samples

Dried açaí suspensions were stained with Nile Red dye and evaluated by confocal laser scanning microscopy (CLSM) to visualize oil droplets distribution in the samples after drying. In brief, a stock solution of Nile Red (0.8 mg mL^{-1}) in acetone was prepared and then diluted with distilled water in a 1:50 (v/v) ratio. Samples were cut into squares (5 mm x 5 mm) and stained with 200 μL of the Nile Red solution for 2 min. A confocal laser scanning microscope (Leica Microsystems, Leica TCS SP5, Germany) was used, and the Nile Red fluorescence was detected using a He-Ne laser with an excitation wavelength of 543 nm and an emission filter at 605 to 640 nm (red). The micrographs of dried samples were obtained by converting a sequence of optical sections (z-stack) into a maximum intensity projection image (two-dimensional image) using Leica LAS AF Lite software.

2.2.6 Characterization of açaí powder

Dried açaí samples in the form of films or flakes, after CTD and freeze-drying processes, were ground using a knife mill (TECNAL, TE 631/2, Brazil) and sieved using a 20-mesh sieve to obtain açaí powders with particle sizes of 850 μm or less.

2.2.6.1 Moisture content and water activity

Moisture content was determined by the gravimetric procedure using a vacuum oven (TECNAL, TE-395, Brazil) at 70 °C (AOAC, 2005). Water activity was determined with a water activity meter (Decagon Devices Inc., Aqualab 4TE, USA). Analyses were performed in triplicate.

2.2.6.2 Lipid content

The lipid content of the açaí powder samples was determined using petroleum ether as the solvent in a Soxhlet extractor, according to AOAC official method no. 930.09 (AOAC, 2005). The measurements were conducted in triplicate. The results were expressed as g of lipid per 100 g of dry solids, and the mass of hydrocolloids was disregarded.

2.2.6.3 Color

A computer vision system was used to determine the color parameters of samples, according to Cárdenas-Pérez et al. (2017), with minor adaptations. The measurements were replicated three times. The color values were expressed as L^* (L^* = 0: black; L^* = 100: white), a^* (-a^* = green; +a^* = red) and b^* (-b^* = blue; +b^* = yellow). The total color difference (ΔE^*) was calculated according to Equation 2, using the FD sample as a reference (L_0^*, a_0^*, and b_0^*).

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

2.2.6.4 Fourier transform infrared (FTIR) spectroscopy

FTIR spectra of the hydrocolloids and açaí powders produced by CTD were obtained using an FTIR spectrometer (Agilent Technologies, Cary 660, USA). Samples (2 mg) and KBr pellets (50 mg) were completely mixed and pressed and then analyzed at room temperature using a scanning range from 400 to 4000 cm^{-1} and resolution of ±2 cm^{-1} at 21 scans min^{-1}.

2.2.6.5 Total monomeric anthocyanin content

The total monomeric anthocyanin content was determined by the pH-differential method as described by Giusti and Wrolstad (2001). Anthocyanins were extracted three times from 0.3 g of açaí powder using 10 mL of an HCl/water/ethanol solution (1:29:70, v/v/v). At each time, the mixture was sonicated for 5 min at room temperature and then centrifuged for 10 min at 3,400 rpm. Supernatants were collected and combined. The extracts were separately diluted with 0.025 mol L^{-1} potassium chloride buffer (pH 1.0) and 0.4 mol L^{-1} sodium acetate buffer (pH 4.5). Total anthocyanin content was expressed in terms of mg of cyanidin-3-rutinoside equivalent per 100 g of dry extract, excluding the mass of the hydrocolloids. Absorbances were measured at 523 and 700 nm, using the molecular weight and molar absorptivity of cyanidin-3-rutinoside of 631 g mol^{-1} and 28,840 L mol^{-1} cm^{-1}, respectively. Cyanidin-3-rutinoside is the predominant anthocyanin in açaí (GORDON et al., 2012; PACHECO-PALENCIA, DUNCAN, and TALCOTT, 2009; TONON, BRABET, and HUBINGER, 2010). Three independent replicates were evaluated.

2.2.6.6 Antioxidant activity

For determination of antioxidant activity, the extracts were obtained according to the method described by Larrauri, Rupérez, and Saura-Calixto (1997), with some modifications. Briefly, 0.5 g of açaí powder was extracted sequentially with 40 mL methanol/water (50:50, v/v) and 40 mL acetone/water (70:30, v/v) at room temperature for a total time of 120 min. After each extraction, the material was centrifuged at 4,700 rpm for 15 min and the supernatant was recovered. Methanol and acetone extracts were combined and made up to 100 mL with distilled water. The antioxidant activity was measured by DPPH radical scavenging activity assay, according to Brand-Willians, Cuvelier, and Berset (1995), with

adaptations. The reaction was carried out with 100 μL of açaí extract and 3.9 mL of 0.06 mmol L^{-1} DPPH methanolic solution for 60 min in the dark and at room temperature. Afterward, the absorbance was measured at 515 nm. The same analysis was performed on the Trolox methanolic solution in six dilutions ranging in concentration from 40 to 1000 μM, allowing the construction of a standard Trolox curve. The results were expressed as μmol of Trolox Equivalent (TE) per g of dried extract (disregarding the mass of hydrocolloid). The analysis was carried out in triplicate.

2.2.6.7 Lipid oxidation products

Primary and secondary lipid oxidation products (hydroperoxides and aldehydes, respectively) were quantified by proton nuclear magnetic resonance (^{1}H NMR) described by Merkx et al. (2018). The oil from the açaí powder samples was extracted for 60 min in a hot water bath at 45 °C with orbital agitation, using petroleum ether as the solvent in a 1:3 (w/w) sample:solvent ratio. The extracts were filtered under a vacuum, and the solvent evaporated using a rotary evaporator (37 °C, 200 mbar) (VARGAS-ORTIZ et al., 2017). Then, 150 μL of oil was collected and 450 μL 5:1 $CDCl_3$/DMSO-d_6 was added. The mixture was transferred to a 5-mm NMR tube and analyzed in a Bruker Avance III 600 MHz NMR spectrometer (Bruker BioSpin, Switzerland) equipped with a 5-mm cryoprobe at a temperature of 295 K. For each sample, a single pulse and a band-selective experiment were performed. The peaks of the glycerol backbone at δ 4.4 ppm were recorded from the single pulse experiment and used for the quantification of the lipid oxidation products. From two band-selective excitation, the signals of hydroperoxides were obtained between δ 11.3 and 10.6 ppm and aldehydes between δ 9.8 and 9.4 ppm. Data were processed using Bruker TopSpin 4.0 software. Concentrations of hydroperoxides and aldehydes were expressed as mmol per kg of oil, and details of calculations were described by Merkx et al. (2018). The analysis was carried out in independent duplicates.

2.2.7 Statistical analysis

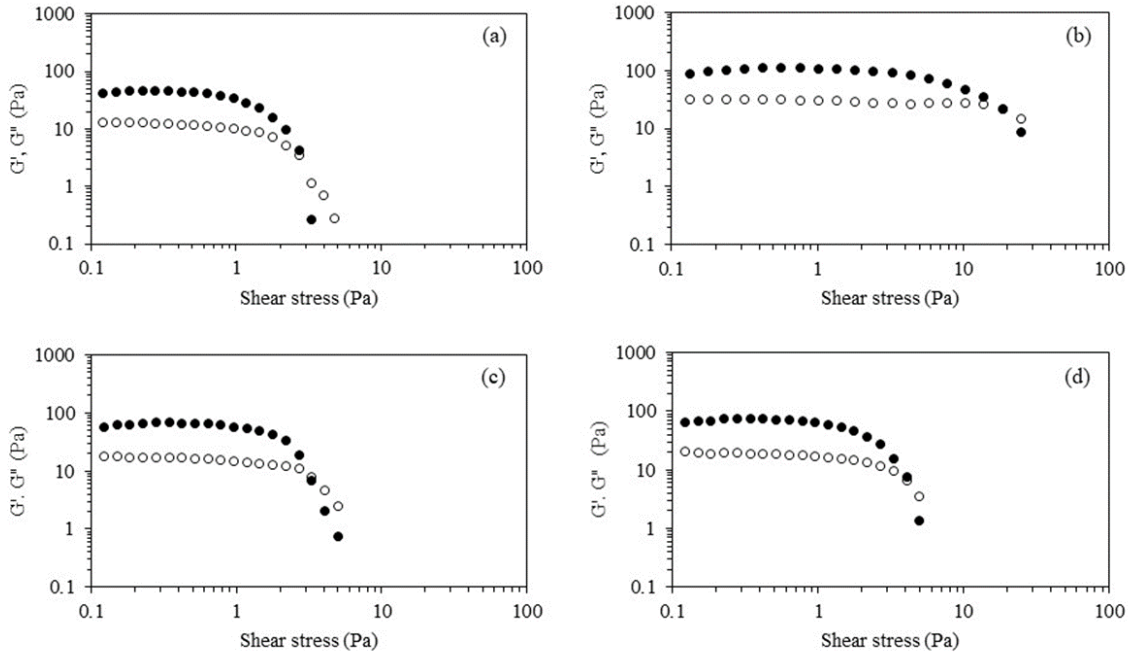
The experimental data were presented as mean ± standard deviation and statistically evaluated by one-way analysis of variance (ANOVA) and Tukey's test at a 95% confidence level ($\alpha = 0.05$). The statistical analyses were performed using the software Statistica 10.0 (StatSoft, Tulsa, USA).

2.3 RESULTS AND DISCUSSION

2.3.1 Viscoelastic properties of the suspensions

Assessing the viscoelastic properties of the açaí suspensions is important for their suitable processing in CTD since information about these materials' internal structure is obtained (BITTERLICH, LUTZ, and ROOSEN, 2002). The mechanical spectra of the pure açaí juice and açaí juice with hydrocolloids are shown in Figure 18. The addition of hydrocolloids increased the storage modulus (G′) of açaí juice due to the higher solid content, increasing the suspensions' elastic properties. Açaí-pectin suspension exhibited the highest G′ values, suggesting a more solid-like behavior.

Figure 18 - Storage (G′, closed symbols) and loss (G″, open symbols) moduli as a function of shear stress of açaí suspensions: (a) pure açaí, (b) açaí-pectin, (c) açaí-maltodextrin, and (d) açaí-gum Arabic.



The suspensions exhibited elastic behavior at low shear stress since solid component values were higher than liquid component values (G′ > G″). At the crossover point (G′ = G″), the storage modulus (G′) suddenly decreased. The critical shear stress values depended on the sample, i.e., about 2.8 Pa for pure açaí juice, 18.7 Pa for açaí-pectin suspension, 3.3 Pa for açaí-

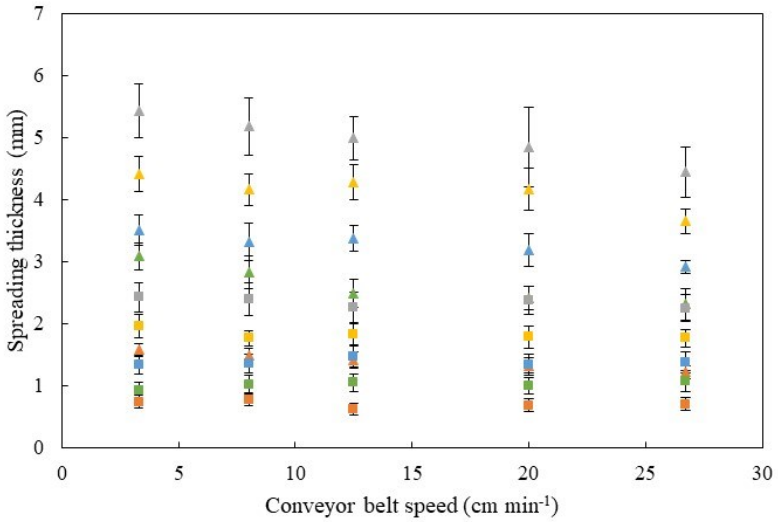
maltodextrin suspension, and 4.1 Pa for açaí-gum Arabic suspension. After the crossover point, the viscous behavior dominated over the elastic behavior (G″ > G′) as the internal structure of the suspension was destroyed. The weak interactions in the suspensions were disrupted by the external shear stress. For spreading suspensions in the CTD apparatus, the critical shear stress must be high enough to prevent inadequate flow and sedimentation of particles during drying but not too high for suspensions to flow under the shear conditions applied during passing through the spreader (BITTERLICH, LUTZ, and ROOSEN, 2002). The açaí-pectin suspension has already shown a high critical shear stress value. Higher values resulted in a non-homogeneous flow out of the suspensions from the spreader under gravitational forces, such as the suspension of açaí juice with 3% pregelatinized starch (Figure 19) with a critical shear stress value of approximately 21 Pa (data not shown).

Figure 19 - Spreading of the açaí juice-pregelatinized starch suspension.

2.3.2 Previous experiments in CTD

The influence of doctor blade gap and conveyor belt speed on the thickness of the spread suspensions was carried out as preliminary experiments in CTD to obtain information about the operational conditions of the spreader gap and belt speed. To that, the suspensions of pure açaí juice and açaí-pectin were tested since these samples showed extreme rheological properties. Five spreader gaps (1.0, 1.5, 2.0, 2.5, and 3.0 mm) and five conveyor belt speeds (3.33, 8.00, 12.5, 20.0, and 26.7 cm min^{-1}) were investigated (Figure 20).

Figure 20 - Thickness of the spread suspensions of (▲) pure açaí juice and (■) açaí-pectin as a function of conveyor belt speed with varying spreader gaps: 1.0 mm (orange), 1.5 mm (green), 2.0 mm (blue), 2.5 mm (yellow), and 3 mm (grey).



The spreading thicknesses of the pure açaí juice using a doctor blade clearance above 2.0 mm, regardless of belt speed, were hard to control due to the low suspension viscosity, resulting in a nonuniformity of the spread film onto the drying support. When the doctor blade gap was set to 1.0 mm, which was the minimum clearance possible to apply, the spreading thickness of the pure açaí juice ranged from 1.22 to 1.59 mm, whereas for açaí-pectin suspension, the thickness ranged from 0.63 to 0.74 mm. Hence, for açaí-pectin suspension, the spreader gap should be adjusted to 2.0 mm so that the spreading thickness varies between 1.33 and 1.48 mm, which is a range close to that of pure açaí juice. Moreover, for all studied spreader clearances, the thickness of the spread pure açaí juice was dependent on the belt speed, i.e., the suspension thickness decreased as increasing the belt speed. On the other hand, the conveyor belt velocity did not influence the thickness of the spread açaí-pectin suspension.

From these results, the doctor blade gaps and conveyor belt speeds for the suspensions were chosen to discard the influence of the spreading thickness on the drying process and to obtain as thin films as possible. Thus, the selected doctor blade gaps were 1 mm and 2 mm for the pure açaí juice and açaí-pectin suspensions, respectively. The selected conveyor belt speed for pure açaí juice was 12.5 cm min^{-1}, which there was no significant difference between the thicknesses of the spread suspensions (Table 8) and allowed the dryer to operate in a continuous regime for the drying of the sample. As the belt velocity did not influence the thickness of açaí-

pectin suspension during spreading, the selected conveyor belt speed was 6.25 cm min^{-1} so the dryer operated continuously.

Table 8 - Spreading thicknesses of pure açaí juice and açaí-pectin suspensions using doctor blade gaps of 1 mm and 2 mm, respectively, at different belt speeds.

Conveyor belt speed (cm min^{-1})	Thickness of the spread suspension (mm)	
	Pure açaí	Açaí-pectin
3.33	1.59 ± 0.09^{aA}	1.33 ± 0.15^{aB}
8.00	1.49 ± 0.12^{abA}	1.36 ± 0.15^{aB}
12.5	1.41 ± 0.13^{bcA}	1.48 ± 0.18^{aA}
20.0	1.33 ± 0.13^{cdA}	1.35 ± 0.17^{aA}
26.7	1.22 ± 0.11^{dB}	1.38 ± 0.16^{aA}

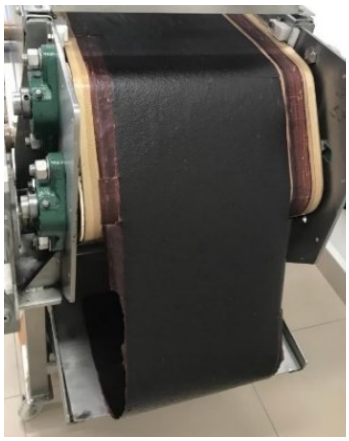
Note: Parameters are expressed as average ± standard deviation (n=10). For each suspension, two independent spreadings were performed, and the measurements were carried out five times. Means followed by different lowercase letters in the same column represent significant differences ($p \leq 0.05$) among the belt speeds and means followed by different uppercase letters in the same row represent significant differences ($p \leq 0.05$) between the suspensions, according to Tukey's test.

2.3.3 Performance of the CTD process

All açaí suspensions were evenly spread as thin layers on the flexible support. The incorporation of hydrocolloids to açaí juice influenced the drying time of the samples in CTD (Table 9). Açaí juice without hydrocolloids showed the lowest drying time (16 min), whereas the suspension added with pectin exhibited the highest process time in CTD, requiring the twice time length (32 min) to reach a moisture content below 5% (w.b.). The higher drying time of açaí-hydrocolloid suspensions may be explained by the water-binding properties of the biopolymers (GUJRAL and BRAR, 2003). Nevertheless, drying times in CTD were shorter than in FD. Similar behavior was reported by Baeghbali, Niakousari, and Farahnaky (2016), who compared refractance window drying and freeze-drying.

The rheological properties of suspensions strongly influence the morphology and quality of the dried product. At the end of the CTD process, the pure açaí juice, açaí-maltodextrin, and açaí-gum Arabic samples were withdrawn from the drying support as flakes. On the other hand, the dried açaí-pectin suspension was removed as a film (Figure 21) due to the more rigid structure formed by interactions between the components of açaí jucie and pectin.

Figure 21 - Detachment of the dried açaí-pectin suspension from the conveyor belt.



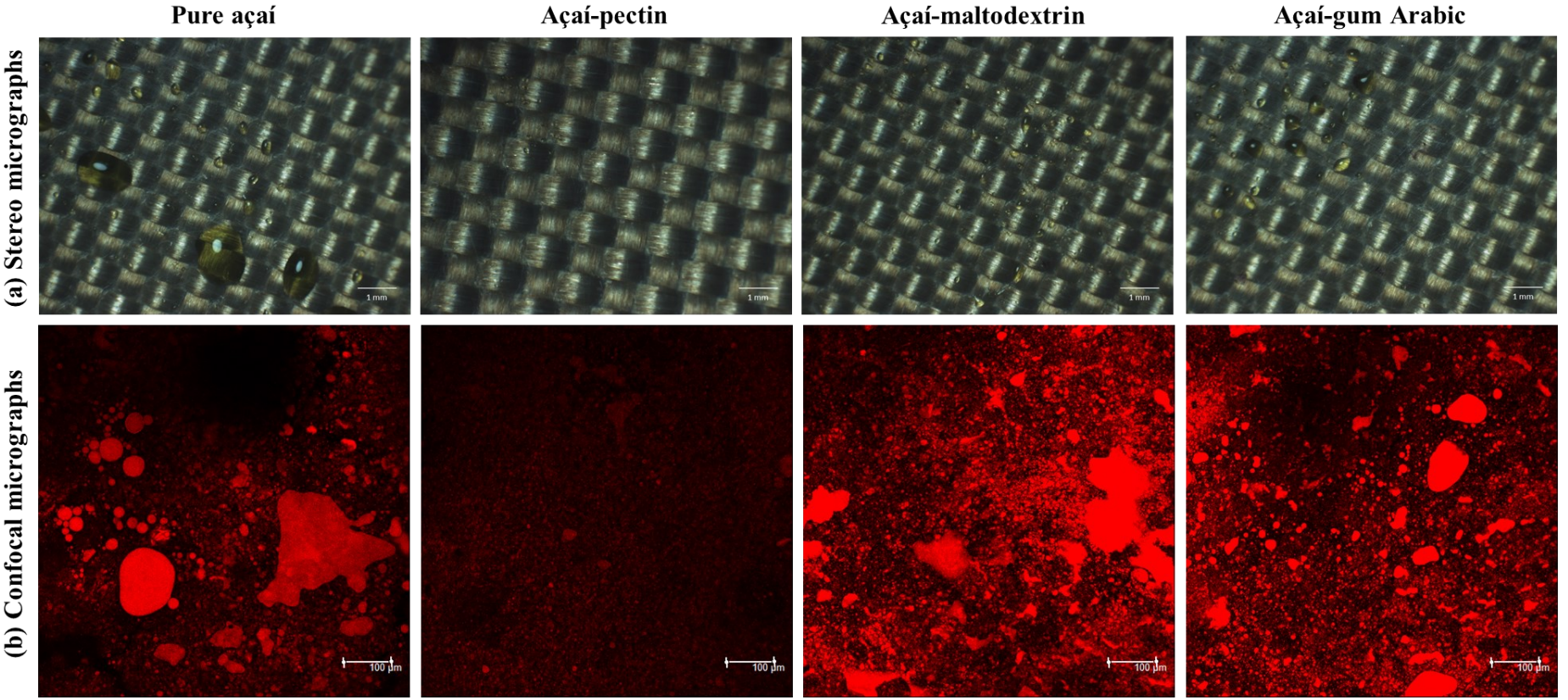
After drying and samples' removal, oil release from the suspensions to the fiberglass support coated with Teflon® was visually observed by stereo micrography (Figure 22a), except for the dried açaí-pectin suspension. This result can be associated with the strongest internal network of the açaí-pectin suspension indicated by the highest storage modulus and critical shear stress. This oil loss was confirmed quantitatively by analyzing the lipid content. The oil released during drying leads to a significant decrease in the product's nutritional value and operational problems, e.g., frequent dryer downtime for cleaning.

Table 9 - Drying time, moisture content, water activity, lipid content, and color of the açaí powders produced by cast-tape drying (CTD) and freeze-drying (FD).

Parameters	**Powder samples**				
	Pure açaí	**Açaí-pectin**	**Açaí-maltodextrin**	**Açaí-gum Arabic**	**Freeze-dried pure açaí**
Drying time (h)	0.27	0.53	0.42	0.42	24
Moisture content (g g^{-1}, d.b.)	0.016 ± 0.009	0.023 ± 0.006	0.016 ± 0.003	0.013 ± 0.004	0.023 ± 0.005
Water activity	0.420 ± 0.033	0.412 ± 0.039	0.327 ± 0.033	0.242 ± 0.046	0.393 ± 0.050
Lipid content (g 100 g^{-1}, d.b.)	49.65 ± 0.64^{b}	55.67 ± 3.69^{a}	49.76 ± 0.78^{b}	50.45 ± 1.17^{b}	52.02 ± 0.20^{ab}
Color					
*L**	12.09 ± 0.92^{a}	12.69 ± 0.61^{a}	12.48 ± 0.71^{a}	12.21 ± 0.29^{a}	12.69 ± 0.74^{a}
*a**	3.64 ± 0.39^{b}	4.69 ± 0.25^{a}	3.92 ± 0.21^{b}	3.94 ± 0.12^{b}	4.79 ± 0.59^{a}
*b**	-3.95 ± 0.43^{a}	-3.86 ± 0.36^{a}	-3.79 ± 0.21^{a}	-3.78 ± 0.03^{a}	-3.65 ± 0.27^{a}
*ΔE**	1.33	0.24	0.90	0.99	-

Note: Parameters are expressed as average ± standard deviation (n=3 or n=9). For each açaí powder, the drying experiments were performed in independent triplicates, and the samples were evaluated in triplicate, except for the determination of lipid content, in which each replicate of the açaí powder was evaluated once. Means followed by different letters in the same row represent significant differences ($p \leq 0.05$), according to Tukey's test.

Figure 22 - (a) Stereo micrographs of fiberglass support coated with Teflon® and (b) confocal analysis of the açaí samples after the cast-tape drying (CTD) process.



2.3.4 Oil distribution in the dried açaí suspensions

Figure 22b shows images obtained by confocal microscopy of the oil droplets distribution in the samples dehydrated by CTD. Large oil droplets were irregularly distributed in the dried açaí suspensions, except for the açaí-pectin sample, in which small oil droplets were uniformly distributed. The pectin may have encapsulated the oil present in the açaí juice since high methoxylated pectin is highly hydrophobic and can interact with hydrophobic molecules, such as açaí oil (REHMAN et al., 2019).

2.3.5 Properties of açaí powders

2.3.5.1 Moisture content, water activity, lipid content, and color

The moisture content and water activity of açaí powder samples produced by CTD and FD were around 0.02 g g^{-1} (dry basis) and lower than 0.420, respectively (Table 9). These values were close to those reported by Tonon et al. (2009c) and Pavan (2010) for producing açaí powder with different carrier agents by spray drying and pure açaí powder using different drying processes (refractance window drying, hot air drying, and freeze-drying), respectively.

Lipids are the main compounds in the açaí juice, representing more than 50% of dry matter, as observed in Table 9. Adding pectin to the açaí juice hindered the oil separation during the drying process by CTD. Thereby, the açaí-pectin powder showed the highest lipid content compared to the other açaí powders (Table 9), asserting the results discussed earlier.

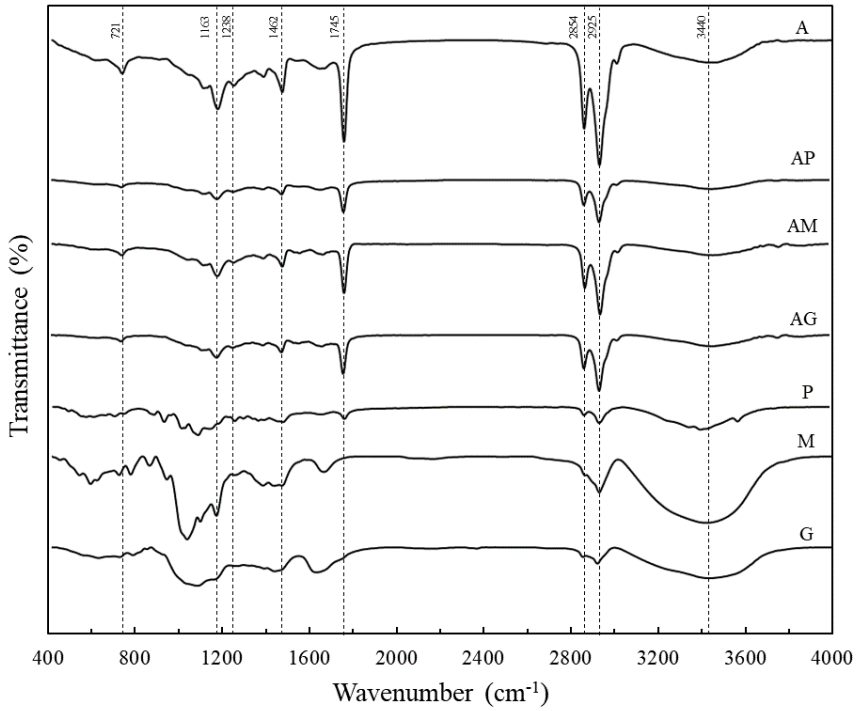
The values of the color parameters (L^*, a^*, and b^*) and total color difference (ΔE^*) of the açaí powders produced by CTD and FD are presented in Table 9. The addition of pectin resulted in a significantly higher a^* value (redness) in comparison with the other powder samples produced by CTD, which could be attributed to the encapsulation of oil by pectin since the açaí oil color is dark green (SILVA and ROGEZ, 2013). Moreover, there was no statistical difference between the color of açaí-pectin powder and freeze-dried pure açaí. On the other hand, higher values of color change were observed between the other samples produced by CTD and freeze-dried powder. This color difference is slightly noticeable by consumers (CSERHALMI et al., 2006). Figure 23 shows images of the açaí powders produced by CTD and FD.

Figure 23 - Açaí powders produced by cast-tape drying (CTD) and freeze-drying (FD).

2.3.5.2 Fourier transform infrared (FTIR) spectroscopy analysis

FTIR spectrometry analysis was applied to evaluate the molecular structure of açaí powders obtained from CTD and possible interactions between açaí juice and hydrocolloids in the powder samples. Figure 24 presents the FTIR spectra of açaí powders with and without hydrocolloids and the spectrum of each hydrocolloid.

Figure 24 - FTIR spectra of açaí powders produced by cast-tape drying (CTD): pure açaí (A), açaí-pectin (AP), açaí-maltodextrin (AM), and açaí-gum Arabic (AG); and hydrocolloids: pectin (P), maltodextrin (M), and gum Arabic (G).



Note: The dashed lines were added to guide the eyes.

Characteristic bands of lipids, flavonoids, and polysaccharides functional groups were contained in the FTIR spectrum of pure açaí powder. The broad peak at 3440 cm^{-1} was associated with the O-H stretching of hydroxyl groups. Bands around 2925 cm^{-1}, 2854 cm^{-1}, and 1745 cm^{-1} were attributed to asymmetric and symmetric CH_2 stretching, and C=O stretching vibrations of esterified carboxyl groups in lipids, respectively. The absorption peak at 1462 cm^{-1} was assigned to the C=C stretching vibrations of the aromatic ring in flavonoids, as well as the bending (scissoring) vibrations of the CH_2 and CH_3 aliphatic groups in lipids. Bands at 1238 cm^{-1} and 1163 cm^{-1} were related to C-O stretching vibration in polysaccharides and at 721 cm^{-1} associated with the C-H bending (rocking) vibrations in lipids (HO et al., 2019; OLIVEIRA et al., 2020; TEIXEIRA-COSTA et al., 2021). The typical absorption bands for pectin, maltodextrin 10DE, and gum Arabic are summarized in Table 10 (BALLESTEROS et al.; 2017; GNANASAMBANDAM and PROCTOR, 2000; RÓŻYŁO et al., 2021; TAO et al., 2017) The FTIR spectra of the açaí powders added with hydrocolloids displayed similar peak positions to pure açaí powder, indicating a physical interaction rather than chemical interaction between açaí juice and hydrocolloids.

Table 10 - FTIR bands assignment for pectin, maltodextrin, and gum Arabic.

Wavenumber (cm^{-1})			Band assignment
Pectin	**Maltodextrin 10DE**	**Gum Arabic**	
3390	3411	3433	O–H stretching broad band of hydroxyl group
2924	2920	2920	Asymmetric CH_2 stretching
2854	2854	2852	Symmetric CH_2 stretching
1745			C=O stretching of esterified carboxyl groups
	1651	1624	C=O stretching of nonesterified carbonyl groups
		1429	C-H bending
1122	1157	1146	C-O stretching of ether group
1070	1082	1072	C-O stretching
	1022		C-H and CH_2 bending
866			α(1–4) glycosidic linkage
	849		C–O–C stretching of glycosidic bonds

2.3.5.3 Total monomeric anthocyanin content and antioxidant activity

The açaí juice used in this study showed an anthocyanin content of 314.2 ± 17.1 mg of cyanidin-3-rutinoside equivalent 100 g^{-1} of dry extract. This value is higher than one reported by Gordon et al. (2012) (17.9 mg of cyanidin-3-rutinoside equivalent 100 g^{-1} of dry extract). However, Bichara and Rogez (2011) found a higher anthocyanin content (1441.3 mg of

cyanidin-3-rutinoside equivalent 100 g^{-1} of dry extract) than that obtained in the present study. These differences could be explained by the wide variability of raw material due to seasonality and region of production, as well as processing conditions (DE JESUS et al., 2020).

The total monomeric anthocyanin content, anthocyanin retention percentages, and antioxidant activity determined by the DPPH assay in the açaí powders produced by CTD, with and without the addition of hydrocolloids, and by FD are presented in Table 11. Both chemical properties were calculated by excluding the lipid content of samples to have the same dry matter content in all açaí powders. The retention of anthocyanins was calculated as the ratio of anthocyanin content in açaí powder to that in açaí juice before drying.

Table 11 - Total monomeric anthocyanin content (TMA), percentage of anthocyanin retention, and antioxidant activity (AA) in the açaí powders produced by cast-tape drying (CTD) and freeze-drying (FD), disregarding the mass of hydrocolloid and lipid content.

Powder samples	**TMA (mg 100 g^{-1} d.b.)**	**Anthocyanin retention (%)**	**AA (µmol TE g^{-1} d.b.)**
Pure açaí	494.7 ± 54.0^{c}	75	268.0 ± 15.8^{c}
Açaí-pectin	636.6 ± 47.2^{a}	97	336.6 ± 37.8^{ab}
Açaí-maltodextrin	453.4 ± 39.4^{c}	69	286.0 ± 20.4^{c}
Açaí-gum Arabic	568.4 ± 28.5^{b}	86	320.6 ± 29.4^{b}
Freeze-dried pure açaí	636.4 ± 58.5^{a}	97	355.2 ± 12.5^{a}

Note: Parameters are expressed as average ± standard deviation (n=6 or n=9). For each açaí powder, the drying experiments were carried out in independent triplicates, and the measurements were performed in triplicate for the determination of TMA and in duplicate for the determination of AA. Means followed by different letters in the same column represent significant differences ($p \leq 0.05$), according to Tukey's test.

Açaí powders added with pectin showed the highest anthocyanin retention (97%) among the powder samples produced by CTD, and the biocompound content was similar to that of freeze-dried açaí powder. The anthocyanin content of açaí-pectin powder was approximately 1.3 times higher than that of pure açaí powder. The anthocyanin retention in the açaí-pectin powder was also higher compared to those reported by Pavan (2010) and Souza (2015), who produced pure açaí powder by refractance window drying. In contrast, the addition of maltodextrin did not protect anthocyanins, i.e., açaí-maltodextrin powder did not display a statistical difference from cast-tape dried pure açaí juice. These results may be related to the chemical structure and the presence of hydrophilic/hydrophobic groups in the hydrocolloids. Lower DE maltodextrins contain a large proportion of long chains and cannot create a dense and less oxygen permeable network, resulting in poor protection to oxygen-sensitive compounds, such as anthocyanins (WAGNER and WARTHESEN, 1995). On the other hand, pectin has been demonstrated to interact with anthocyanins through hydrogen bonding or

hydrophobic interactions and these interactions are influenced by the structural flexibility of the hydrocolloid (LARSEN et al., 2019).

The antioxidant activity of samples followed a similar tendency to the anthocyanin content. The addition of pectin resulted in an açaí powder with significantly higher antioxidant activity than that of cast-tape dried pure açaí sample and comparable to that of freeze-dried powder. Other authors have already reported the existing relation between anthocyanin content and antioxidant activity in açaí products (DEL POZO-INSFRAN, BRENES, and TALCOTT, 2004; PACHECO-PALENCIA, DUNCAN, and TALCOTT, 2009; TONON, BRABET, and HUBINGER, 2010). These results suggest the protective effect of pectin on anthocyanin retention and antioxidant activity preservation during the drying process of açaí by CTD.

2.3.5.4 Lipid oxidation products

Açaí contains a high unsaturated fatty acids content, representing approximately 74% of all fatty acids. Oleic acid, linoleic acid, and linolenic acid, which are monounsaturated and polyunsaturated fatty acids, constitute around 56%, 13%, and 1% of the total fatty acids in açaí, respectively (SCHAUSS et al., 2006). During processing and storage, açaí is potentially prone to lipid oxidation, a chemical reaction responsible for forming primary and secondary oxidation compounds. The formation of hydroperoxides and aldehydes, which are primary and secondary lipid oxidation products, respectively, after the drying processes of the açaí samples were determined by ^{1}H NMR. Figure 25 shows ^{1}H NMR spectra of the açaí oils extracted from powdered samples produced by CTD and FD, and the assignment of hydroperoxide and aldehyde signals according to Merkx et al. (2018) is given in Table 12. The oxidation process of linolenic acyl groups formed cyclized hydroperoxides (peak 1), 12- and 13-hydroperoxide isomers (peak 2), and 9- and 16-hydroperoxide isomers (peak 3). The cyclized hydroperoxides are generated by the internal cyclization of 12- and 13-hydroperoxides (FRANKEL, 2012). Two conjugated diene hydroperoxides (peak 4), i.e., 9-hydroperoxy-*trans*-10,*trans*-12-octadecadienoate (*trans*,*trans*-9-OOH) and 13-hydroperoxy-*trans*-9,*trans*-11-octadecadienoate (*trans*,*trans*-13-OOH), were formed in the oxidation of linoleic acyl groups. The formation of 8-hydroperoxy-cis-9-octadecenoate (*cis*-8-OOH) and 11-hydroperoxy-cis-9-octadecenoate (*cis*-11-OOH) isomers (peak 5) resulted from the degradation of oleic acyl groups (FRANKEL, 2012; MERKX et al., 2018). Although a difference in the intensities of signals in the hydroperoxide region can be observed when comparing the spectra of açaí powders, the

concentration of the primary lipid oxidation products (Table 13), did not show a statistically significant difference among samples.

Table 12 - ^{1}H NMR signals assignments of the products generated in the lipid oxidation process of the açaí powders.

	Peak	Chemical shift (ppm)	Compounds
Hydroperoxides			
	1	11.05-11.00	Cyclized
	2	10.99-10.96	12-OOH; 13-OOH
	3	10.95-10.91	9-OOH; 16-OOH
	4	10.87-10.82	*trans*,*trans*-9-OOH; *trans*,*trans*-13-OOH
	5	10.74-10.71	*cis*-8-OOH; *cis*-11-OOH
Aldehydes			
	6	9.75-9.73	*n*-alkanals ($n > 5$)
	7	9.58-9.54	4-hydro(pero)xy-*trans*-2-alkenals
	8	9.53-9.50	*trans*,*trans*-2,4-alkadienals; 4,5-epoxy-*trans*-2-alkenals

Secondary oxidation compounds were also generated during the drying process of the açaí samples as a result of the extended exposure of hydroperoxides to oxidation conditions. Aldehydes were the secondary oxidation products detectable by ^{1}H NMR with signals between δ 9.8 and 9.4 ppm (Figure 25b). *n*-alkanals (peak 6), 4-hydro(pero)xy-*trans*-2-alkenals (peak 7), and *trans*,*trans*-2,4-alkadienal and 4,5-epoxy-*trans*-2-alkenals (peak 8) were the aldehydes found in the samples. Similar compounds were reported by Guillén and Ruiz (2008), who evaluated corn oil and linseed oil, which are oils rich in polyunsaturated groups, submitted to 190 °C with aeration in a convection oven or microwave action without exceeding 190 °C. There was no statistical difference between the concentration of total aldehydes in the cast-tape dried samples (Table 13). However, the concentration of aldehydes was significantly lower in the açaí oil from freeze-dried pure açaí, possibly due to the lower formation of 4-hydro(pero)xy-*trans*-2-alkenals, *trans*,*trans*-2,4-alkadienal, and 4,5-epoxy-*trans*-2-alkenals in the sample, which could be correlated to temperature and oxygen exposure conditions during the drying process. The formation of 4-hydro(pero)xy-*trans*-2-alkenals and 4,5-epoxy-*trans*-2-alkenals, which are oxygenated α,β-unsaturated aldehydes, require special attention since they have been considered possible causative agents of several degenerative diseases (GUILLÉN and GOICOECHEA, 2008; ALBUQUERQUE, COSTA, and OLIVEIRA, 2022). The occurrence of these compounds has also been observed in different foods rich in unsaturated fatty acids, such as dry nuts (PAPASTERGIADIS et al., 2014), dried seaweeds (HARRYSSON et al., 2021), corn oil stored at room temperature (GUILLEN and GOICOECHEA, 2009), and

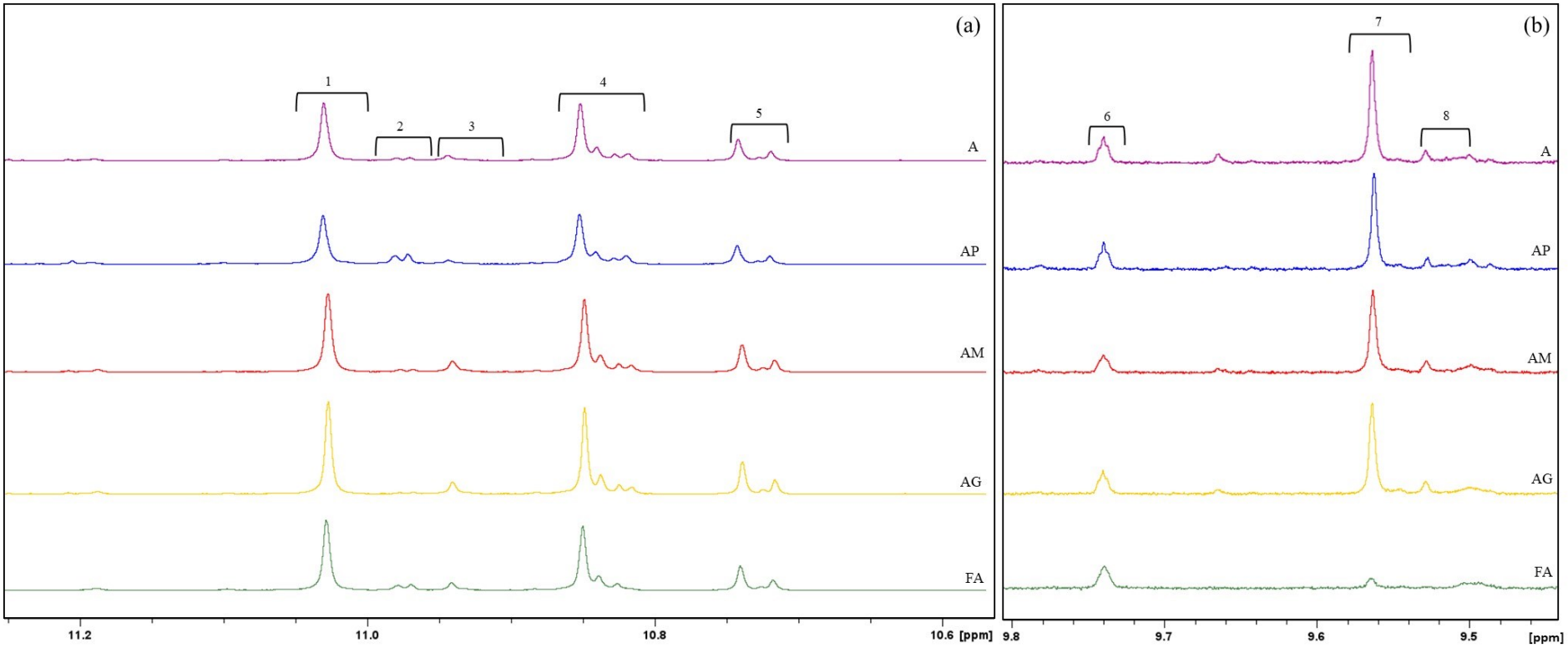
linolenic acid-enriched egg yolk powder (MEYNIER et al., 2014). To the best of our knowledge, this is the first time that the nature and proportions of these different aldehydes generated in the açaí oil have been determined.

Table 13 - The concentration of hydroperoxides and aldehydes in the açaí powders produced by cast-tape drying (CTD) and freeze-drying (FD).

Powder samples	**Hydroperoxides (mmol kg^{-1} oil)**	**Aldehydes (mmol kg^{-1} oil)**
Pure açaí	13.8 ± 2.0^{a}	2.45 ± 0.21^{a}
Açaí-pectin	15.1 ± 1.3^{a}	2.50 ± 0.14^{a}
Açaí-maltodextrin	18.2 ± 0.8^{a}	2.35 ± 0.07^{a}
Açaí-gum Arabic	16.7 ± 3.7^{a}	2.35 ± 0.07^{a}
Freeze-dried pure açaí	14.3 ± 2.8^{a}	1.10 ± 0.00^{b}

Note: Parameters are expressed as average ± standard deviation (n=2). For each açaí powder, the measurements were performed in independent duplicates Means followed by different letters in the same column represent significant differences ($p \leq 0.05$), according to Tukey's test.

Figure 25 - ^{1}H NMR spectra of the (a) hydroperoxide region (δ 11.25 – 10.6 ppm) and (b) aldehyde region (δ 9.81 – 9.44 ppm) of the açaí powders produced by cast-tape drying (CTD): pure açaí (A), açaí-pectin (AP), açaí-maltodextrin (AM), and açaí-gum Arabic (AG); and by freeze-drying (FD): freeze-dried pure açaí (FA).



2.4 CONCLUSION

The type of added hydrocolloid to açaí juice played an important role in the processing characteristics of CTD and final product properties. The addition of pectin, maltodextrin, and gum Arabic increased the elastic properties of the suspensions, influencing thus the choice of the spreader gap and belt velocity. The CTD process allowed dehydrating the açaí suspensions in shorter drying times than FD. The açaí-pectin suspension showed the longest drying time compared to other suspensions dried by CTD. However, the addition of pectin hampered the oil release from the sample and facilitated the removal of the dried material as a film from the belt due to the strongest internal network formed in the sample. In addition, açaí powder produced with pectin stood out for its color, anthocyanin content, and antioxidant activity similar to those of freeze-dried açaí juice, suggesting the protective effect of this hydrocolloid during CTD of açaí juice. However, all powdered samples produced by CTD presented a higher formation of aldehydes, which are secondary lipid oxidation products. Among the detected aldehydes, 4-hydro(pero)xy-*trans*-2-alkenals were the major generated compounds. The formation of these compounds has caused concern for food scientists due to their possible toxicity. Therefore, additional studies about the determination of oxygenated α,β-unsaturated aldehydes concentration in the açaí oil of cast-tape dried samples, as well as the risk assessment for human health on the consumption of these powder products, are recommended.

CHAPTER 3. ASSESSMENT OF RELATIVE HUMIDITY EFFECT ON THE STABILITY OF AÇAÍ POWDERS DRIED BY CAST-TAPE DRYING

In this chapter, açaí powders, with and without the addition of pectin, produced by cast-tape drying were equilibrated at different relative humidities at 25 °C. Moisture sorption isotherms, total monomeric anthocyanin content, and instrumental color measurements of the stored açaí powders were evaluated. The BET model estimated the monolayer moisture content. The addition of pectin increased the hygroscopicity of açaí powders, showing a higher monolayer moisture content (0.037 g g^{-1} d.b.) than pure açaí powder (0.011 g g^{-1} d.b.). The corresponding water activities to these monolayer moisture contents were approximately 0.52 and 0.23, respectively. These values were supposed as the conditions for good storage stability. However, the anthocyanin degradation in the açaí powders was favored at the storage water activities below the critical water activity corresponding to the BET monolayer. On the other hand, açaí-pectin powders conditioned at higher relative humidities showed higher a^* values, resulting in a reddish tinge of samples. Moreover, the color parameters weakly correlated with the anthocyanin content of the açaí powders stored at different relative humidities, suggesting that color measurements cannot be useful as indicators of anthocyanin stability in açaí powders under the studied conditions. This study was performed at the Laboratory of Physical Properties of Food (PROFI) at the Federal University of Santa Catarina (Brazil).

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3.1 INTRODUCTION

Fruit powders represent an interesting alternative for preserving, adding value, and diversifying consumption options of several fruits. They are convenient and healthy food products that can be used as ingredients for developing other food products or reconstituting fruit juices or pulps. However, powdered foods are sensitive to heat, moisture, oxygen, and light, which are crucial external factors to determine the stability of these products. Certain conditions during the storage of food powders are required to avoid since they result in functional property changes or deterioration of sensorial or nutritional quality of products (HEDEGAARD and SKIBSTED, 2013).

The degree of water in food to participate in reactions is related to water activity and plays an essential role in degrading dehydrated foods (JANGAM and MUJUMDAR, 2010). The water activity level influences the product stability during storage, affecting the rate of physical alterations, such as caking and stickiness, as well as color loss, biocompounds degradation, and lipid oxidation, resulting in sensorial and nutritional damages to the product (HEDEGAARD and SKIBSTED, 2013).

Açaí (*Euterpe oleracea* Mart.) is an Amazon fruit that has received much attention in the last years because of the benefits to human health associated with its nutritional composition (YAMAGUCHI et al., 2015). Açaí is an important source of anthocyanins, which are the compounds responsible for açaí color. These pigments also account for approximately 90% of the antioxidant activity of açaí (PACHECO-PALENCIA, DUNCAN, and TALCOTT, 2009). Nevertheless, anthocyanins are highly unstable and susceptible to degradation during the processing and storage of foods, which might result in undesirable changes in colors and antioxidant activity. Their stability depends on many factors, including temperature, water activity, light, oxygen, pH, and ascorbic acid, among others (CASTAÑEDA-OVANDO et al., 2009; WROLSTAD, DURST, and LEE, 2005). Tonon, Brabet, and Hubinger (2010) studied the effect of two water activities (0.328 and 0.529) on the anthocyanin degradation in spray-dried açaí powders during storage. The authors observed that increasing storage water activity resulted in lower anthocyanin retention. Other authors have also reported the negative effect of relative humidity (RH) during storage on anthocyanin retention in dehydrated fruit products (AGUDELO-LAVERDE, SCHEBOR, AND BUERA, 2013; SIMÃO et al., 2022; SYAMALADEVI et al., 2011).

The addition of hydrocolloids to fruit juice or pulp is a strategy for aiding in the stability of fruit powders during storage. This incorporation may limit the oxygen diffusion inside of the product and thus reduce the degradation rates, such as biocompounds oxidation. Tonon et al. (2009c) evaluated the total polyphenolics and antioxidant activity in açaí powders produced by spray drying with the addition of different hydrocolloids (maltodextrin 10DE, maltodextrin 20DE, gum Arabic, and tapioca starch) and by freeze-drying (without hydrocolloid) during storage in two different relative humidities (32.8% and 84.3%). At both relative humidities, a higher reduction in total polyphenolics and antioxidant activity was observed for the freeze-dried sample, indicating the protective effect of additives.

Therefore, this study aimed to evaluate the impact of storage relative humidity on the stability of the açaí powders with and without the addition of pectin. It was hypothesized that the powders' storage at relative humidities below the critical water activity corresponding to the monolayer moisture content would contribute to the stability of anthocyanins and color in the açaí powders. For this purpose, the following points were considered: (i) determination of moisture sorption isotherms for both samples; (ii) investigation of the changes in the total monomeric anthocyanin content and color parameters of the samples stored in a wide water activity range; and (iii) correlation between anthocyanin content and color measurements of the açaí powders.

3.2 MATERIAL AND METHODS

3.2.1 Preparation of açaí powders

Frozen açaí juice bars (Norfrutas Eireli, Belém, Brazil) were purchased at a local market in Florianópolis, Brazil. The açaí juice, with a moisture content of 84.6 ± 0.6 g 100 g^{-1} (w.b.), was thawed at room temperature until its temperature achieved 10 °C. GENU® high methoxyl pectin type 106 BP (CPKelco, Limeira, Brazil) with a DM of 68% was used as the hydrocolloid. Two suspensions were prepared: pure açaí juice and açaí-pectin. Pectin was added to açaí juice at a concentration of 3% (3 g of hydrocolloid per 100 g of açaí juice), using a household hand blender (Oster, FPSTHB2610R-057, Brazil) at the minimum speed for 2 min.

Açaí powders were produced by continuous cast-tape drying (CTD). The suspensions were evenly spread over a fiberglass conveyor belt coated with Teflon® (Indaco, Lençol Armalon® Standard, Brazil) using a doctor blade with a gap of 1 mm for pure açaí juice and

2 mm for açaí-pectin suspension. The resulting thickness of spread suspensions was approximately 1.5 mm. Steam released from hot water at 98 °C was used as the heating medium to provide the heat latent of water evaporation during the drying process. The evaporated water during drying was removed by natural convection. The ambient relative humidity was between 64% and 67%, and the temperature ranged from 24.2 °C to 24.8 °C. The suspensions were dried until moisture reached less than 5% (w.b.), following the Brazilian legislation (MAPA, 2018). Açaí powders were obtained by grounding the dried samples using a knife mill (TECNAL, TE 631/2, Brazil) and sieving using a 20-mesh sieve.

3.2.2 Storage conditions

Açaí powders were previously freeze-dried for 24 h to remove the residual moisture content. Then, about 1 g of the samples were conditioned at 25 °C in hermetic desiccators containing six different saturated salt solutions (LiCl, CH_3COOK, $MgCl_2$, K_2CO_3, $Mg(NO_3)_2$, and NaBr), giving relative humidities of 11.3%, 22.5%, 32.8%, 43.2%, 52.9%, and 57.6% (GREENSPAN, 1977). Samples had also been stored in desiccators with saturated salt solutions maintaining the ambient relative humidity above 60%. However, chloroform, which was added to prevent microbial growth in the samples, favored the oil extraction from açaí powders, modifying thus their hygroscopicity.

Stored samples were weighed at regular time intervals until achieving the equilibrium, which took 60 days and was obtained when the difference between two consecutive weighings was less than 1 mg g^{-1} solids (LABUZA, KAANANE, and CHEN, 1985). Once the equilibrium was reached, moisture sorption isotherms, anthocyanin content, and color parameters were determined for the pure açaí powder and açaí-pectin powder. All samples were analyzed in dependent triplicate.

3.2.3 Moisture sorption isotherm

Moisture sorption isotherms of açaí powders were determined by the static gravimetric method. After equilibrium, the moisture content of samples conditioned at different RH was determined in a vacuum oven (TECNAL, TE-395, Brazil) at 70 °C (AOAC, 2005). The Brunauer-Emmett-Teller (BET) and Guggenheim-Anderson-de Boer (GAB) models (Equations 3 and 4, respectively) were fitted to the experimental data of moisture sorption

isotherms. The parameters of models were estimated by the Solver algorithm of Microsoft Excel (Microsoft Corporation, Microsoft 365, USA). The fit goodness was evaluated by the coefficient of determination (R^2).

$$X_{eq} = \frac{X_m.C_{BET}.a_w}{(1-a_w).(1+(C_{BET}-1).a_w)} \quad (3)$$

where X_{eq} is the equilibrium moisture content (g g^{-1} d.b.), X_m is the monolayer moisture content (g g^{-1} d.b.), C_{BET} is the BET constant related to the sorption heat of the net, and a_w is the water activity. The BET model is adequate within 0-0.55 water activity (LABUZA and ALTUNAKAR, 2007).

$$X_{eq} = \frac{X_m.C_{GAB}.k.a_w}{(1-k.a_w).(1-k.a_w+C_{GAB}.k.a_w)} \quad (4)$$

where X_{eq} is the equilibrium moisture content (g g^{-1} d.b.), X_m is the monolayer moisture content (g g^{-1} d.b.), C_{GAB} is the Guggenheim constant related to the total sorption heat of the monolayer, k is the GAB constant related to the total sorption heat of the multilayer, and a_w is the water activity. The GAB model is valid in the water activity range between 0 and 0.95 (LABUZA and ALTUNAKAR, 2007).

3.2.4 Total monomeric anthocyanin content

The total monomeric anthocyanin content in the stored açaí powder samples was determined using the pH-differential method proposed by Giusti and Wrolstad (2001). For extraction of anthocyanins, about 0.3 g of açaí powder was mixed with 10 mL of HCl/water/ethanol solution (1:29:70, v/v/v), sonicated for 5 min, and then centrifuge for 10 min at 3,400 rpm. These steps were performed three times. Supernatants were collected and pooled. Afterward, two different buffer solutions (pH 1.0 and pH 4.5) were used to dilute the extracts. Absorbances were read at 523 and 700 nm, using a molecular weight of 631 g mol^{-1} and molar absorptivity (ε) of 28,840 L mol^{-1} cm^{-1}. The results were expressed in mg of cyanidin-3-rutinoside equivalent per 100 g of dry extract, disregarding the mass of pectin. According to the literature, cyanidin-3-rutinoside is the predominant anthocyanin present in açaí (GORDON et

al., 2012; PACHECO-PALENCIA, DUNCAN, and TALCOTT, 2009; TONON, BRABET, and HUBINGER, 2010).

3.2.5 Color

The color parameters (L^*, a^*, and b^*) of açaí powders stored at different relative humidities were evaluated using a computer vision system (Cárdenas-Pérez et al., 2017). Images obtained with a photograph camera (Nikon Corporation, Nikon D5500, Japan) were processed in the ImageJ 1.53k software (National Institutes of Health, USA). The color space converter plug-in was applied to convert from the RGB system to the CIELab scale.

3.2.6 Statistical analysis

The results are presented as mean ± standard deviation. One-way analysis of variance (ANOVA) and Tukey's test at the 95% confidence level ($\alpha = 0.05$) were applied to analyze the experimental data. The color parameters and anthocyanin content were correlated by linear regression, and the fit goodness was determined by the coefficient of determination (R^2), coefficient of correlation (r), and p-value (p). The analyses were performed using Statistica 10.0 software (StatSoft, USA).

3.3 RESULTS AND DISCUSSION

3.3.1 Moisture sorption isotherm

The analysis of moisture sorption isotherm provides essential information to determine the appropriate processing and storage conditions of food products. The equilibrium moisture content of the açaí powders produced by cast-tape drying (CTD) for each storage water activity at 25 °C is shown in Table 14. Noticeable differences between the equilibrium moisture content data of the powders were observed for water activity above 0.432. Samples containing pectin were more hygroscopic than pure açaí powders. This behavior could be related to the oil separation on the particle surface of the pure açaí powder and the ability of pectin to encapsulate the oil in the açaí-pectin powder, as discussed in the previous chapter, as well as the presence of hydrophilic sites in the hydrocolloid.

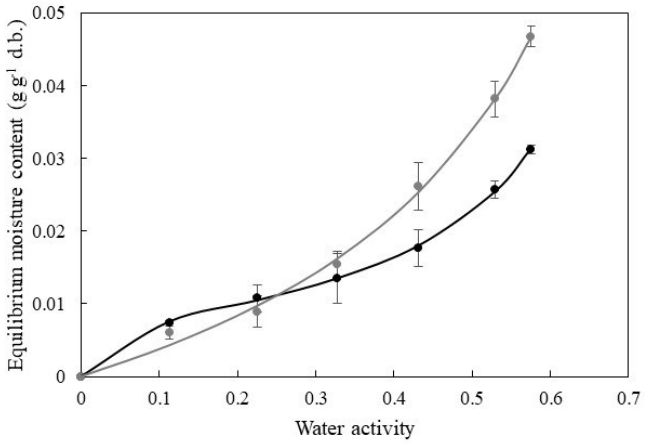
Table 14 - The equilibrium moisture content as a function of water activity for the pure açaí and açaí-pectin powders at 25 °C.

Water activity	Equilibrium moisture content (g g^{-1} d.b.)	
	Pure açaí	Açaí-pectin
0.113	0.007 ± 0.000[a]	0.006 ± 0.001[a]
0.225	0.011 ± 0.002[a]	0.009 ± 0.002[a]
0.328	0.014 ± 0.003[a]	0.015 ± 0.002[a]
0.432	0.018 ± 0.003[b]	0.026 ± 0.003[a]
0.529	0.026 ± 0.001[b]	0.038 ± 0.002[a]
0.576	0.031 ± 0.001[b]	0.047 ± 0.001[a]

Note: Parameters are expressed as average ± standard deviation (n=3). For each açaí powder, the measurements were performed in triplicate. Means followed by different letters in the same row represent significant differences ($p \leq 0.05$), according to Tukey's test.

The estimated parameters of the BET and GAB models are presented in Table 15. The GAB model well mathematically described the experimental data of moisture sorption isotherms over the whole water activity range ($R^2 > 0.997$). Thus, the GAB model was selected to represent the experimental data of the açaí powders in Figure 26. Even though the GAB model provides a better mathematical representation of moisture sorption isotherm over the wide water activity range, the BET model had more physical meaning. According to Chirife et al. (1992), the *k* value of the GAB equation higher than 1 is physically unacceptable, indicating infinite moisture sorption. Thereby, the BET monolayer is more reasonable for food stability determination than the GAB monolayer (RAHMAN and LABUZA, 2007).

Figure 26 - Moisture sorption isotherms of the pure açaí (black) and açaí-pectin (grey) powders produced by cast-tape drying (CTD).



Note: The filled symbols represent experimental data. The full lines represent the GAB model.

The monolayer moisture content (X_m) is an important parameter to predict the physical and chemical stability of dried food products since it represents the amount of water strongly adsorbed to specific polar sites on the food surface. This bound water is not available to participate in degradation (bio)chemical reactions (RAHMAN and LABUZA, 2007; TAOUKIS and RICHARDSON, 2007). The estimated X_m value of the BET model for the pure açaí powder was 0.011 g g^{-1} (d.b.), while it was 0.037 g g^{-1} (d.b.) for the açaí-pectin powder. The corresponding water activities to these X_m values were approximately 0.23 and 0.52 for the pure açaí and açaí-pectin powders, respectively. Frabetti et al. (2021) also reported an increase in the X_m value of the strawberry leather produced by CTD with the addition of pectin.

The C_{BET} value indicates how strong the bond is between the water and solid matrix (RAHMAN et al., 2009). Thus, the lower C_{BET} value for açaí-pectin powder indicated weak binding. C_{BET} values fall within the ranges reported in the literature for the types II (sigmoid shape) and III (J-shape) isotherms according to the classification proposed by Brunauer et al. (1940). The moisture sorption isotherm curve of the pure açaí powder corresponded to type II ($2 < C_{BET} < 50$). Pavan (2010) showed the same behavior for the pure açaí juice dried by freeze-drying, refractance window drying, and hot air drying. On the other hand, a type IIII isotherm was observed for açaí-pectin powder ($C_{BET} < 2$). Tonon et al. (2009b) also reported a type III isotherm for powdered açaí produced by spray-drying using filtered açaí juice with different carrier agents (maltodextrin 10DE, maltodextrin 20DE, gum Arabic, and tapioca starch).

Table 15 - Estimated BET and GAB parameters from the experimental data of the moisture sorption isotherms for pure açaí and açaí-pectin powders produced by cast-tape drying (CTD).

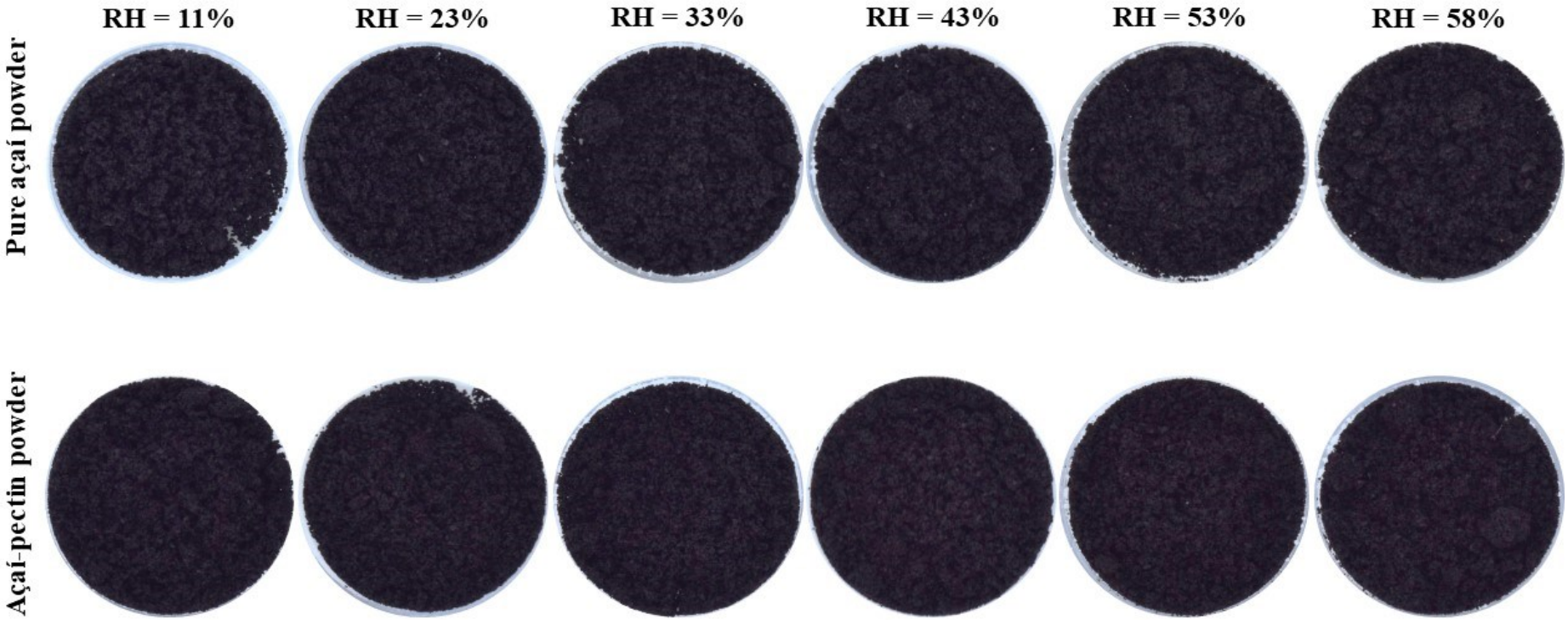
Model	Parameters	Powder samples	
		Pure açaí	Açaí-pectin
BET	X_m (g g^{-1} d.b.)	0.011	0.037
	C_{BET}	11.0	0.87
	R^2	0.999*	0.997**
GAB	X_m (g g^{-1} d.b.)	0.008	0.026
	C_{GAB}	20.0	1.23
	k	1.28	1.09
	R^2	0.999	0.997

Note: *BET model fitted till a_w = 0.529. **BET model fitted till a_w = 0.576.

Figure 27 shows some physical changes in the açaí powders stored at the different relative humidities at 25 °C. Small particle agglomeration commenced being observed in the

pure açaí powders stored from 11% RH. The oil on the powder surface may have contributed to this physical change. At the higher storage relative humidities, there was the formation of larger agglomerates for both powders. Agglomeration could hamper the powder flowability. In addition, a slight color change was visually notated for the açaí-pectin powder stored from 43% RH, in which samples seemed slightly purplish.

Figure 27 - Images of açaí powders stored at different relative humidities at 25 °C.



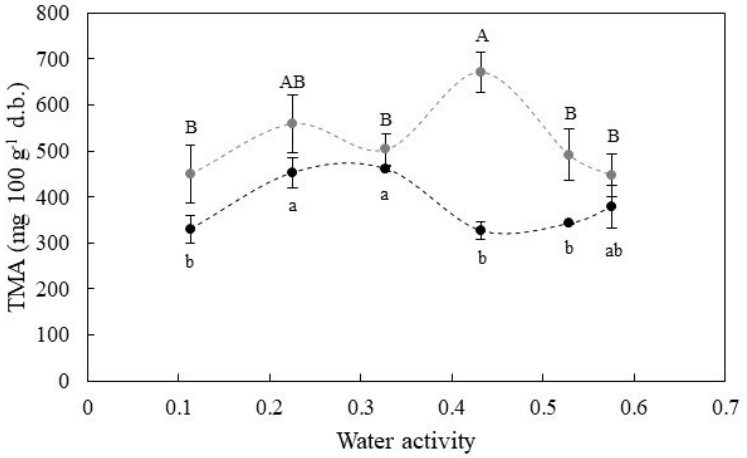
3.3.2 Total monomeric anthocyanin content

Anthocyanins are very prone to degradation during storage. The anthocyanin degradation may be caused by enzymatic reactions, non-enzymatic oxidation reactions (i.e., oxygen can react directly with anthocyanins and/or it can oxidize other compounds that react with anthocyanins), as well as by condensation reactions with other compounds (BUVÉ et al., 2018).

Figure 28 shows the total monomeric anthocyanin content determined for each sample stored at different relative humidities after açaí powders reached the equilibrium moisture content (60 days of storage). The anthocyanin content was highly affected by the storage water activity for both powder samples, and this influence depended on the powder composition. The higher anthocyanin content in the pure açaí powder was observed for water activities of 0.225 and 0.328, whereas in the açaí-pectin powder, it was greater at the water activity of 0.432. These water activities were close to correspondents to the monolayer. As expected, the anthocyanin content at the higher storage water activities was reduced, which may be associated with more water availability to participate in the degradation reactions or act as a vehicle that facilitates the diffusion of substrates involved in the reactions (MORAGA et al., 2012; SYAMALADEVI et al., 2011). Agudelo-Laverde, Schebor, and Buera (2013) reported that the anthocyanin degradation in freeze-dried strawberries stored at different relative humidities (11%-75%) was particularly important above 43% RH.

Nevertheless, in more dry environments (below monolayer moisture content), for example, at water activities of 0.113 and from 0.113 to 0.328 for pure açaí and açaí-pectin powders, respectively, the anthocyanin degradative reaction was also favored. Syamaladevi et al. (2011) evaluated the anthocyanin retention in freeze-dried raspberries in the rubbery state during storage at different relative humidities at 23 °C. The authors reported that the water activity corresponding to the monolayer moisture content was 0.23. After the sample achieved the equilibrium (37 days), the retention percentage of anthocyanins at this storage water activity was less than those observed for the sample stored at higher water activities (0.33-0.66). Conversely, at higher storage times (from 187 days), higher anthocyanin retention at the monolayer water activity was observed. According to Moraga et al. (2012), in the rubbery state of the amorphous matrix, a specific storage time is required for the reactants to contact each other.

Figure 28 - Total monomeric anthocyanin (TMA) content of the pure açaí (black) and açaí-pectin (grey) powders as a function of the storage water activity.

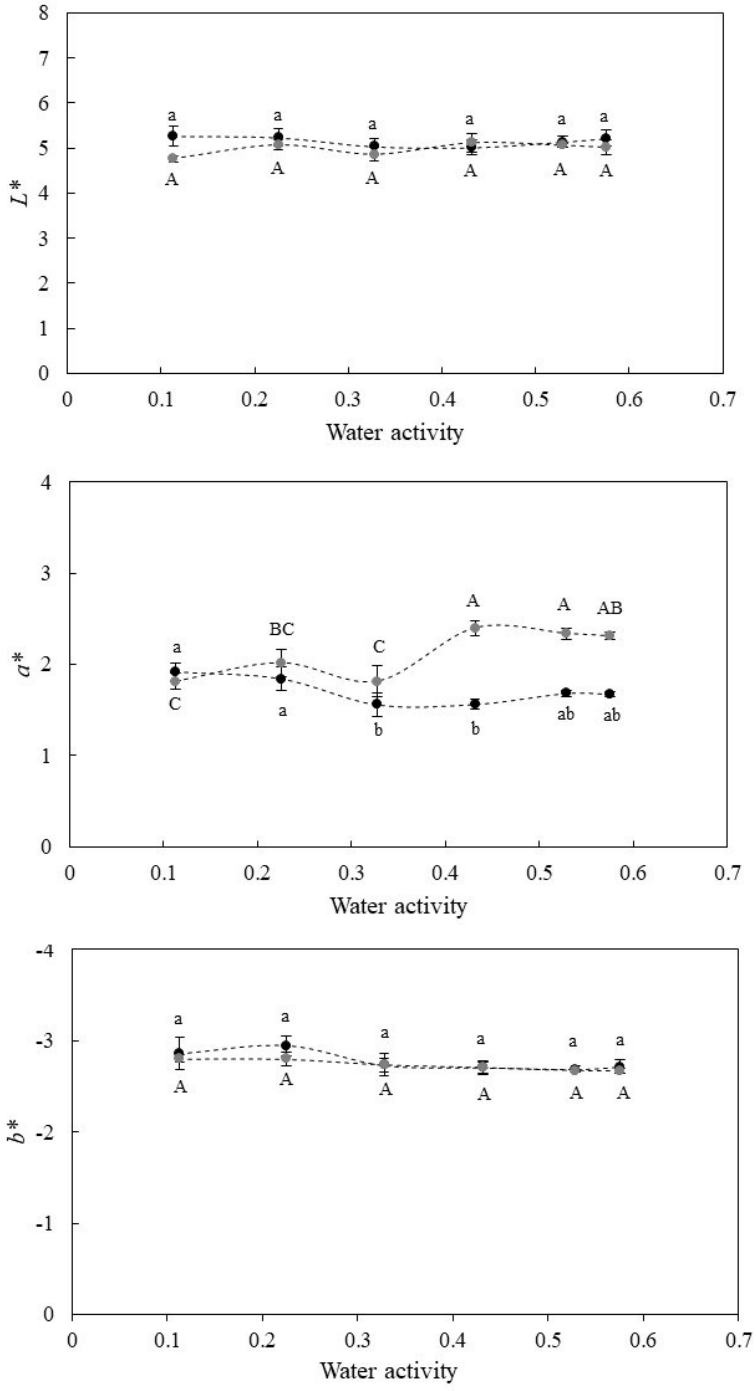


Note: The symbols represent the average of three measurements. The error bars represent the standard deviation of the experimental data (n=3). The same letters represent no significant difference ($p > 0.05$) between storage relative humidities, according to Tukey's test. The dashed lines were added to guide the eyes.

3.3.3 Color parameters

The color changes during the açaí powder storage can be attributed to enzymatic, non-enzymatic browning reactions and/or anthocyanin degradation. The CIELab color coordinates (L^*, a^*, and b^*) for the pure açaí and açaí-pectin powders equilibrated at different relative humidities are presented in Figure 29.

Figure 29 - Values of color parameters (L^*, a^*, and b^*) of the pure açaí (black) and açaí-pectin (grey) powders stored at different relative humidities.



Note: The symbols represent the average of three measurements. The error bars represent the standard deviation of the experimental data (n=3). The same letters represent no significant difference ($p > 0.05$) between storage relative humidities, according to Tukey's test. The dashed lines were added to guide the eyes.

For both samples, the luminosity (L^* value) and blueness (b^* value) were not significantly influenced by increasing water content. On the other hand, the redness (a^* value) increased at water activities above 0.432 for the açaí-pectin powder samples, which is consistent

with that visually perceived. This result may be associated with the dilution of browning reactants caused by increasing water content due to equilibration at higher relative humidity (MORAGA et al., 2011; VENIR et al., 2007).

3.3.4 Correlation between anthocyanin content and color parameters

An instrumental color attribute correlated to anthocyanin content is a useful tool for the food industry and academia due to the possible application of a non-destructive, simple, and less time-consuming analytical technique. Hence, a linear correlation analysis between the total monomeric anthocyanin content and color parameters of açaí powders equilibrated at different relative humidities was performed in order to find potential indicators of the chemical property (Table 16). Nevertheless, color parameters weakly correlated with the anthocyanin content of the açaí powders in the studied storage conditions, suggesting that these variables could not be used as an analytical indicator of anthocyanin levels in the açaí powders.

Table 16 - Correlation coefficients (*r*) between the total monomeric anthocyanin content and color parameters of açaí powders stored at different relative humidities.

Color parameters	**Total monomeric anthocyanin content**	
	Pure açaí powder	**Açaí-pectin powder**
*L**	-0.04	0.63
*a**	-0.08	0.39
*b**	-0.38	0.01

Note: Symbol (*) represents significant correlations at $p < 0.05$.

3.4 CONCLUSION

The GAB model well mathematically described the water adsorption of açaí powders with and without pectin dehydrated by the CTD process. However, the BET model showed more physical meaning. Low anthocyanin content in the açaí powder samples was observed at the storage water activities below the critical water activity corresponding to the BET monolayer. This result suggested that only monolayer moisture content was not enough to be considered a safe limit to ensure the anthocyanin stability of the açaí powders. Determining the physical state of the açaí powders (rubbery or glass) during storage can help better predict the samples' stability. Although the dark purple color of açaí juice is due to the high concentration of anthocyanins, the anthocyanin content weakly correlated with color parameters of açaí

powders stored at different relative humidities. Thereby, the anthocyanin degradation of the açaí powders was not the main factor responsible for color changes in stored samples.

GENERAL CONCLUSION

This thesis proposed the application of alternative drying techniques to produce açaí powder, aimed at elaborating high-quality powders with short drying times. The application of low drying pressures and the addition of hydrocolloids, in small amounts (3% w/w), to açaí juice resulted in açaí powders from the conductive thin-film drying processes (vacuum drum drying and cast-tape drying) having high anthocyanin retention and preservation of antioxidant activity. The powders were produced in a maximum of 32 min of drying. Nevertheless, conductive drying processes favored the formation of secondary lipid oxidation products (aldehydes) at a higher concentration than conventional methods (hot air drying and freeze-drying). Thereby, the stability of açaí powders to lipid oxidation during conductive drying processes is still a concern from a sensory and nutritional point of view. Moreover, the monolayer moisture contents estimated from moisture sorption isotherms were not enough to predict anthocyanin stability in the açaí powders during storage at different relative humidities.

SUGGESTIONS FOR FUTURE STUDIES

- Optimize the vacuum conductive thin-film drying process conditions based on the quality of the açaí powder.
- Study the effect of exhaustion/ventilation system on drying time, açaí powder quality, and energy efficiency during cast-tape drying.
- Evaluate the functional (such as dispersion time and solubility) and sensorial properties to obtain information on the rehydration behavior and consumers' acceptability of açaí powders produced by the conductive thin-film drying processes.
- Assess the benefits and/or risks for human health on consuming açaí powders.
- Investigate the impact of ascorbic acid addition on lipid oxidation of the conductive thin-film dried açaí powders.
- Determine the physical state of the açaí powders produced by the conductive drying processes during storage for better predicting the storage conditions.

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