UNIVERSIDADE FEDERAL DO PARÁ

INSTITUTO DE TECNOLOGIA

PROGRAMA DE PÓS-GRADUAÇÃO EM CIÊNCIA E TECNOLOGIA DE ALIMENTOS

ADRIANO LUCENA DE ARAÚJO

ESTUDO DO PROCESSO COMBINADO DE DESIDRATAÇÃO OSMÓTICA E SECAGEM CONVECTIVA PARA O JAMBOLÃO (*Syzygium cumini*)

BELÉM – PARÁ

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Tese de Doutorado apresentada ao Programa de Pós-Graduação em Ciência e Tecnologia de Alimentos da Universidade Federal do Pará, Instituto de Tecnologia, como um requisito para obtenção do título de Doutor em Ciência e Tecnologia de Alimentos.

Orientador: Prof. Dr. Rosinelson da Silva Pena

BELÉM - PA
2022

Dados Internacionais de Catalogação na Publicação (CIP) de acordo com ISBDSistema de Bibliotecas da Universidade Federal do Pará

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A658e Araújo, Adriano Lucena de.
Estudo do processo combinado de desidratação osmótica e secagem convectiva para o jambolão (Syzygium cumini) / Adriano Lucena de Araújo. — 2022.
xiv, 127 f. : il. color.

Orientador(a): Prof. Dr. Rosinelson da Silva Pena
Tese (Doutorado) - Universidade Federal do Pará, Instituto de Tecnologia, Programa de Pós-Graduação em Ciência e Tecnologia de Alimentos, Belém, 2022.

1. Desidratação osmótica. 2. Secagem convectiva. 3. Isotermas de sorção de umidade. 4. Compostos bioativos. 5. Planejamento experimental. I. Título.

CDD 664.0284

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Data da Avaliação: ____/____/____

Nota/Conceito: _________________

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À Deus, aos meus pais Edinaldo e Silvana, ao meu irmão André e à Dona Sissinha, aos meus amigos e familiares que sempre estiveram comigo e acompanharam a minha jornada, dedico com carinho.

AGRADECIMENTOS

À **Deus**, que me concedeu o dom da vida e me guiou nos momentos difíceis.

Aos meus pais, **Edinaldo** e **Silvana**, por seus constantes cuidados. Nada seria possível sem o auxílio emocional, psicológico e financeiro de vocês. Nunca esquecerei de todo o amor e empenho que vocês depositaram em mim. Eu não seria nada sem vocês.

Ao meu irmão **André**, que sempre me inspirou e motivou a alcançar meus objetivos. À **Dorothy**, por ser esse exemplo de pessoa íntegra. Agradeço por ter investido no meu estudo, pois me ajudou bastante a concretizar as minhas metas profissionais.

Ao **Bruno Malato**, pelo companheirismo, pelo afeto e pelas inúmeras conversas construtivas que tivemos, pois, através delas, pude amadurecer em diversos aspectos.

Ao meu amigo **Fábio Farias**, pelo constante cuidado e irmandade durante todos esses anos. Por transmitir a mim o verdadeiro poder da amizade, da cumplicidade e da fé.

Ao Programa de Pós-Graduação em Ciência e Tecnologia de Alimentos (**PPGCTA**), da Universidade Federal do Pará (**UFPA**).

À Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (**CAPES**), pela concessão da bolsa de estudos.

Ao Prof. Dr**. Rosinelson da Silva Pena**, por ter confiado a mim o desenvolvimento da pesquisa. Agradeço imensamente pelas correções e contribuições, pois impulsionaram a melhorar a qualidade do trabalho escrito.

Aos membros da banca examinadora, Profª. Drª. **Alessandra Santos Lopes**, Prof. Dr. **Renan Campos Chisté**, Prof. Dr. **Lênio José Guerreiro de Faria** e Prof. Dr**. Eder Augusto Furtado Araujo**, pelas valiosas contribuições e sugestões.

Ao laboratório de **Tecnologia de Alimentos**, pelo suporte técnico.

Aos membros do LAFAMI: **Juliana do Carmo**, **Estela Cruz**, **Tatyane Myllena**, **Ana Paula Campos**, **Joseane Pombo**, **Rosane Patrícia**, **Mayara Galvão**, **Adriano Rondinelli**, **Brenda Brito**, **Elivaldo Modesto**, **Fernanda Rafaele** e **Gisélia de Sousa**, pela amizade, apoio e, principalmente, pelas inúmeras lembranças que guardarei com muito carinho. Sem dúvidas, o convívio diário com vocês tornava o meu dia mais colorido. Agradeço, em especial, à **Jaqueline de Fátima** pelo companheirismo e carinho.

Agradeço a todos que, de alguma forma, contribuíram para a realização deste trabalho.

RESUMO

Os frutos do jambolão (*Syzygium cumini*) são *berries* com formato elipsoide, que pertencem à família *Myrtaceae* e se caracterizam por possuírem uma polpa carnosa, que envolve uma única semente. A casca do fruto possui uma fina película, que exibe a coloração roxo intenso no estádio final de maturação, devido a presença de antocianinas. Estudos recentes têm avaliado a ação das principais atividades biológicas envolvendo as diversas partes do jamboleiro, as quais têm apontado a presença de potenciais anti-inflamatório e antidiabético. Apesar do jambolão apresentar importantes fitoquímicos na composição, o fruto é pouco conhecido pela população brasileira e, por essa razão, seu consumo é pouco expressivo, durante o curto período de safra (de 30 a 40 dias). Assim, visando aproveitar o potencial tecnológico do jambolão e prolongar a vida de prateleira do fruto, que é muito perecível, o objetivo desta pesquisa foi estudar o processo combinado de desidratação osmótica (DO) e secagem convectiva do jambolão, de modo a estender a sua vida útil e agregar valor ao fruto. Para isso, um estudo inicial foi realizado para avaliar a composição centesimal da polpa e da semente do fruto e estudar a estabilidade destas frações durante armazenamento, através de isotermas de dessorção de umidade (25-55ºC), a partir das quais foram estimadas as demandas energéticas (propriedades termodinâmicas) exigidas para a desidratação do fruto. Em seguida, foram realizadas a caracterização físico-química e biométrica dos frutos e avaliado o efeito das principais variáveis envolvidas na DO do jambolão, por meio de um planejamento fatorial fracionado 2^{6-2}. Por fim, foi avaliado o efeito da DO sobre a cinética de secagem dos frutos a 50, 60 e 70ºC; em função dos teores de antocianinas monoméricas e de compostos fenólicos. Os resultados indicaram que os níveis de umidade que garantem a maior estabilidade degradativa à polpa e à semente do jambolão, durante o armazenamento, são 8 g H_2O/100 g (base seca - bs) e 5,1 g H_2O/100 g bs, respectivamente. Foi observado também que as energias envolvidas no processo de desidratação da polpa foram maiores do que da semente. A análise biométrica evidenciou que, a polpa e a casca do jambolão, contribuem em mais de 70% do peso do fruto e a análise de composição indicou quantidades promissoras de antocianinas (147 ± 1.85 mg.100^{-1} g) e de compostos fenólicos (391 ± 18.89 mg GAE.100^{-1} g). A seleção de variáveis, para o processo de DO do jambolão, evidenciou que a temperatura, a concentração de sacarose, a concentração de lactato de cálcio e a pressão apresentaram efeitos significativos sobre a resposta perda de água, a qual é de grande importância na DO. Por outro lado, foram observadas perdas de antocianinas monoméricas (46,6%) e de compostos fenólicos (26,9%), após 72 horas de DO. Finalmente, a pesquisa mostrou que os frutos submetidos a DO demandaram menores tempos de secagem, do que os frutos não submetidos a este pré-tratamento. Estes resultados indicam que a combinação da DO com a secagem convectiva é uma alternativa tecnológica relevante para a conservação do fruto do jambolão.

Palavras-chave: bioma amazônico; ameixa-do-Pará; desidratação; processamento; compostos bioativos.

ABSTRACT

Jambolan (*Syzygium cumini*) fruits are ellipsoid-shaped berries that belong to the *Myrtaceae* family and present a fleshy pulp, which involves a single seed. The fruit peel has a thin film that displays an intense purple color in the final stage of maturation, due to the presence of anthocyanins. Recent studies have evaluated the action of the main biological activities involving the different parts of the jambolan tree, which have indicated the presence of anti-inflammatory and antidiabetic potentials. Although jambolan has important phytochemicals in its composition, the fruit is little known by the Brazilian population and, for this reason, its consumption is not very expressive during the short harvest period (30 to 40 days). Thus, in order to take advantage of the technological potential of jambolan and prolong the shelf life of the fruit, which is very perishable, the objective of this research was to study the combined process of osmotic dehydration (OD) and convective drying of jambolan, in order to extend its expiry date and add value to the fruit. For this, an initial study was carried out to evaluate the proximate composition of the fruit's pulp and seed and to study the stability of these fractions during storage, through moisture desorption isotherms (25-55ºC), where the energy demands (thermodynamic properties) were estimated, which are required for the dehydration of the fruit. Then, the physical-chemical and biometric characterization of the fruits were carried out and the effect of the main variables, involved in the OD of jambolan, was evaluated through a fractional factorial design 2^{6-2}. Finally, the effect of OD on the drying kinetics of fruits at 50, 60 and 70ºC was evaluated; depending on the content of monomeric anthocyanins and phenolic compounds. The results indicated that the moisture levels that guarantee the greatest degradative stability to the jambolan pulp and seed during storage are 8 g H_2O/100 g (dry basis - db) and 5.1 g H_2O/100 g db, respectively. It was also observed that the energies involved dehydration process were higher for pulp than seed. The biometric analysis showed that the jambolan pulp and peel contribute with more than 70% of the fruit weight and the composition analysis indicated promising amounts of anthocyanins (147 ± 1.85 mg.100^{-1} g) and phenolic compounds (391 ± 18.89 mg GAE.100^{-1} g). The selection of variables for the jambolan OD process showed that temperature, sucrose concentration, calcium lactate concentration and pressure had significant effects on the water loss response, which is of great importance in OD. On the other hand, losses of monomeric anthocyanins (46.6%) and phenolic compounds (26.9%) were observed after 72 hours of OD. Finally, the research showed that the fruits submitted to OD required shorter drying times than the fruits not submitted to this pre-treatment. These results indicate that the combination of OD and convective drying is a relevant technological alternative for the conservation of jambolan fruit.

Keywords: Amazon biome; Pará plum; dehydration; processing; bioactive compounds.

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ORGANIZAÇÃO DA TESE

O estudo foi organizado em diferentes capítulos, os quais se encontram descritos abaixo:

- CAPÍTULO I: **Introdução Geral e Objetivos**: neste capítulo foram apresentadas as justificativas do estudo, bem como os objetivos geral e específicos da tese.
- CAPÍTULO II**: Referencial teórico**: neste capítulo foram abordadas as fundamentações teóricas dos principais temas relacionados à tese. Dentre eles, foram descritas as características gerais do jambolão (*Syzygium cumini*), bem como o seu potencial nutricional e tecnológico. Além disso, foram abordados os seguintes temas: desidratação osmótica, secagem convectiva e isotermas de sorção de umidade.
- CAPÍTULO III: **Artigo 1 - Moisture desorption behavior and thermodynamic properties of pulp and seed of jambolan (*Syzygium cumini*):** neste capítulo se encontra o primeiro artigo da tese que é uma pesquisa original publicada no periódico Heliyon. A pesquisa avaliou o comportamento higroscópico da polpa e da semente do jambolão, visando não apenas estabelecer condições de armazenamento do fruto, mas principalmente expressar as demandas energéticas requeridas no processo de desidratação/secagem, por meio do cálculo das propriedades termodinâmicas de dessorção de umidade.
- CAPÍTULO IV: **Artigo 2 - Influence of process conditions on the mass transfer of osmotically dehydrated jambolan fruits**: este capítulo é um artigo original e o segundo da tese. Esta pesquisa já se encontra publicada no periódico Food Science and Technology (Campinas) e investigou a influência de variáveis inerentes ao processo de desidratação osmótica do jambolão, verificando os principais efeitos dos fatores de entrada sobre as respostas relacionadas com a transferência de massa.
- CAPÍTULO V: **Artigo 3 - Combined osmotic dehydration and convective air-drying process of jambolan fruits**: este capítulo corresponde a um estudo original que será submetido ao periódico LWT – Food Science and Technology e contempla o terceiro artigo da tese. Neste estudo a desidratação osmótica (DO) foi avaliada como um pré-tratamento para o processo de secagem do jambolão. Foi avaliada a secagem dos frutos do jambolão, com e sem a aplicação da DO.
- CAPÍTULO VI: **Conclusão geral**: Neste capítulo são abordadas as principais conclusões do estudo.

SUMÁRIO

Capítulo I

1 INTRODUÇÃO GERAL

O jambolão (*Syzygium cumini*) é uma fonte promissora de compostos bioativos, especialmente antocianinas, que conferem à casca do fruto a coloração roxa (PANGHAL et al., 2018). O *Syzygium cumini* é bastante difundido em função de suas propriedades medicinais e terapêuticas (CHHIKARA et al., 2018). Estudos recentes apontaram a presença de várias atividades biológicas envolvendo os extratos da polpa, folha e sementes do fruto, sendo que os principais benefícios à saúde incluíam: o potencial antimicrobiano, antioxidante, anti-proliferativo, anti-diabético e anti-inflamatório (PAVAN KUMAR et al., 2010; TUPE et al., 2015; AQIL et al., 2016; SINGH et al., 2016; SANTOS et al., 2020).

O jambolão é um fruto oriundo do continente Asiático e, atualmente, encontra-se difundido pelas regiões tropicais e/ou subtropicais da Austrália, Quênia, Filipinas e Estados Unidos. Os frutos da espécie são elipsoides, medem de 1,5 a 3 cm de comprimento e possuem uma polpa carnosa, que envolve uma única semente. Embora o jambolão seja comumente encontrado no território brasileiro, o consumo de seus frutos é escasso e a planta é pouco conhecida pela população em geral. Além de possuir uma safra muito curta (de 30 a 40 dias), o fruto sofre grandes perdas durante a pós-colheita, devido à natureza perecível. Por esta razão, o jambolão só pode ser armazenado por poucos dias (1-2 dias) à temperatura ambiente (LOGUERCIO et al., 2005; MANJESHWAR et al., 2013; SINGH et al., 2015; GHOSH et al., 2017) e, portanto, carece de tecnologias eficazes para a sua conservação.

Visando estender a vida útil do jambolão, a desidratação osmótica (DO), que se baseia na remoção parcial de água através da imersão do fruto em soluções hipertônicas, surge como uma importante alternativa. A diferença de potencial químico entre o tecido vegetal e a solução circundante possibilita a retirada de água do fruto, a incorporação de sólidos ao mesmo e a transferência de solutos (ácidos orgânicos, minerais, entre outros) do produto para a solução. Embora esta última transferência não cause uma perda de massa quantitativamente significativa, ela é essencial para a qualidade final do produto (TORREGGIANI, 1993; ABRAAO et al., 2013).

A DO tem várias aplicações na indústria de processamento de frutas e hortaliças e se apresenta como uma opção econômica e segura para a preservação de frutos. Porém, a DO não assegura ao produto um nível de umidade que garanta a sua estabilidade durante o armazenamento. Assim, para assegurar a estabilidade do produto, a DO deve ser combinada com outros métodos de conservação, como a secagem (LAZARIDES, 2001).

A secagem convectiva é um dos métodos de secagem mais utilizados, apesar do consumo alto de energia. A incorporação de tecnologias emergentes, aliada a práticas de secagem tradicionais, tem sido apontada para diminuir efetivamente o tempo total de secagem. Assim, quando combinada com algum pré-tratamento (ex. DO), pode promover mudanças físicas e/ou químicas benéficas, bem como melhorar a eficiência do transporte de massa (ROSS et al., 2020).

Diversos autores têm destacado a utilização de técnicas combinadas de DO e secagem para a preservação de frutos (BADWAIK et al., 2013; SAHIN; ÖZTÜRK, 2016; ASSIS; MORAIS; MORAIS, 2017; CORRÊA et al., 2017; JUNQUEIRA; CORRÊA; ERNESTO, 2017; CORRÊA et al., 2021; PAVKOV et al., 2021), uma vez que o emprego dessas tecnologias promove a redução da umidade e minimiza a deterioração microbiana e a velocidade de reações químicas indesejáveis ao produto (SERENO; MOREIRA; MARTINEZ, 2001).

Poucos trabalhos abordaram a influência da DO, combinada com a secagem, sobre a degradação de compostos bioativos de frutos. Ademais, a escassez de pesquisas que relatam o efeito da DO sobre as propriedades funcionais de alimentos é ainda mais preeminente que a própria secagem, uma vez que essas pesquisas focam, principalmente, nas cinéticas do processo em termos de perda de água e ganho de sólidos (LANDIM, BARBOSA, BARBOSA JÚNIOR, 2016).

Outra ferramenta importante para os produtos submetidos à desidratação e secagem é a avaliação higroscópica, realizada a partir das isotermas de sorção de umidade. Este estudo auxilia na solução de problemas relacionados com a embalagem, o armazenamento, o transporte, a predição da vida de prateleira, o controle de processos, bem como para o cálculo das demandas energéticas de produtos submetidos à desidratação/secagem, através do estudo das propriedades termodinâmicas (NOSHAD et al., 2012).

As propriedades termodinâmicas de dessorção de umidade podem estimar a quantidade mínima de energia requerida por um processo de secagem, além de dar informações sobre o estado da água no produto e inferir, de maneira aproximada, sobre a microestrutura e as mudanças físicas que acontecem na superfície do material (AGUERRE; SUAREZ; VIOLAZZ, 1986). Propriedades como entalpia/entropia diferenciais, energia livre de Gibbs e a teoria da compensação entalpia-entropia são consonantes neste processo e recentemente foram calculadas para uma mistura em pó de polpa e semente de jambolão (PAUL; DAS, 2019). Todavia, nenhum estudo contemplou, de maneira separada, as propriedades termodinâmicas do fruto e da semente do jambolão *in natura*.

Neste sentido, novas abordagens são necessárias para prever o consumo energético do fruto fresco durante a etapa de desidratação. Ressalta-se ainda, a necessidade de ampliar o conhecimento das variáveis que realmente apresentam um efeito significativo sobre a DO do jambolão e, somado a isto, está a falta de estudos que incorporam técnicas combinadas de DO com secagem convectiva para o fruto, uma vez que o estudo mencionado (SHARMA; DAS, 2019) não abordou essa temática. É importante ressaltar que o jambolão é uma fonte potencial de compostos bioativos e possui uma safra muito curta, o que motivou a pesquisa.

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2 OBJETIVOS

2.1 OBJETIVO GERAL

Estudar o processo combinado de desidratação osmótica e secagem convectiva do jambolão (*Syzygium cumini*), visando o aproveitamento tecnológico do fruto.

2.2 OBJETIVOS ESPECÍFICOS

- **Capítulo III**

- Realizar a caracterização centesimal da polpa e da semente do jambolão (*Syzygium cumini*);
- Obter as isotermas de dessorção de umidade a 25, 35, 45 e 55°C para a polpa e semente do jambolão;
- Identificar modelos matemáticos capazes de predizer, com boa precisão, os dados de dessorção de umidade da polpa e semente do jambolão;
- Calcular o comportamento das propriedades termodinâmicas de dessorção de umidade da polpa e da semente do jambolão: entalpia/entropia diferencias, energia livre de Gibbs e a teoria da compensação entalpia/entropia.

- **Capítulo IV**

- Realizar a análise biométrica nos frutos do jambolão (*Syzygium cumini*);
- Realizar a caracterização centesimal e físico-química do jambolão;
- Aplicar um planejamento fatorial fracionário do tipo 2^{6-2} para identificar entre as variáveis temperatura, concentração de sacarose, pressão, tempo de pulso de vácuo, concentração de lactato de cálcio e número de pulsos de vácuo, as que influenciam no processo de desidratação osmótica do jambolão.

- **Capítulo V**

- Estudar a desidratação osmótica do jambolão (*Syzygium cumini*) a 25ºC;
- Estudar a secagem do jambolão sem tratamento osmótico e com tratamento osmótico, nas temperaturas de 50, 60 e 70ºC;
- Avaliar o ajuste de modelos matemáticos aos dados de desidratação osmótica e secagem do jambolão;
- Avaliar o efeito da desidratação osmótica e da secagem convectiva sobre compostos bioativos do jambolão.

Capítulo II

3 REFERENCIAL TEÓRICO

3.1 CARACTERÍSTICAS GERAIS DO JAMBOLÃO

O jambolão (*Syzygium cumini*) é um fruto originário da Ásia tropical, pertencente à família *Myrtaceae*, que engloba 121 gêneros e de 3800 a 5800 espécies, difundidas pelas regiões tropicais e subtropicais do mundo. No Brasil, as principais espécies da *Myrtaceae* são: a jabuticaba (*Myrciaria cauliflora* (Mart.) O. Berg), o camu-camu (*Myrciaria dubia* Mc. Vaugh), a cagaita (*Eugenia dysenterica* DC), o cambuci (*Campomanesia phaea* (O. Berg.) Landrum) e a pitanga (*Eugenia uniflora*) (CARVALHO et al., 2017). Os frutos do jambolão (Figura 1) são *berries* de formato oval, que medem de 2 a 3 centímetros de comprimento e são revestidos por uma casca fina de coloração roxo escuro, uma polpa praticamente incolor, com uma única semente; semelhantes ao mirtilo no formato e cor (BENHERLAL; ARUMUGHAN, 2007).

Figura 1. Frutos do jambolão.

No Brasil, o fruto inicia sua floração de setembro a novembro, com posterior maturação entre os meses de dezembro a fevereiro. Os frutos (quatro a vinte) ficam dispostos em cachos que não amadurecem simultaneamente e, quando imaturos, são de coloração verde. À medida em que os frutos amadurecem, eles adquirem a cor magenta claro e, finalmente, a cor roxa escura, quando em estádio final de maturação. Os frutos são não-climatéricos e, por esta razão, é necessário que se atinja o tempo completo de amadurecimento para a colheita. Ademais, a síntese dos compostos que ocorre durante a maturação confere ao fruto impactos marcantes no aroma, cor e *flavour*, como resultado da associação entre açúcares, ácidos orgânicos e compostos fenólicos. Os taninos, por sua vez, conferem adstringência à polpa e, durante a maturação, a associação dos taninos com os açúcares reduz a intensidade da adstringência. Quando não colhidos, os frutos maduros alimentam morcegos, esquilos e macacos (WARRIER; NAMBIAR; RAMANKUTTY, 1996; SABINO; BRITO; SILVA JÚNIOR, 2018), além de insetos; porém, muitos frutos maduros que caem ao chão, sofrem danos físicos e se deterioram.

O jambolão não apresenta elevada doçura, devido à elevada concentração de maltose. A acidez do fruto é atribuída, principalmente, à presença dos ácidos málico, cítrico e gálico. Os compostos fenólicos geram compostos voláteis responsáveis pelo aroma do fruto (AYYANAR; SUBASH-BABU, 2012). Em relação à composição de minerais, a polpa do fruto contribui com 5,93 g/100 g matéria seca - MS; sendo os principais minerais: o potássio (3,8 g/100 g MS), o cálcio (1,04 g/100 g MS), o magnésio (0,78 g/100 g MS) e o sódio (0,31 g/100 g MS) (SERAGLIO et al., 2018). O jambolão apresenta uma complexa composição química, constituída por diferentes compostos de valor nutricional e biológico. A composição centesimal do jambolão é apresentada na Tabela 1.

Tabela 1. Composição centesimal do jambolão.

Composição*	**Brito et al. (2017)**	**Albuquerque et al. (2019)**	**Santos et al. (2020)**	**Araújo e Pena (2022a)**	**Araújo e Pena (2022b)**
Carboidratos	11,40	nd	8,52	13,90	14,40
Umidade	87,20	89,43	83,51	84,62	84,76
Cinzas	0,23	0,27	1,38	0,43	0,29
Lipídeos	0,49	0,23	0,97	0,56	0,34
Proteínas	0,85	0,72	5,62	0,48	0,47

*****[g/100 g – base úmida (bu)]; nd: não determinado.

O jambolão é considerado um fruto perecível, em especial pela fragilidade da polpa e do pericarpo, que oferecem pouca proteção contra danos mecânicos e agentes infecciosos. Assim, o manejo na colheita e pós-colheita é essencial para a extensão da vida útil do fruto, visto que em condições ambientes, a vida de prateleira do jambolão não ultrapassa dois dias. Em função dos aspectos nutricionais e biológicos, existe um grande potencial para a industrialização do jambolão nos setores alimentícios e/ou farmacêuticos. Entretanto, observa-se que, independente da região produtora, a falta de investimentos em tecnologias pós-colheita resulta em altas taxas de resíduo, o que gera a necessidade de incentivos tanto para o cultivo, quanto para a comercialização do fruto (SABINO; BRITO; SILVA JÚNIOR, 2018). Neste contexto, algumas pesquisas têm se concentrado em aproveitar as potencialidades do jambolão (Tabela 2).

Tabela 2. Resumo das pesquisas que envolvem o aproveitamento do jambolão.

Produto	**Principais observações**	**Referência**
Jambolão atomizado	Altas temperaturas no ar de entrada do atomizador proporcionaram o aumento da umidade e, por conseguinte, o aumento da higroscopicidade dos pós atomizados. Ademais, as melhores condições de processamento e das características de cor do produto foram as que utilizaram a temperatura de ar de entrada entre 140 a 150ºC.	Santhalakshmy et al. (2015)
Resíduo de jambolão seco em leito de jorro	Apesar da redução no teor de antocianinas, as amostras secas mantiveram a atividade antioxidante, independente dos valores das variáveis de processo: temperatura (70 e 80ºC) e velocidade (8 e 10 m/s) do ar de secagem.	Mussi et al. (2015)
Chappati suplementado de jambolão liofilizado	A adição de jambolão liofilizado à formulação de *chappati* (pão) proporcionou uma maior quantidade de compostos bioativos ao produto, em comparação com o jambolão em pó obtido por secagem convencional.	Kapoor, Ranote e Sharma (2015)
Jambolão seco em leito de espuma	A elevação da temperatura do processo afetou negativamente o teor de antocianinas totais. Os flavonóis e os taninos hidrolisáveis foram mais afetados pela oxidação e tempo de aquecimento, respectivamente, que pela temperatura.	Carvalho et al. (2017)
Jambolão osmoticamente desidratado	O uso de ultrassom e pressão de vácuo permitiu maior perda de água e ganho de sólidos no fruto, em comparação com tratamentos de desidratação osmótica (DO) à pressão atmosférica ou com pulso de vácuo, apenas.	Sharma e Das (2019)
Jambolão atomizado	As condições ótimas do processo se estabeleceram quando a concentração de maltodextrina foi igual a 10% e a temperatura do ar de entrada do atomizador foi de 185ºC. O produto otimizado apresentou características nutricionais e funcionais promissoras, além de apresentar baixa higroscopicidade.	Singh, Paswan e Rai (2019)
Suco misto de jambolão e camu-camu	O estudo demonstrou que uma formulação com 12,5% de jambolão, 37,5% de camu-camu e 50% de água, manteve a estabilidade das antocianinas monoméricas até cinco dias de armazenamento a 25ºC.	Campos, Chisté e Pena (2021)
Jambolão osmoticamente desidratado	O estudo permitiu a triagem das variáveis influentes no processo de DO e os fatores com efeito significativo sobre a resposta perda de água, foram: temperatura, concentração de sacarose, concentração de lactato de cálcio e pressão absoluta.	Araújo e Pena (2022a)
Jambolão liofilizado	Durante o armazenamento dos pós do jambolão, liofilizados em diferentes estádios de maturação, notou-se que embalagens a base de filmes metalizados proporcionaram maior proteção à perda de cor, além de apresentar maior barreira contra o vapor de água, se comparada com embalagem transparente. A redução de antociacinas durante o armazenamento foi de 44% em embalagens metalizadas e de 56% em filmes transparentes.	Mussi e Pereira (2022)

3.2 COMPOSTOS BIOATIVOS

Os compostos bioativos são fitoquímicos encontrados em frutas, vegetais e grãos, capazes de modular os processos metabólicos e atuar na promoção da saúde. Eles exibem efeitos benéficos, como atividade antioxidante, inibição ou indução de enzimas, inibição de receptores de atividade e indução ou inibição da expressão gênica (CORREIA et al., 2012). A atividade antioxidante dos compostos bioativos de frutos está relacionada com teores de antocianinas, compostos fenólicos, carotenoides, ácido ascórbico e vitaminas, dentre outros (ALMEIDA et al., 2011).

A coloração intensa da casca do jambolão, por exemplo, é associada ao alto teor de antocianinas, que é maior que em outros frutos pertencentes à mesma família, como: a jabuticaba, o camu-camu e a pitanga (COSTA et al., 2013). Em relação ao perfil de antocianinas, estudos têm reportado a presença de delfinidina, cianidina, petunidina, peonidina e malvidina na polpa e na casca do jambolão. Ao longo do amadurecimento do fruto, a delfinidina é a antocianina predominante (LESTARIO et al., 2017). A Tabela 3 expressa os principais compostos bioativos encontrados no jambolão *in natura*.

Tabela 3. Teores dos compostos bioativos do jambolão i*n natura*.

Composto bioativo	**Faria, Marques e Mercadante (2011)**	**Ghosh et al. (2017)**
Fenólicos totais (mg eq. ácido gálico/100 g)	148,3	203,76
Flavonoides totais (mg eq. catequina/100 g)	91,2	Nd
Antocianinas monoméricas (mg cianidina-3-glicosídio/100 g)	210,9	195,58
Taninos (mg eq. ácido tânico /100g)	3,9	94,52
Ácido ascórbico (mg/100 g)	< 0,01	49,78
Carotenoides totais (µg/100 g)	89,2	Nd

nd: não determinado.

Os compostos bioativos presentes no jambolão têm atraído a atenção da comunidade científica e da indústria de alimentos, principalmente pela possibilidade de associar a ingestão do fruto à vários benefícios à saúde, o que torna possível a utilização do mesmo como uma fonte potencial no desenvolvimento de novos produtos (CARVALHO et al., 2017). Na medicina tradicional, os frutos, as folhas e as sementes do jambolão são utilizados em função das propriedades funcionais que desempenham, pois apresentam ação antioxidante, antimutagênica e antimicrobiana, dentre outras (SANTOS et al., 2020).

3.2.1 Compostos fenólicos

Os compostos fenólicos são estruturas químicas caracterizadas pela presença de hidroxilas e anéis aromáticos, nas formas simples ou poliméricas, naturais ou sintéticas. Quando presentes em vegetais podem estar na forma livre ou complexada com açúcares e proteínas. Os principais grupos de compostos fenólicos são: os flavonoides, os ácidos fenólicos, os taninos e os tocoferóis, como os antioxidantes fenólicos mais comuns de fontes naturais (CORRÊA et al., 2015a).

Os flavonoides e os ácidos fenólicos são os compostos fenólicos comumente encontrados em frutas e se apresentam sob a forma de conjugados solúveis em água (glicosídeos) e formas insolúveis. Os ácidos fenólicos são evidenciados na natureza nas formas insolúveis ou conjugadas, ao passo que os flavonoides exibem a forma glicosilada, com um ou mais açúcares ligados através de um grupo hidroxila (OH-glicosídeo) ou através de ligações carbono-carbono (C-glicosídeo) (ACOSTA-ESTRADA; GUTIÉRREZ-URIBE; SERNA-SALDÍVAR, 2014).

Os flavonoides se apresentam como antioxidantes, capazes de inibir ou reduzir a ação de radicais livres. Os radicais livres são vinculados a patologias de diversas doenças humanas, pois aumentam o estresse oxidativo, e potencialmente, podem danificar moléculas biológicas como proteínas, carboidratos, lipídeos e DNA. Assim, os compostos biologicamente ativos têm demostrado efeitos positivos sobre doenças crônicas, tais como as doenças cardiovasculares, câncer e diabetes (HERNÁNDEZ-RODRÍGUEZ; BAQUERO; LARROTA, 2019).

Em produtos naturais, grande parte dos compostos responsáveis pela coloração pertence à classe dos flavonoides. Os flavonoides possuem a estrutura evidenciada pela presença de um esqueleto com 15 átomos de carbono, divididos em classes, dependendo do estado de oxidação do anel central de pirano (MARÇO; POPPI; SCARMINIO, 2008). A Figura 2 apresenta a estrutura química das principais subclasses de flavonoides.

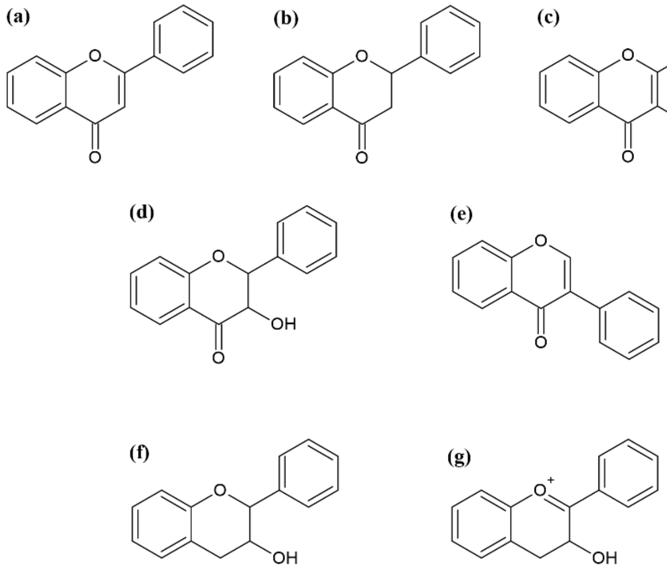

Figura 2. Estrutura dos flavonoides. **(a)** Flavona, **(b)** Flavanona, **(c)** Flavonol, **(d)** Flavanonol, **(e)** Isoflavona, **(f)** Flavanol, **(g)** Antocianidina.

Fonte: adaptado de Xiao e Ho (2017).

3.2.2 Antocianinas

As duas classes de flavonoides consideradas como as mais importantes são os flavonóis e as antocianidinas. As antocianidinas não possuem grupamentos glicosídicos e a maioria possui hidroxilas nas posições 3, 5 e 7. As antocianinas são pigmentos derivados das antocianidinas que apresentam uma ou mais hidroxilas ligadas a açúcares, sendo as mais comuns a glicose, a xilose, a arabinose, a raminose, a galactose ou dissacarídeos constituídos por estes açúcares, que podem estar ligados a ácidos fenólicos. O açúcar presente nas moléculas de antocianinas confere maior solubilidade e estabilidade a estes pigmentos, quando comparados com as antocianidinas (SWANSON, 2003).

A estabilidade química das antocianinas tem sido o foco de muitos estudos recentes e este conhecimento pode ser utilizado para minimizar a sua degradação, por meio da seleção adequada de processos. As principais variáveis que influenciam na degradação das antocianinas estão relacionadas com fatores intrínsecos, como a estrutura química e a copigmentação intramolecular; e também a fatores extrínsecos, com o pH, a temperatura, a presença de enzimas degradativas, o ácido ascórbico, o dióxido de enxofre, íons metálicos e açúcares (HE et al., 2015).

A temperatura é um dos principais fatores que influenciam na estabilidade das antocianinas. A degradação pelo calor não é dependente apenas da temperatura, mas também do tempo de processo, sendo causada, principalmente, pela oxidação e clivagem das ligações covalentes. O mecanismo da degradação térmica das antocianinas ainda não foi totalmente elucidado; porém, três vias principais têm sido propostas e estão esquematizadas na Figura 3. Na primeira via (A), o cátion flavilium se transforma em base quinoidal e, posteriormente, transforma-se em vários intermediários, que resultam em derivados da cumarina e em um composto equivalente ao anel B. Na segunda via (B), o cátion flavilium se transforma em base carbinol incolor e, posteriormente, transforma-se em chalcona, que resulta em produtos de degradação de cor marrom. A terceira via (C) ocorre de maneira semelhante ao mecanismo (B); no entanto, ocorre, primeiramente, a desglicolisação da molécula. Deste modo, a hidrólise da antocianina resulta na perda da ligação glicosídica e na formação de antocianinas instáveis, nos quais os produtos de degradação expressam as cores marrom e amarelo (SCHWARTZ et al., 2017).

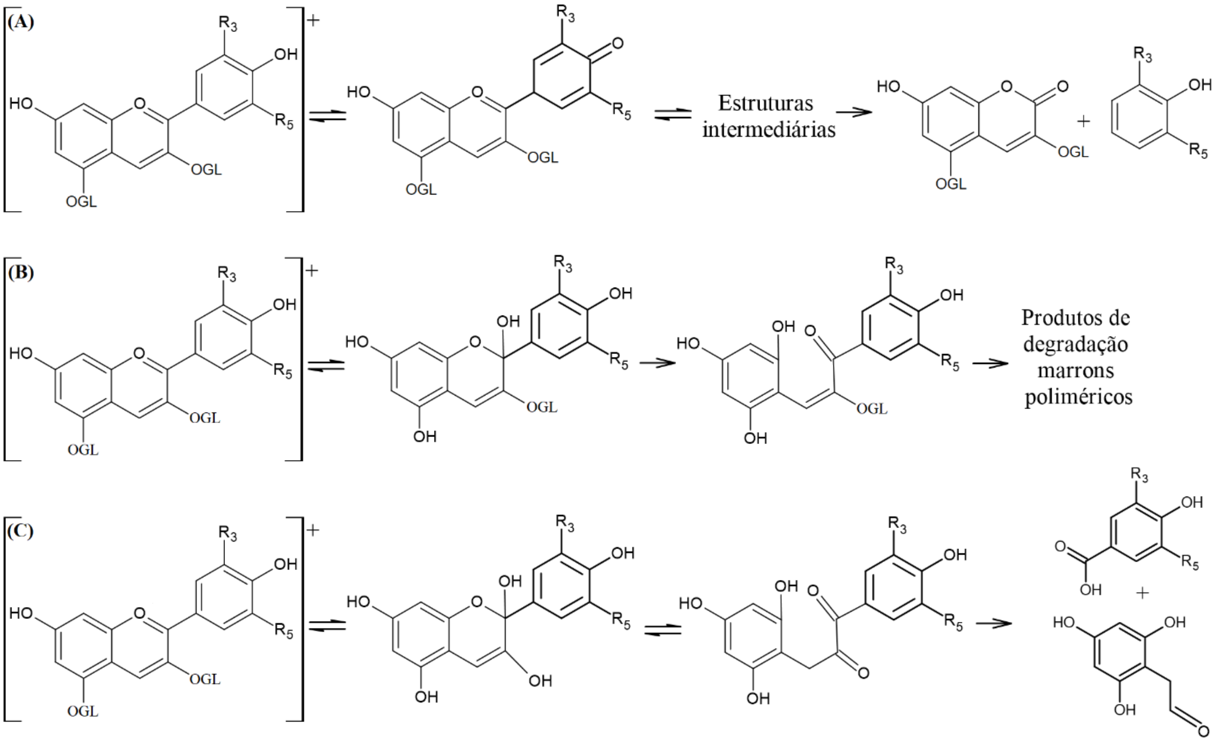

Figura 3. Mecanismos de degradação para a antocianidina-3,5-diglicosídeo e a antocianidina-3-diglicosídeo. R$_3$, R$_5$ = -OH, -H, -OCH$_3$ ou -OGL; GL, grupo glicosil.

Fonte: Schwartz et al. (2017).

3.3 DESIDRATAÇÃO OSMÓTICA

O tecido vegetal é composto por um aglomerado de células conectadas firmemente umas as outras através da lamela média, cuja função é impedir a migração celular. Ao contrário das células animais, as células vegetais possuem paredes celulares que permitem às mesmas exercerem grandes pressões hidrostáticas internas (pressão de turgor). Este fenômeno é essencial em muitos processos fisiológicos e promove a rigidez e a estabilidade mecânica dos tecidos vegetais não lignificados. O citoplasma, por sua vez, é a região que abrange as organelas e citoesqueleto suspensos no citosol (fase hidrossolúvel e coloidal que preenche o interior do citoplasma), sendo que o limite externo fluido do citoplasma é denominado de membrana plasmática ou plasmalema. Durante a regulação das células vegetais, as mesmas necessitam transportar moléculas tanto para o seu interior quanto para o exterior. Neste contexto, a difusão permite esse transporte de molélucas e representa o movimento espontâneo de substâncias através de regiões de maior concentração para regiões de menor concentração. Em escala celular, a difusão é o modo de transporte dominante (TAIZ et al., 2014).

A difusão da água através de uma membrana plasmática é denominada de osmose e envolve o fluxo de água da solução que tem maior potencial hídrico (menor concentração de soluto) para a de menor potencial hídrico (maior concentração de soluto). Deste modo, a presença do soluto diminui o potencial hídrico e gera uma diferença de potencial hídrico a favor do gradiente de concentração (CATH; CHILDRESS; ELIMELECH, 2006). Na Figura 4 podem ser observadas as mudanças de uma célula vegetal em nível microestrutural durante a desidratação.

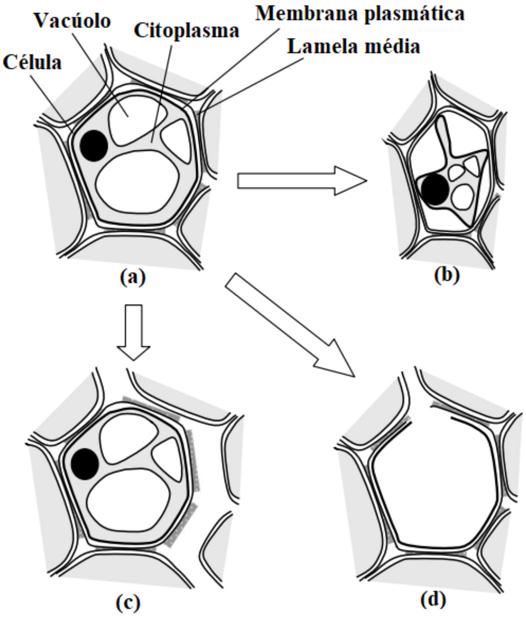

Figura 4. Mudanças da célula vegetal em nível microestrutural durante a desidratação. **(a)** célula túrgida; **(b)** plasmólise celular; **(c)** descolamento celular; **(d)** ruptura celular.

Fonte: adaptado de Mayor, Pissarra e Sereno (2008).

No primeiro caso (Figura 4a), a célula vegetal é imersa em solução com alto potencial hídrico, o protoplasto (citoplasma e núcleo) se expande, a membrana plasmática se estira e exerce uma pressão contrária à parede celular, o que deixa a célula túrgida. O turgor é mantido na maioria das células vegetais, pois elas geralmente vivem em ambiente com potencial hídrico relativamente elevado. No segundo caso (Figura 4b), a célula é colocada em solução com baixo potencial hídrico (ex.: solução de açúcar) e, assim, o vacúolo (responsável pelo armazenamento de substâncias) e o restante do protoplasto se retraem e causam o afastamento da membrana com a parede celular, num fenômeno conhecido como plasmólise. A perda do turgor, durante a plasmólise, resulta no "murchamento" dos tecidos vegetais (EVERT; EICHHORN, 2014).

Quando um material celular é imerso em solução hipertônica, as células da primeira camada entram em contato com a solução osmótica e perdem água, em função do gradiente de concentração entre as células e a solução. Após as células da primeira camada perderem água, uma diferença de potencial hídrico entre a primeira e a segunda camadas de células é estabelecida. Posteriormente, as células da segunda camada começam a transportar água para as células da primeira camada e, em seguida, ela encolhe. Os fenômenos de transferência de massa e encolhimento do tecido são difundidos da superfície ao centro do material, em função do tempo da operação. Finalmente, as células centrais perdem água e o processo de transferência de massa tende a se equilibrar, após um longo período de contato sólido-líquido (MAGUER; SHI; FERNANDEZ, 2003).

Outro fenômeno que pode ser observado durante a desidratação é o descolamento da lamela média, ou descolamento celular (Figura 4c). Este fenômeno pode ser originário da degradação ou desnaturação dos componentes da lamela média, assim como às microtensões produzidas no tecido vegetal, em detrimento da remoção da água. Este deslocamento celular pode influenciar as propriedades mecânicas do produto, assim como a porosidade do mesmo, uma vez que espaços intercelulares são formados. A ruptura celular (Figura 4d) decorre da degradação da membrana e da parede celular, bem como de micro estresses ocasionados pela remoção da água. Tal fenômeno leva à formação de cavidades de diferentes tamanhos e formas, que podem aumentar a porosidade do produto (MAYOR; PISSARRA; SERENO, 2008).

Por outro lado, a desidratação osmótica (DO) ou a impregnação por imersão é caracterizada pela imersão do alimento (inteiro ou em pedaços) em soluções aquosas hipertônicas de açúcares, como a sacarose, a glicose, a frutose e o sorbitol e/ou sais, como o cloreto de sódio. A DO reduz a quantidade de água no alimento e possibilita a diminuição da deterioração química, microbiológica e a depreciação sensorial (RAOULT-WACK, 1994).

A diferença de pressão osmótica, existente no sistema, promove o fluxo de água do material celular para a solução osmótica. Em alimentos, de 78 a 95% da água é encontrada nos espaços intracelulares e está fracamente associada ao substrato. A água que compõe a parede celular (2-5%) representa a água fortemente associada. A água encontrada nos espaços intercelulares, corresponde à água livre e pode ser facilmente removida durante a desidratação. Paralelamente à saída de solvente, ocorre o fluxo contrário de solutos, de menor proporção, da solução osmótica para o produto. Há, ainda, um terceiro fluxo correspondente à lixiviação de sólidos do produto para a solução; de menor relevância quantitativa, mas que pode causar importante perda da qualidade nutricional do produto (Figura 5) (BARBOSA-CÁNOVAS; VEGA-MERCADO, 1996; KHAN et al., 2016).

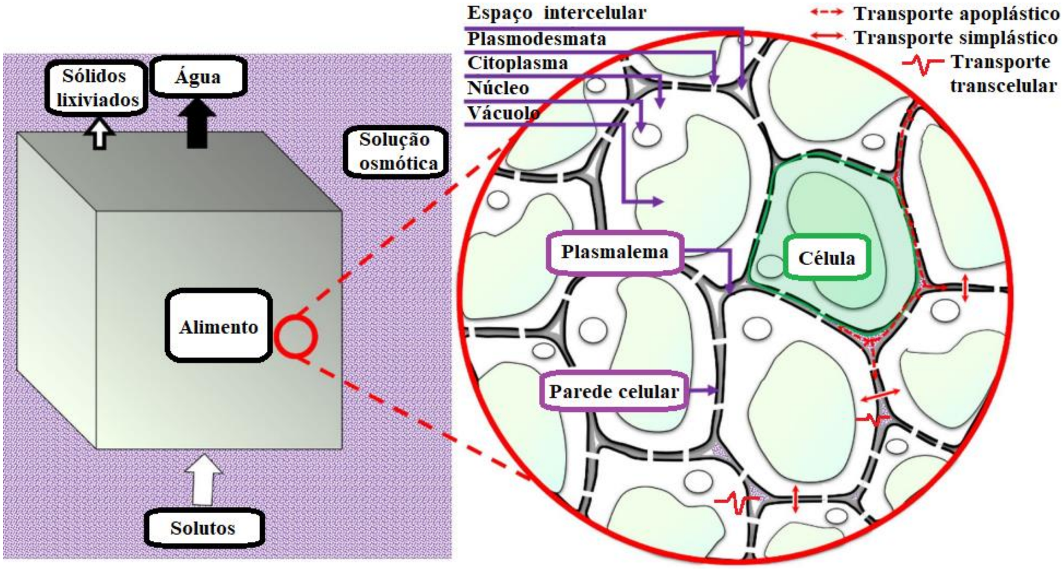

Figura 5. Transferência de massa durante o processo de desidratação osmótica.

Fonte: adaptado de González-Pérez, Ramírez-Corona e López-Malo (2021).

Na DO em tecidos vegetais (Figura 5), a água e solutos presentes no alimento são transportados para a solução osmótica por meio de três rotas, os transportes apoplástico, simplástico e transcelular. O primeiro atua entre as paredes celulares e espaços intercelulares. O segundo envolve a transferência de massa de uma célula a outra, através dos plasmodesmatas, que são canais responsáveis pela conexão citoplasmática entre células vizinhas. O terceiro, por sua vez, é definido como o transporte de solvente e soluto entre o interior da célula (citoplasma e vacúolo) e o exterior da célula (parede celular e espaço intercelular), por intermédio da membrana plasmática (TOUPIN; MARCOTTE; LE MAGUER, 1989).

A membrana plasmática é uma estrutura semipermeável que regula a quantidade, tipo, frequência e direção do movimento de substâncias que passam através dela, sendo constituída por uma dupla camada lipídica, na qual estão imersas as proteínas globulares. A estrutura de bicamada lipídica é anfipática, isto é, a porção interna (cauda) é hidrofóbica e porção externa (cabeça) é hidrofílica. Enquanto o grupo com a cabeça polar se comunica com o ambiente aquoso externo, as caudas hidrofóbicas constituídas de ácidos graxos impedem a difusão desordenada de solutos entre os compartimentos celulares (CRANG; LYONS-SOBASKI; WISE, 2018). A Figura 6 evidencia a estrutura da membrana plasmática e os mecanismos de transporte que ocorrem através dela.

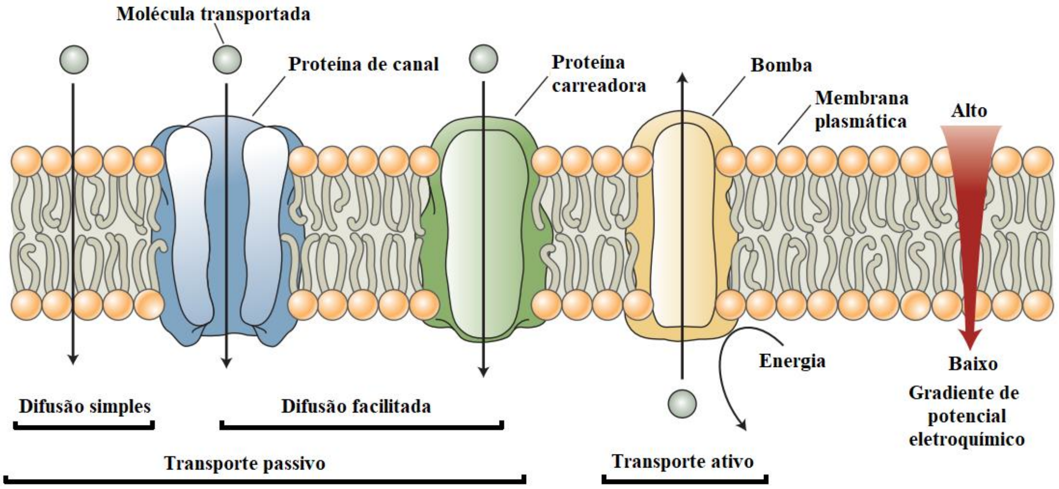

Figura 6. Transportes através da membrana plasmática.

Fonte: adaptado de Taiz et al. (2014).

Pequenas moléculas polares não carregadas eletricamente (ex. água) podem permear a membrana por difusão simples. A difusão facilitada é o transporte de substâncias polares que ocorre através de proteínas carreadoras e proteínas de canal. As primeiras ligam-se a um soluto específico (íons Ca^{+2} ou moléculas de açúcar) e as últimas permitem que solutos selecionados (ex. Na^+ e K^+) passem por poros preenchidos com água. Tanto a difusão simples quanto a facilitada ocorrem a favor do gradiente de concentração e representam o transporte passivo. O transporte ativo move substâncias contra seus gradientes de concentração ou gradientes eletroquímicos, sendo intermediado por proteínas de transporte, conhecidas como bombas. Este tipo de transporte envolve gasto de energia (Figura 6) (EVERT; EICHHORN, 2014).

É importante ressaltar que o movimento de água pela membrana plasmática não envolve apenas a difusão simples de moléculas de água através da dupla camada lipoproteica, como observado na Figura 6, mas envolve também a difusão linear através de poros seletivos para a água, constituídos por proteínas integradas a membrana, denominadas de aquaporinas. Apesar das aquaporinas alterarem a taxa de movimento da água através do plasmalema, elas não mudam a direção do transporte, tampouco a força motriz para a realização desse movimento. No entanto, as aquaporinas podem ser reversivelmente reguladas ao oscilarem entre um estado aberto e outro fechado, em detrimento de parâmetros fisiológicos (ex. níveis intracelulares de pH e Ca^{+2}) (TAIZ et al., 2014). A Figura 7 mostra o transporte de água através da membrana plasmática, por simples difusão e através das aquaporinas.

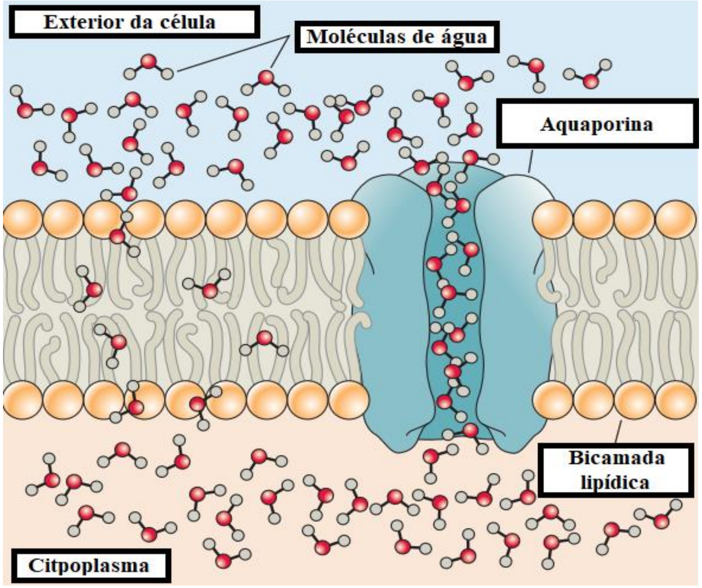

Figura 7. Transporte de água pelo plasmalema, através da difusão simples e das aquaporinas.

Fonte: adaptado de Taiz et al. (2014).

Geralmente, a DO é um processo lento e apresenta certa resistência ao fluxo de massa, que ocorre entre a membrana celular de frutos e o agente osmótico; assim, pode ser necessária até uma semana para que a umidade desejável seja atingida. O prolongado tempo de processo pode resultar em alterações fisiológicas e bioquímicas e, eventualmente, pode afetar a qualidade dos produtos finais. Além disso, a carga microbiana é frequentemente considerada uma das principais preocupações para a qualidade microbiológica, de produtos desidratados osmoticamente (AHMED; QAZI; JAMAL, 2016).

Visando reduzir o tempo da DO, diversos fatores têm sido estudados para melhorar a eficiência das taxas de perda de água (PA) e de ganho de sólidos (GS), sendo os principais: a concentração da solução, a temperatura da solução, o tempo de contato, a velocidade de agitação da solução, a forma e o tamanho do sólido, a razão solução/sólido e a pressão (atmosférica ou vácuo) (RASTOGI et al., 2002). Na Tabela 4 são apresentadas algumas variáveis que exercem efeito sobre a desidratação osmótica de matrizes vegetais.

Tabela 4. Variáveis envolvidas na desidratação osmótica (DO) de matrizes vegetais.

Vegetal	**Agente osmótico (Concentração)**	**Temperatura**	**Pulso de vácuo (Tempo de pulso)**	**Tempo de imersão**	**Referência**
Manga	Sacarose (45-65%)	20-40ºC	50-200 mbar (0-20 min)	1-5 h	Ito et al. (2007)
Goiaba	Sacarose (40-60%)	40ºC	100 mbar (10-15 min)	0.25-5 h	Corrêa et al. (2010)
Goiaba	Sacarose (40-60%)	20-40ºC	100 mbar (0-20 min)	3 h	Vieira, Pereira, Hubinger (2012)
Abóbora	Sacarose (15-55%), cloreto de sódio (0-20%)	40ºC	100-200 mbar (5-25 min)	5 h	Corrêa et al. (2014)
Maçã	Sacarose (40-50%), lactato de cálcio (2-4%)	27ºC	-	1-6 h	Silva, Fernandes e Mauro (2014)
Morango	Sacarose (25-50%)	40ºC	50 mbar (10 min)	0.5-3 h	Cheng et al. (2014)
Tomate	Cloreto de sódio (0-10%)	25-45ºC	25-112,5 mbar (10 min)	5 h	Corrêa et al. (2015b)
Pêra	Sacarose (30-60%), cloreto de cálcio (0-2%)	25ºC	-	3 h	Ribeiro, Aguiar-Oliveira e Maldonado (2016)
Berinjela	Cloreto de sódio (5-10%), cloreto de cálcio (0,5-1%), cloreto de potássio (2,5-4%)	30ºC	145 mbar (10 min)	0.2-6 h	Junqueira et al. (2017)
Maçã	Sacarose (40%), lactato de cálcio (4%), ácido ascórbico (2%)	25ºC	-	0,5-4 h	Tappi et al. (2017)
Castanha	Sacarose (53-87%)	20-70ºC	-	0,8-9,2 h	Delgado et al. (2018)
Beterraba, cenroura e beringela	Sacarose (40%), cloreto de sódio (10%)	35ºC	40-80 kPa (10 min)	5 h	Junqueira et al. (2018)
Figo verde	Sacarose (40-60%)	40ºC	74 mm Hg (5 min)	4 h	Mello Jr. et al. (2019)
Azeitona de outono	Açúcar comercial (70%)	70ºC	300 mbar (5-10 min)	0,5-10 h	Grellam et al. (2021)

O controle das principais variáveis da DO pode resultar em dois processos distintos: a desidratação, que ocorre quando há o predomínio da perda de água em relação ao ganho de sólidos e, nesses casos, geralmente são utilizados solutos com alto peso molecular. Em contrapartida, a impregnação por imersão é o resultado do maior ganho de soluto em comparação com a perda de água, a qual é favorecida, principalmente, por solutos de baixo peso molecular (RAOULT-WACK, 1994).

O tipo de agente osmótico afeta diretamente as cinéticas do processo de DO. Assim, aqueles que apresentam carboidratos são os mais utilizados na desidratação de frutos e a sacarose é o açúcar mais empregado para esta finalidade, pois tem baixo valor comercial. Além de favorecer a estabilidade de pigmentos durante o armazenamento, a presença de sacarose na superfície do alimento pode representar uma barreira contra a entrada de oxigênio e acarretar na redução do escurecimento enzimático (TONON; BARONI; HUBINGER, 2006). A quantidade de água removida na OD é também fortemente dependente da concentração da solução osmótica. Portanto, com o aumento da concentração, a PA é mais influenciada do que o GS, devido ao aumento da pressão osmótica na solução (SHETE et al., 2018).

A adição de sais de cálcio em processos de DO tem contribuído para preservar a integridade estrutural da parede celular de alimentos desidratados osmoticamente, de modo a favorecer as características de textura do produto final. O uso dos sais de cálcio permite a interação entre os íons Ca^{+2} e os grupos carboxil da pectina que compõe a parede celular, proporcionando maior rigidez na estrutura celular dos frutos (STANLEY et al., 1995).

A temperatura utilizada no tratamento osmótico também é considerada um dos fatores mais importantes que afetam as cinéticas de DO, pois o seu aumento favorece a PA. No entanto, estudos com DO de frutas, que utilizam temperaturas de até 50ºC, têm alguns efeitos adversos minimizados, como a deterioração do *flavour*, da textura, dos compostos bioativos termo sensíveis e do escurecimento enzimático (AKBARIAN; GHASEMKHANI; MOAYEDI, 2014).

O uso do vácuo na DO é uma forma eficiente de prevenir a perda da coloração de frutas, devido à oxidação ou ação enzimática, o que é atribuído a remoção do oxigênio dos poros do produto (ZHAO; XIE, 2004). A impregnação à vácuo possibilita inserir quantidades controladas de uma solução dentro da estrutura dos poros das frutas e vegetais. A solução utilizada pode conter um componente fisiologicamente ativo, redutores da a_w e do pH, e elementos antimicrobianos, com o intuito de obter um produto estável (FITO et al., 2001).

A aplicação de pulso de vácuo, por um curto período, no início do processo de DO, promove a expansão da estrutura porosa dos tecidos vegetal e animal, associada à evacuação dos gases ou líquidos ocluídos nos poros. No primeiro momento, o sistema é submetido a uma pressão subatmosférica, onde o gás ocluso nos poros se expande até o equilíbrio com a pressão imposta ao sistema, o que promoverá a saída do gás e a drenagem de parte do líquido nativo, presente no interior dos poros. Em seguida, a pressão atmosférica do sistema é restabelecida, o produto é mantido imerso na solução por um tempo adicional e o líquido que está em contato com o alimento penetra no interior dos poros, devido aos gradientes macroscópicos de pressão e à capilaridade (FITO, 1994).

3.4 SECAGEM

A secagem é uma operação unitária que transforma um material sólido, semissólido ou líquido em um produto sólido, cuja umidade é considerada baixa. Tal fenômeno implica na transferência de água do material para uma fase gasosa não saturada. De maneira geral, a remoção da água acontece por evaporação e envolve a aplicação de energia térmica, que promove a mudança de fase da água líquida para vapor. Durante a secagem de materiais sólidos, dois processos simultâneos podem ocorrer: a transferência de calor para evaporar o líquido, onde o calor se propaga da superfície do sólido para o seu interior; e a transferência de massa, onde o líquido ou vapor são transportados do interior para a superfície exposta do sólido (TELIS; MAURO, 2016).

O calor necessário para evaporar a água dos alimentos é transmitido por três mecanismos: condução, convecção e radiação. Na secagem por ar quente, onde o produto entra em contato com a corrente de ar quente, o calor é essencialmente transmitido por convecção. A maior parte da água está presente no interior dos alimentos e, para atingir a superfície e ser evaporada, a água se move por diferentes mecanismos (ORDOÑEZ, 2005). Alguns dos possíveis mecanismos, utilizados para explicar o movimento de umidade, são (TELIS; MAURO 2016):

- ***Difusão líquida***: a água é difundida pelo sólido em função do gradiente de concentração existente entre as porções internas do sólido (maior potencial hídrico) e a superfície (menor potencial hídrico);
- ***Difusão de vapor em razão do gradiente de pressão parcial de vapor***: a água se move do interior do sólido na forma de vapor, desde que um gradiente de temperatura seja estabelecido por aquecimento, o que resulta num gradiente de pressão de vapor;
- ***Movimento de líquido em razão de forças capilares***: a água se movimenta por meio de interstícios capilares, através da atração molecular sólido/líquido. À medida em que a

água é removida dos poros, uma pressão capilar é estabelecida e promove a sucção do líquido contido nos capilares;

- ***Movimento de líquido ou vapor provocado por diferenças na pressão total***: ocorre a partir de forças externas, através da contração do material (encolhimento), aplicação de altas temperaturas e capilaridade.

As transferências de calor e massa que ocorrem na secagem são analisadas a partir das curvas de secagem, na qual é expressa a umidade do produto ao longo do tempo. A Figura 8 mostra uma curva típica de secagem para condições constantes de temperatura, umidade relativa e velocidade do ar de secagem, juntamente com os períodos característicos das taxas de secagem e a mudança de temperatura no produto.

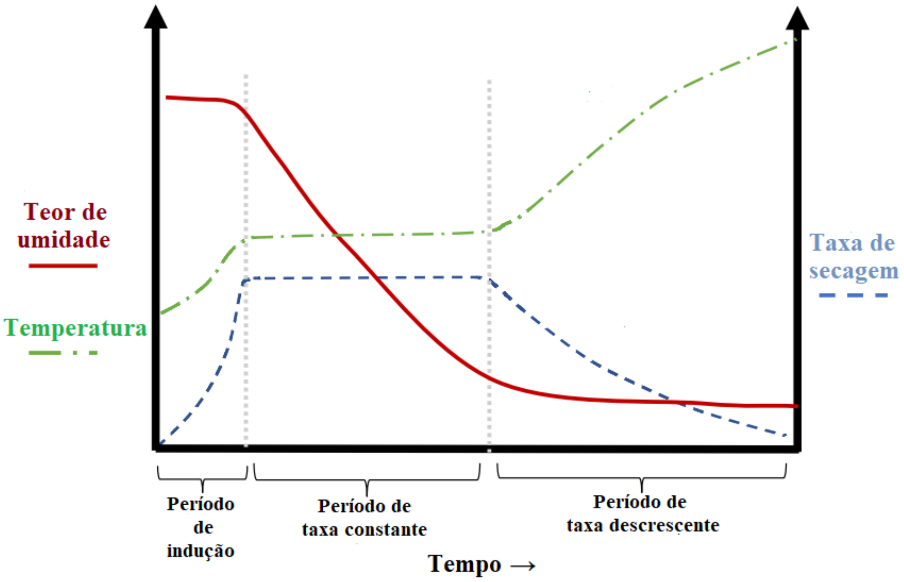

Figura 8. Curva típica de secagem para condições constantes.

Fonte: adaptado de Breslin (2020).

A primeira etapa corresponde ao período de indução ou fase de aquecimento, onde o produto absorve energia do ar quente do secador e aumenta a sua temperatura interna. Assim que o aumento da temperatura cessa, a fase de aquecimento termina. No período de taxa constante, qualquer energia absorvida pelo produto, através do ar quente, é equilibrada pela energia perdida devido à evaporação e, portanto, a temperatura da superfície do produto permanece relativamente constante. A umidade no ponto de transição entre a segunda fase (período de taxa constante) e a terceira fase (período de taxa decrescente) é denominado de umidade crítica e marca o início da terceira fase. Nesta etapa, a resistência interna aumenta e a o movimento da água, na forma líquida ou na forma de vapor, diminui. Assim, a velocidade com que a água da superfície do produto evapora é maior do que a velocidade com que a água interna migra para a superfície. Fisicamente, isso resulta em um aumento da temperatura na superfície do produto e é um indicativo do quão firmemente as moléculas de água estão associadas aos solutos. Como resultado, mais energia é necessária para remover as moléculas de água no período de taxa decrescente (BRESLIN, 2020).

Para predizer o comportamento da curva de secagem de um produto são utilizados modelos matemáticos, os quais devem considerar, simultaneamente, os processos de transferência de calor e massa, que ocorrem durante o processo de secagem. A modelagem matemática é importante para o dimensionamento de secadores, previsão das taxas de secagem, aprimoramento das condições operacionais e avaliação do processo (NASCIMENTO; BIAGI; OLIVEIRA, 2015).

A retirada parcial ou total da água, durante a secagem, diminui a ocorrência de reações bioquímicas indesejáveis, responsáveis por processos degradativos e implica em melhor preservação do alimento. A remoção da água também evita danos causados por micro-organismos, o que aumenta a vida de prateleira do produto (CACCAVALE; DE BONIS; RUOCCO, 2016).

Embora a secagem promova a estabilidade do alimento por um longo período de armazenamento, as condições do processo podem provocar depreciações na qualidade do produto, tais como: dureza excessiva e degradação da cor, aroma e sabor (RATTI, 2001). Baseado nisto, a desidratação osmótica (DO) é apontada como um pré-tratamento a ser utilizado para diminuir esses efeitos adversos, que geralmente ocorrem quando o produto é submetido diretamente à secagem convectiva (RASTOGI; RAGHAVARAO, 1997).

A combinação das técnicas de DO e secagem tem sido vinculada como uma alternativa barata e segura para a conservação de produtos alimentícios, além de possibilitar a obtenção de produtos desidratados de melhor qualidade, quando comparado com os produtos desidratados convencionalmente (BRANDÃO et al., 2003). Pesquisas recentes têm destacado a importância de se acompanhar a qualidade do produto durante o processo combinado de DO e secagem. Neste contexto, a Tabela 5 expressa os trabalhos que envolvem a influência da combinação de DO e secagem no teor de compostos bioativos.

Tabela 5. Influência da DO e secagem sobre o teor de compostos bioativos de matrizes vegetais.

Vegetal	Técnica aplicada	Composto bioativo	Principais observações	Referência
Cereja ácida	DO + CD + VMD	Compostos fenólicos	O uso da DO, antes do processo híbrido de secagem, resultou numa ligeira degradação de compostos fenólicos. No entanto, a maior degradação ocorreu em cerejas sem sementes.	Nowicka et al. (2015)
Casca de limão	DO + CD	Compostos fenólicos	O pré-tratamento osmótico ocasionou perdas de compostos fenólicos e as quantidades de polifenóis, que permaneceram no produto osmo-desidratado, quase não sofreram redução durante a posterior etapa de secagem.	Romdhane et al. (2017)
Pequi	DO + VD	Carotenoides totais e vitamina C	O pré-tratamento osmótico e os longos períodos de secagem foram associados a uma redução no teor de compostos bioativos. Por outro lado, a secagem à vácuo (nas temperaturas mais baixas) reteve os maiores teores de carotenoides totais e de vitamina C.	Mendonça et al. (2017)
Baga *Goji*	DO + CD	Compostos fenólicos	O produto final apresentou maior concentração de compostos fenólicos quando comparado ao produto seco, sem o pré-tratamento osmótico.	Dermesonlouoglou, Chalkia e Taoukis (2018)
Mirtilo vermelho	DO + VMD	Compostos fenólicos, flavonoides totais e antocianinas monoméricas	A pesquisa revelou que a maior quantidade de compostos bioativos, encontrada no mirtilo seco, ocorreu no tratamento que utilizou a DO, em comparação com as amostras secas por liofilização ou com as amostras secas por micro-ondas à vácuo.	Zielinska, Zielinska e Markowski (2018)
Ameixa	USOD + CD	Compostos fenólicos	Percebeu-se que as ameixas secas e desidratadas osmoticamente, com solução de sacarose, apresentaram maiores retenções de compostos fenólicos do que aquelas que foram pré-tratadas osmoticamente com solução de glicose.	Rahaman et al. (2019)
Caqui	USOD + CD	Compostos fenólicos	O produto final, obtido a partir da combinação da desidratação osmótica com secagem, apresentou menor teor de compostos fenólicos do que o produto submetido apenas à secagem.	Bozkir et al. (2019)
Gengibre chinês	PVOD + USOD + MWD + CD	Compostos fenólicos e flavonoides totais	O estudo demonstrou que a maior quantidade de compostos fenólicos e flavonoides totais foi encontrada em gengibres secos que passaram por pré-tratamento osmótico.	An et al. (2019)
Mamão	DO + CD	Compostos fenólicos e vitamina C	Em relação ao produto desidratado osmoticamente e seco, observou-se que os teores de compostos fenólicos e de vitamina C apresentaram as maiores retenções quando, durante o pré-tratamento osmótico, foram utilizadas soluções osmóticas mais concentradas, em temperaturas mais baixas.	Islam et al. (2019)
Mangostão	DO + CD	Vitamina B, β-caroteno, vitamina C, flavonoides totais e compostos fenólicos	Verificou-se que a melhor forma de preservar os constituintes fitoquímicos do fruto foi através de um tratamento osmótico, com frutose como agente osmótico, aliado à secagem a 45ºC. Neste tratamento, os teores de vitamina B (B_1, B_2 e B_3), flavonoides totais e compostos fenólicos apresentaram os maiores valores, em comparação com os tratamentos osmóticos com solução de cloreto de sódio ou sacarose.	Hossain, Dey e Joy (2021)
Cenoura	DO + MWD + CD	Carotenoides totais	Cenouras secas e pré-tratadas osmoticamente apresentaram menores teores de carotenoides totais quando comparadas com cenouras secas em sistema híbrido de secagem por micro-ondas e ar quente.	Souza et al. (2022)
Citrus japônica	DO + VD	Compostos fenólicos	Amostras secas do citrus japônica apresentaram maiores teores de compostos fenólicos em comparação com as amostras secas, que passaram por DO previamente.	Özkan-Karabacak et al. (2022)

DO: desidratação osmótica; PVOD: desidratação osmótica por pulso de vácuo; USOD: desidratação osmótica por ultrassom; CD: secagem convectiva; VD: secagem à vácuo; MWD: secagem por micro-ondas; VMD: secagem por micro-ondas à vácuo.

3.5 ISOTERMAS DE SORÇÃO DE UMIDADE

A água é o componente majoritário dos alimentos, sendo fundamental para a estabilidade química, física e microbiológica dos componentes alimentares. A determinação da umidade é utilizada para quantificar o teor total de água presente no alimento. Porém, o desenvolvimento microbiano e as reações químicas não podem ser preditos, com segurança, por meio da relação direta com a umidade; pois, produtos distintos podem diferir em suas propriedades, mesmo apresentando umidades iguais (FRAZIER, 2015).

Isso ocorre porque existem diferenças na intensidade com que a água se associa aos constituintes não aquosos do alimento. Esta intensidade é mensurada através da atividade de água (a_w). Neste sentido, a água fortemente associada aos solutos do alimento é menos capaz de estar associada aos processos degradativos, do que a água que está fracamente associada (FENNEMA, 1996) ou aquela que se encontra livre.

Parte da água, que se encontra fortemente associada aos sítios específicos do alimento, pode interagir com grupos hidroxila, dos polissacarídeos, assim como com grupos carbonila e amino, de proteínas, ou por meio de outras ligações químicas fortes. As moléculas de água que estão fracamente associadas ao alimento ainda não se encontram disponíveis para agir como solvente nos vários componentes alimentares, solúveis em água. Assim, à porção de água que atua como solvente, denomina-se água livre (Figura 9) (BARBOSA-CÁNOVAS; JULIANO, 2007).

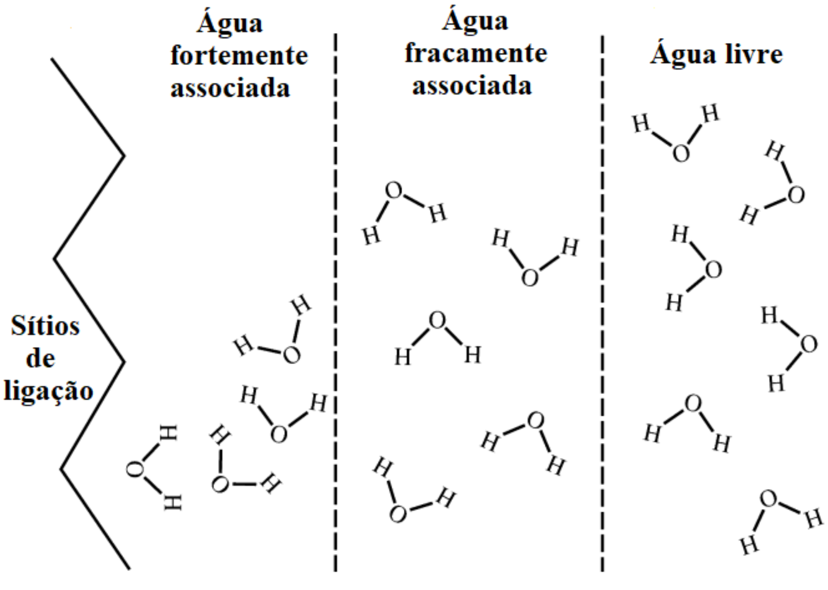

Figura 9. Água fortemente associada, fracamente associada e água livre em alimentos.

Fonte: adaptado de Barbosa-Cánovas e Juliano (2007).

Ao correlacionar a umidade de um produto (expresso como a massa de água por unidade de matéria seca) com a a_w correspondente, à temperatura constante, obtêm-se as isotermas de sorção de umidade ou curvas de equilíbrio (LABUZA, 1975). As isotermas de sorção de umidade podem ser de adsorção ou dessorção (Figura 10). A adsorção é associada ao ganho de umidade, ao passo que a dessorção é caracterizada pela perda de umidade de um produto, quando o mesmo é exposto à ambientes com temperatura e umidades relativas (UR) controladas, até que a umidade de equilíbrio seja atingida. Uma isoterma de adsorção é obtida quando a amostra seca é colocada em contato com várias atmosferas com UR crescentes. Uma isoterma de dessorção, por outro lado, é obtida quando a amostra úmida é exposta em ambiente com UR decrescentes (LABUZA, 1968).

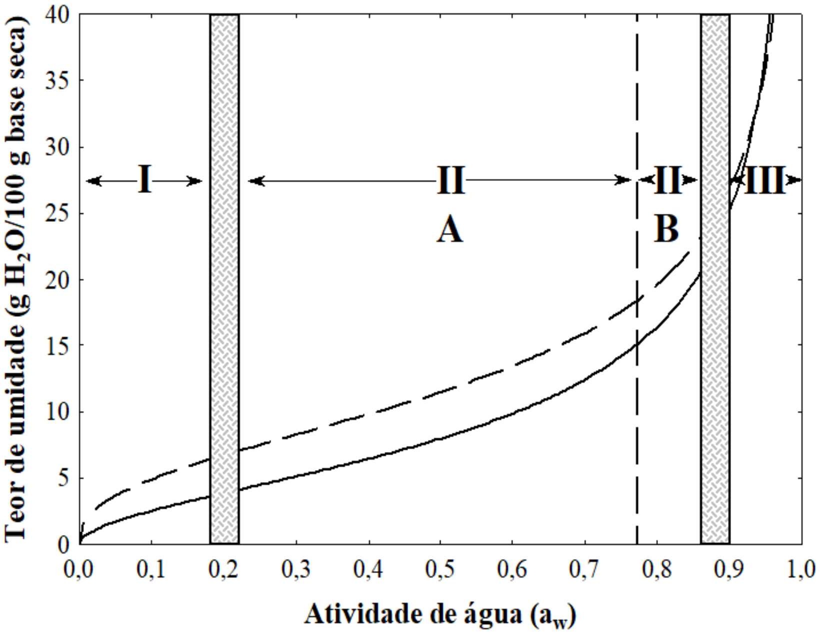

Figura 10. Isoterma de adsorção (———) e dessorção (— — — —) de umidade, generalizada para um alimento, à temperatura constante.

Fonte: adaptado de Damodaran (2017).

O formato sigmoide da isoterma apresentada na Figura 10 sugere que a água se encontra em diferentes estados acoplados ao alimento, caracterizados por três regiões (zonas), com diferentes níveis de umidade. A água da zona I, que se estende até o primeiro ponto de inflexão ($a_w \approx 0{,}2\text{-}0{,}25$), caracteriza-se por estar associada a grupos iônicos (interação íon-dipolo) e alguns grupos polares (interação dipolo-dipolo). A porção extrema da zona I representa a região de monocamada, onde a água está confinada nos sítios de ligação com alta afinidade. Na zona II ($a_w \approx 0{,}2\text{-}0{,}85$), a quantidade de água correspondente a sub-região II-A, encontra-se associada ao alimento por ligações de hidrogênio, ao passo que a água da sub-região II-B está interagindo fracamente com as superfícies apolares do alimento, por interações dipolo-induzido-dipolo. Como a água da zona II se encontra fracamente associada, ela é mais móvel que a água da zona I; no entanto, menos móvel do que a água livre (3º região). Assim, quando a água atinge a zona III, a sua mobilidade molecular aumenta e, portanto, torna-se disponível para participar de processos biológicos e alterar a velocidade das reações químicas, assim como as propriedades de textura dos alimentos (DAMODARAN, 2017).

Para predizer o comportamento higroscópico de alimentos, equações matemáticas são ferramentas comumente utilizadas e, na literatura, existem mais de duzentas equações capazes de prever o fenômeno de equilíbrio higroscópico de produtos agrícolas. Esses modelos diferem na sua base teórica ou empírica e na quantidade de parâmetros envolvidos (AL-MUHTASEB et al., 2010). As curvas de sorção de umidade fornecem informações importantes para a definição do processo de secagem, para a seleção do material a ser utilizado na embalagem e para a previsão da estabilidade durante o armazenamento e o transporte do produto (TUNC; DUMAN, 2007).

A partir dos dados de equilíbrio de sorção, em diferentes temperaturas, podem-se obter as propriedades termodinâmicas. Tais propriedades contribuem para a projeção de secadores e auxiliam nos cálculos energéticos, requeridos na etapa de secagem. Além disso, as propriedades termodinâmicas englobam o estudo da água adsorvida, da microestrutura dos alimentos e dos fenômenos físicos que ocorrem na superfície dos mesmos (OLIVEIRA et al., 2011).

A entalpia diferencial, também denominada de calor isostérico de sorção (Q_{st}), é a propriedade termodinâmica que indica a força exercida pela ligação entre as moléculas de água e os sítios de sorção do produto. Por outro lado, a entropia diferencial é proporcional ao número de sítios de sorção presentes em um determinado nível energético, que acontece na interface água/soluto. Já a energia livre de Gibbs, relaciona a espontaneidade da interação água-adsorbato e proporciona a medida da disponibilidade energética de uma reação. Assim, valores negativos indicam um processo de sorção espontâneo, enquanto que valores positivos sinalizam um processo não espontâneo. Por fim, a teoria da compensação entalpia-entropia, avalia os fenômenos físicos e químicos envolvidos no mecanismo de sorção de umidade e identifica se a adsorção e dessorção são processos governados pela entalpia ou pela entropia (TSAMI et al., 1990; MADAMBA, DRISCOLL, BUCKLE, 1996; RIZVI, 2005; SHARMA et al., 2009).

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Capítulo III

ARTIGO 1 – MOISTURE DESORPTION BEHAVIOR AND THERMODYNAMIC PROPERTIES OF PULP AND SEED OF JAMBOLAN (*Syzygium cumini*)

PUBLISHED IN THE JOURNAL ENTITLED **HELIYON**

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Submitted in 29/10/2021. Accepted in 11/05/2022. Published online in 15/05/2022.

ABSTRACT

The study objectives were to establish isotherms and thermodynamic properties for the moisture desorption process of jambolan pulp (JP) and jambolan seed (JS), harvested in the city of Belém (Brazil). These characteristics can contribute for the proper selection of the operating drying conditions. Thus, the following essays were made for both JP and JS. Firstly, proximate composition was performed, followed by moisture desorption essays at 25, 35, 45 and 55ºC. In addition, six mathematical models were fitted to the experimental data to simulate the desorption behavior; and based on the chosen models, the thermodynamic properties were calculated. The results have shown that JP isotherms followed the typical behavior of products rich soluble solids, and JS isotherms were more influenced by protein components. The influence of temperature was evidenced throughout the entire range of water activity (a_w) studied. The GAB and Oswin models represented the best fitted equations for the JP and JS, respectively. In general, the energies involved in the desorption process of jambolan showed a greater dependence of JP with the equilibrium moisture content (EMC), in comparison with JS. Still, there was an increasing tendency of the thermodynamic properties with EMC decreasing. Besides of being non-spontaneous processes, desorption phenomena of JP and JS were enthalpy-driven mechanisms.

Keywords: *Myrtaceae*; equilibrium moisture; mathematical modeling; thermodynamics**.**

1 INTRODUCTION

Syzygium cumini is a tropical fruit from the *Myrtaceae* family, popularly known as jambolan, jamun, black plum or java plum. This berry presents an ovoid form, a skin varying from purple-red to black color in the ripe stages, and a total length of 2 to 3 cm. It also presents an astringent taste in a freshly pink/white pulp (FARIA; MARQUES; MERCADANTE, 2011). All parts of the fruit, besides their nutritional constituents, contain many phytochemicals, and thus, they are widely used for medicinal purposes (PAUL; DAS, 2019). Some of the fruit's constituents have been attributed to the presence of anthocyanins, flavonoids, and terpenes (AQIL et al., 2014).

Jambolan, however, presents a short seasonal availability and a high perishable nature, which enhances the use of preservative techniques for lowering the moisture content of these fruits and make them available throughout the year (PAUL; DAS, 2019). There are still few works regarding the use of new processes that can improve the shelf life of jambolan; in this sense, drying could be used as the preservative process and the dried pulp could be used as food ingredient in the preparation of new products. Moreover, due to the large application in the traditional medicine, jambolan pulp and seed have a great potential for commercialization in the dried state, facilitating the transportation and storage stages as well.

In order to optimize the drying processes and guarantee stability of the dried products during storage, the knowledge of the product's moisture sorption isotherms is necessary, which expresses the graphical relationship between the food's equilibrium moisture content (EMC) and its correspondence water activity (a_w), over a range of values and at constant temperature (DAMODARAN, 2017).

Brunauer et al. (1938) classified sorption isotherms by their shape and processes. Type II is one of the most frequent isotherms in foods, and may be classified into three zones. In general, a hypothetical food system of the first zone is associated with small quantities of water and its molecules are tightly bound to polar sites. In zone II, the water is less bound, occurring as multilayer, right after the monolayer. At this region, chemical and biochemical reactions take place. In zone III, water fills the macro capillaries, exhibiting almost the full properties of bulk water, as microbial growth becomes the major deteriorative reaction (AGUILERA; STANLEY, 1999).

Furthermore, foodstuffs rich in soluble compounds, such as sugars, usually behave as type III, according to the classification mentioned in the previous paragraph. Falade and Aworh (2004) theorizes that, at low a_w values, it can occur a local dissolution of sugar alcohols, a swelling of proteins and an arising of new active sites. In the intermediate a_w range, sorption appear at the less active sites, while at higher a_w, dissolution of sugars gradually takes place and results in the complete exudation of sugars in solution.

In the case of fresh fruits and seeds, desorption isotherms are essential tools in the drying stages, working for the proper selection of operating conditions in regard to drying, packaging, and storage requirements for a desired shelf life. For this, thermodynamic properties of moisture sorption have been appointed as one of the approaches used to understand the water properties in the food matrix, and calculate the energy demands of heat and mass transfer in the food drying (KHIARI et al., 2020).

The study of these thermodynamic properties is of great importance for the projection and dimensioning of equipment in several preservation processes. Such properties include enthalpy, entropy, Gibbs free energy and enthalpy-entropy compensation theory; featuring essential roles in describing the reactions and phenomena that occur at the intermolecular level in food materials (SORMOLI; LANGRISH, 2015).

No literature was found for sorption isotherms of jambolan seeds, only studies involving other seeds (PRETTE et al., 2013; ASLAN-TONTUL, 2020). In contrast, some current works have analyzed the moisture sorption characteristics of fresh jambolan pulp (BISWALL et al., 2017), freeze-dried jambolan (SANTANA et al., 2014), and microwave-convective hot air dried jambolan (PAUL; DAS, 2019). The latter work focused on evaluating the hygroscopic behavior from a mixture composed by the pulp and seed powders of jambolan. The present study, however, evaluated the hygroscopic behavior for both pulp and seed of jambolan, separately. Furthermore, no studies have contemplated the thermodynamic properties of fresh jambolan pulp and seed, separately, in order to understand better the energy requirements for their drying processes.

Thus, this study aimed: (I) to determine the desorption isotherms of jambolan pulp and seed in a relative humidity (RH) range of 90–10%, at 25, 35, 45 and 55ºC; (II) to define the most suitable model fitted to the desorption isotherms for jambolan pulp and seed; and (III) to estimate the thermodynamic properties of moisture desorption for jambolan pulp and seed.

2 MATERIAL AND METHODS

2.1 RAW MATERIAL

Jambolan (*Syzygium cumini*) fruits were harvested during the rainy season, in the period of December 2020 to January 2021, from trees located at the Federal University of Para – UFPA (latitude 1º28’ S, longitude 48º29' W), in the city of Belém (Brazil). It is worth mentioning that this study refers only to the jambolans grown in the city of Belém. The climate of this region is tropical monsoon, with annual air temperature between 22ºC to 31.5ºC, and daily precipitations around 25 mm, at average (INMET, 1992). In Brazil, jambolan flowering goes from September to November, and the full ripening goes from December to February (SABINO; BRITO; SILVA JÚNIOR, 2018).

Right after harvesting, ripe fruits were transported in polystyrene isothermal boxes to the Laboratory of fruits at UFPA. Posterior to discard of the injured jambolans, the fruits were rinsed in running water, and sanitized by immersion in hypochlorite solution at 20 mg/L for 15 minutes, followed by rinsing and drying with an absorbent paper, as described by Araújo and Pena (2021). Then, jambolan pulp (JP) was separated from the jambolan seed (JS), with a stainless steel laboratory spatula, in order to proceed with the next analyzes.

2.2 PROXIMATE COMPOSITION

JP and JS were submitted to the following analyzes: moisture content (n. 934.06), ashes (n. 940.26), total proteins (with a nitrogen-to-protein conversion factor of 6.25) (n. 920.152) and total lipids (n. 945.38F). These procedures were performed according to AOAC (2010) methodology. In addition, carbohydrates were calculated by the difference between 100 and the sum from the percentage of moisture, total proteins, total lipids and ashes (FAO, 2003). Water activity (a_w) was determined by direct reading in the Vapor Sorption Analyzer (VSA) (Aqualab VSA, Decagon, USA). The experiments were obtained in triplicates, expressed as mean ± standard deviation.

2.3 MOISTURE DESORPTION DATA ACQUISITION

The moisture desorption data were acquired by the Vapor Sorption Analyzer (VSA). The following procedure was adopted for both JP and JS: approximately 2000 mg of sample were weighed in an analytical balance (M214AIH, BEL, Brazil), and ground with the aid of a mortar and a pestle. Before initiating the desorption test, the ground sample was placed inside the glass desiccator, with silica gel at the base, to decrease the a_w level of the sample. This step was interrupted until the sample reached around 0.9 a_w. Subsequently, around 1600 to 1800 mg of the sample were weighed in the stainless steel capsule of the VSA, using the equipment's own micro analytical balance. The equipment was programmed to obtain desorption data in the range of 0.9 to 0.1 a_w, through the Dynamic Vapor Sorption (DVS) method. The condition of equilibrium was arranged for a change in mass per change in time (trigger % dm/dt value) below 0.1 for two consecutive measures. The device was arranged to obtain the equilibrium data in 0.1 a_w intervals. After completing the analysis, the sample's dry mass was determined in the drying oven at 105°C. Desorption isotherms were obtained at 25, 35, 45, and 55°C, for both JP and JS.

2.4 DESORPTION ISOTHERMS MODELING

The monolayer moisture content (m_o) for each temperature analyzed (25, 35, 45 and 55ºC) was determined by linear regression using the linearized form of the BET equation (Eq. 1) (BRUNAUER; EMMER; TELLER, 1938). This model was used to calculate the desorption m_o of JP and JS, in the a_w range from 0.4 to 0.1.

$$\frac{a_w}{(1-a_w)m} = \frac{1}{m_o C} + \frac{(C-1)}{m_o C} a_w \qquad (1)$$

where: m = equilibrium moisture content (g H_2O/100 g dry basis); a_w = water activity (dimensionless); m_o = monolayer moisture content (g H_2O/100 g db); C = constant related to the sorption heat.

The following mathematical models (Eq. 2-7) (CHIRIFE; IGLESIAS, 1978; MAROULIS et al., 1988; CHOWDHURY; DAS, 2010) were tested to fit the desorption data of JP and JS at different temperatures:

GAB: $$m = \frac{m_o.c.k.a_w}{\left[(1-k.a_w).(1+(c-1).k.a_w\right]} \qquad (2)$$

Halsey: $$m = \left[\frac{-a}{\ln a_w}\right]^{\frac{1}{b}} \qquad (3)$$

Oswin: $$m = a.\left[\frac{a_w}{1-a_w}\right]^{b} \qquad (4)$$

Henderson: $$m = \left[\frac{-\ln(1-a_w)}{1-a_w}\right]^{\frac{1}{b}} \qquad (5)$$

Smith: $$m = b + a.\ln(1-a_w) \qquad (6)$$

Peleg: $$m = k_1.a_w^{n_1} + k_2.a_w^{n_2} \qquad (7)$$

where: m = equilibrium moisture content (g H_2O/100 g db); a_w = water activity (dimensionless); m_o = monolayer moisture content (g H_2O/100 g db); a, b, c, k_1, k_2, n_1, n_2 = model constants.

2.5 THERMODYNAMIC PROPERTIES CALCULATION

The net isosteric heat of desorption (q_{st}) or differential enthalpy of desorption (ΔH) was determined by Eq. 8, from the angular coefficient of the line obtained by the $\ln(a_w)$ versus 1/T correlation, for different levels of moisture. The isosteric heat of desorption (Q_{st}), in turn, was calculated by adding to q_{st} the latent heat of vaporization of pure water (λ_{vap}) (Eq. 9), at the mean temperature of the desorption processes (40ºC) (RIZVI, 2005).

$$q_{st} = -R\left[\frac{d(\ln a_w)}{d(1/T)}\right] \tag{8}$$

$$Q_{st} = q_{st} + \lambda_{vap} \tag{9}$$

where: R = gas constant (0.4619 kJ/kg.K); a_w = water activity (dimensionless); T = absolute temperature (K); λ_{vap} = latent heat of vaporization of pure water at the average of the temperatures considered in this study (2,405.1 kJ/kg at 40°C).

The change in differential entropy (ΔS) was calculated according to Eq. 10, from the linear coefficients ($\Delta S/R$) of the slope $\ln(a_w)$ versus 1/T correlation, for different levels of moisture. The Gibbs free energy was calculated from Eq. 11 (RIZVI, 2014).

$$-\ln(a_w)\big|_m = \frac{Q_{st}}{RT} - \frac{\Delta S}{R} \tag{10}$$

$$\Delta G = -RT\ln a_w \tag{11}$$

where: ΔS = change in differential entropy (kJ/kg.K); ΔG = Gibbs free energy (kJ/kg).

Moreover, the enthalpy–entropy compensation theory is a linear relationship between ΔH and ΔS. From the ΔH *versus* ΔS graphic, the isokinetic temperature (T_β) could be determined (Eq. 12). To validate the compensation theory, a statistical test is recommended and consists in the comparison between T_β and the the harmonic mean temperature (T_{hm}), obtained by the following equation (Eq. 13) (KRUG, HUNTER, GRIEGER, 1976).

$$\Delta H = \Delta G + T_\beta.\Delta S \tag{12}$$

$$T_{hm} = \frac{n}{\sum_{1}^{n}(1/T)} \tag{13}$$

where: T_β = the isokinetic temperature (K); T_{hm} = the harmonic mean temperature (K); n is the number of isotherms.

For the Q_{st} prediction, the following model was tested (Eq. 14), for JP and JS. This equation was proposed by Mulet et al. (1999).

$$Q_{st} = d.\exp(-g.m) + \lambda_{vap} \tag{14}$$

where: Q_{st} = isosteric heat of desorption (kJ/kg); m = equilibrium moisture content (g H_2O/100 g db); λ_{vap} = latent heat of vaporization of pure water; d and g are model parameters.

2.6 STATISTICAL ANALYSIS

The proximate composition results of JP and JS were assessed using Student's t-test, at 5% significance, for comparison of means. Nonlinear regression analysis was used to fit the mathematical models to the moisture desorption data for JP and JS. The Levenberg-Marquardt algorithm was used with a 10^{-6} convergence criterion. To evaluate the quality of the model fits, the coefficient of determination (R^2), the relative mean deviation (P) (Eq. 15) and the distribution of residues were used.

$$P = \frac{100}{n}\sum_{i=1}^{n}\frac{\left|m_{exp,i} - m_{pre,i}\right|}{m_{exp,i}} \tag{15}$$

where: $m_{exp,i}$ and $m_{pre,i}$ = ith observed and predicted equilibrium moisture contents, respectively, and n = number of observations.

3 RESULTS AND DISCUSSION

3.1 PROXIMATE COMPOSITION AND a_w FOR JP AND JS

Table 1 presents the centesimal composition for both JP and JS (in wet basis – wb), as well as the results for a_w at 25ºC. These findings corroborate to the results observed by Araújo and Pena (2021), whose proximate composition, for JP, was: 84.80 ± 0.26 g/100 g moisture, 0.43 ± 0.01 g/100 g ashes, 0.56 ± 0.03 g/100 g total lipids, 0.48 ± 0.05 g/100 g total proteins, 13.90 ± 0.03 g/100 g carbohydrates; and the a_w was 0.98 ± < 0.01 (25ºC). For JS, in turn, no studies have been found regarding its proximate composition.

Statistical analysis revealed significant differences ($p \leq 0.05$) between the proximate composition of JP and JS, except for total lipids. Moreover, the composition and physicochemical properties of JP and JS have evidenced their perishable nature, especially in function of the high contents of moisture and a_w, which may not ensure the microbial stability to JP and JS ($a_w > 0.6$) (SCOTT, 1957; RAHMAN, 2009).

Table 1. Proximate composition and a_w for JP and JS.

Properties	**Result***	
	JP	**JS**
Moisture (g/100 g)	$84.76^a \pm < 0.01$	$45.64^b \pm 0.2$
Ashes (g/100 g)	$0.29^b \pm 0.01$	$0.99^a \pm < 0.01$
Total lipids (g/100 g)	$0.34^a \pm 0.04$	$0.36^a \pm 0.02$
Total proteins (N x 6.25) (g/100 g)	$0.47^b \pm 0.03$	$3.11^a \pm 0.04$
Total carbohydrates (g/100 g)	$14.40^b \pm 0.03$	$49.90^a \pm 0.23$
a_w at 25ºC	$0.98^a \pm < 0.01$	$0.97^b \pm < 0.01$

*Mean of three replications ± standard deviation. Different letters at the same line indicate significant difference by the Student's t-test ($p \leq 0.05$).

3.2 MOISTURE DESORPTION ISOTHERMS

Figure 1 shows the moisture desorption isotherms for JP and JS at 25, 35, 45 and 55ºC, highlighting the effect of temperature. The equilibrium moisture content (EMC) of JP (Figure 1A) decreased relatively fast (exponentially) at ≈ 0.9-0.5 a_w, in contrast with the region below 0.5 a_w, in which a linear decrease was observed for all temperatures studied. The desorption isotherms from JS (Figure 1B), in turn, indicated that when the a_w value drops from 0.9 to 0.7, the isotherms undergo an exponential decrease. Below this a_w level, a linear behavior is evidenced. Thus, under these conditions (90% ≥ RH ≥ 50% for JP, and 90% ≥ RH ≥ 70% for JS), the products will be more susceptible to moisture loss and, therefore, small changes in RH lead to great changes in EMC. According to Brunauer et al. (1938) classification, the JP isotherms (Figure 1A) presented a type III behavior, which is a characteristic of foods rich in soluble solids, while JS isotherms (Figure 1B) showed type II behavior, indicating the presence of protein components. However, JP isotherms followed the type II - more to the solution-like behavior, while JS isotherms followed the behavior of type II - more to the Langmuir-like, according to the quantitative criteria established by Yanniotis and Blahovec (2009). These results are in agreement with JP and JS composition, found in Table 1.

Regarding JP isotherm shapes, similar trends were evidenced in freeze-dried jambolan (SANTANA et al., 2014), microwave-convective hot air dried jambolan (PAUL; DAS, 2019) and fresh jambolan (BISWALL et al., 2017). On the other hand, jackfruit seeds (PRETTE et al., 2013) and chinoa seeds (ASLAN-TONTUL, 2020) followed a sigmoid format, as observed in JS.

At constant a_w, the EMC decreases as the temperature increases, which indicates that both JP (Figure 1A) and JS (Figure 1B) are less hygroscopic at high temperatures. This phenomenon is attributed to the excitation states of molecules due to temperature increasing, which increases their distance and; therefore, decreases the attractive forces between them (HASSINI et al., 2015). Additionally, it can be stated that the JP will be microbiologically stable ($a_w < 0.6$) (SCOTT, 1957) when its moisture content is less than 28.04 g H_2O/100 g db, if stored at 25°C. For JS, this stability corresponds to a moisture content value of less than 14.55 g H_2O/100 g db (25ºC); half of the observed value for JP. However, if the temperature is extrapolated, the limiting moisture content for JP continuously drops to 25.84, 23.26, and 20.16 g H_2O/100 g db, at 35, 45 and 55ºC, respectively. Moreover, the JS's limiting moisture content value will drop to 13.68, 12.30, and 10.60 g H_2O/100 g db, if the temperature is equivalent to 35, 45 and 55ºC, respectively.

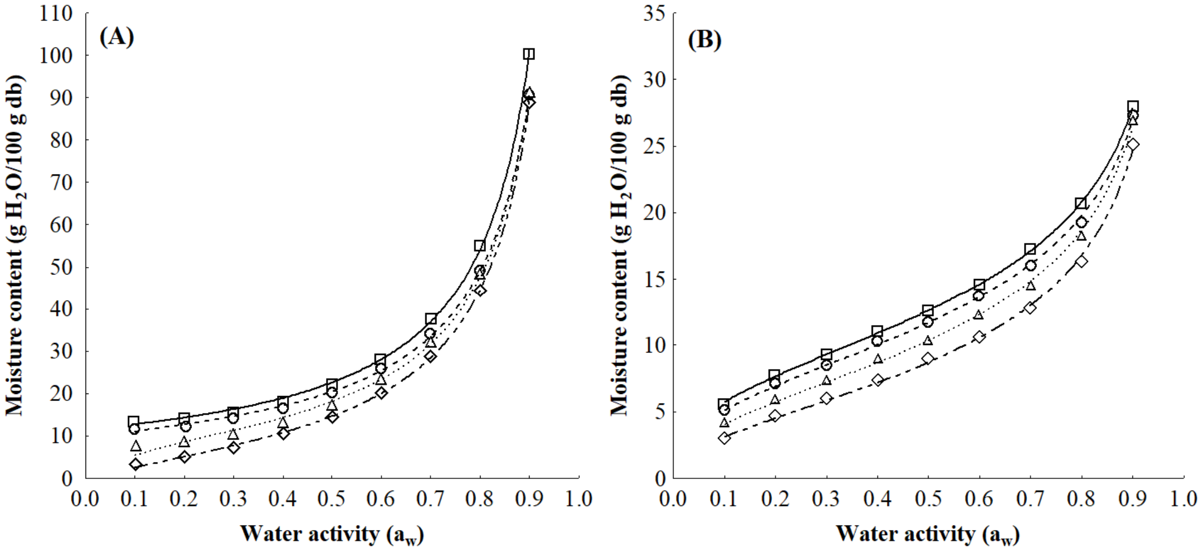

Figure 1. Moisture desorption isotherms of JP (A) and JS (B), at 25°C (□), 35ºC (○), 45ºC (△), 55ºC (◇), and predicted data from the GAB (A) and Oswin (B) models, at 25ºC (——), 35ºC (- - - - -), 45ºC (············), and 55ºC (— — — —).

Still, according to isotherms (Figure 1), JP is more hygroscopic than JS, at all a_w and temperature range studied. The minimum EMC variation observed between JP and JS isotherms, for a same a_w level, was 6.12 g H_2O/100 g db (25°C), 5.03 g H_2O/100 g db (35°C), 2.62 g H_2O/100 g db (45°C) and 0.45 g H_2O/100 g db (55ºC), in the range of a_w from 0.1 to 0.3. The greatest variations occurred in a_w levels above 0.6, when there were observed EMC variations from 13.49 to 72.21 g H_2O/100 g db at 25ºC, 12.16 to 63.43 g H_2O/100 g db at 35ºC, 10.96 to 64.33 g H_2O/100 g db at 45ºC and 9.57 to 63.80 g H_2O/100 g db at 55ºC. The EMC variation increased with a_w increasing, and it was higher at 25°C.

3.3 MATHEMATICAL MODELING

The fits of the linearized form of the BET model (Eq. 1) to the experimental data generated desorption monolayer moisture content (m_o) values of 10.56 g H_2O/100 g db (25ºC), 9.75 g H_2O/100 g db (35ºC), 8.06 g H_2O/100 g db (45ºC) and 7.84 g H_2O/100 g db (55ºC), for JP ($R^2 > 0.95$). In turn, desorption m_o values for JS corresponded to 7.08 g H_2O/100 g db (25ºC), 6.60 g H_2O/100 g db (35ºC), 5.86 g H_2O/100 g db (45ºC) and 5.08 g H_2O/100 g db (55ºC), with $R^2 > 0.99$. The m_o values decreased with temperature increasing. This behavior is already expected, since the increase in temperature elevates the energy level of water molecules and promotes the detachment between the interaction sites of water and solute, thus decreasing the stability of water molecules bonding and resulting in the reduction of EMC at a given a_w (PALIPANE; DRISCOLL, 1993; PAHLEVANZADEH; YAZDANI, 2005).

The behavior observed for m_o diverged from the study of Santana et al. (2014), who found values of m_o in the order of 7.64 g H_2O/100 g db at 25ºC and 12.08 g H_2O/100 g db at 35ºC for the freeze-dried jambolan. The authors suggested that the unusual behavior occurred probably due to modifications in the three-dimensional rearrangement, where the polar groups became available, and the solubility increased with temperature increasing. For practical purposes, the value of m_o for the desorption process indicates the moisture content limit, from which it is no longer necessary to extend the drying stage for JP and JS, to avoid unnecessary power consumption.

As previously mentioned, the suitability of the fits was assessed by means of R^2, P and residual plots, for the desorption isotherms of JP and JS, at different temperatures (Table 2). According to the statistical results, the equations that best fitted to the experimental data were the GAB model ($R^2 > 0.99$, $P < 6.4\%$, random residues) for JP and the Oswin model ($R^2 > 0.99$, $P < 2.3\%$, random residues) for JS.

Generally, models with a P value of less than 10% are considered acceptable for practical purposes (PENG et al., 2007). However, to ensure that the suitable model is able to express a given phenomenon, the residues' dispersion should be verified for the different models; since, even if the statistical parameters show good fits, the model can be ineffective if it presents a biased residue distribution (BOWMAN; ROBINSON, 1990). Thus, these models (GAB - JP, Oswin - JS) have been chosen not only by their lowest P values (at average), but also by their random distribution of residues, at all temperatures studied. It is worth mentioning that, although Peleg's equation was not chosen to represent the desorption data, it also showed good fits for both JP and JS. The Peleg model is an equation with four parameters, which justifies its good fit. However, Peleg's equation has a more difficult mathematical solution.

Table 2. Mathematical modeling parameters for the desorption process of JP and JS.

Model / Parameter		**Jambolan pulp (JP)**				**Jambolan seed (JS)**			
		Temperature (°C)							
		25	**35**	**45**	**55**	**25**	**35**	**45**	**55**
GAB	m_o	11.5	10.49	10.33	10.18	7.90	7.10	6.06	5.15
	C	$2.2x10^5$	170.27	8.22	2.74	24.20	25.57	21.76	14.50
	K	0.990	0.983	0.99	0.99	0.80	0.82	0.86	0.88
	R^2	0.994	1.000	0.998	1.000	0.998	0.995	0.997	0.997
	P	2.62	2.62	6.32	3.98	1.66	2.70	3.02	3.59
	Residues	R	R	R	R	B	R	R	B
Halsey	A	39.03	34.80	19.43	11.27	174.27	115.66	54.58	27.33
	B	1.28	1.29	1.16	1.04	2.21	2.11	1.90	1.72
	R^2	0.996	0.997	0.999	0.999	0.989	0.992	0.994	0.993
	P	7.46	6.22	4.19	10.25	6.08	5.80	5.69	7.84
	Residues	B	B	R	B	B	B	B	B
Oswin	A	23.14	20.96	17.75	14.38	12.63	11.71	10.36	8.72
	B	0.66	0.66	0.74	0.83	0.36	0.38	0.43	0.47
	R^2	0.982	0.984	0.996	1.000	1.000	0.999	0.999	0.999
	P	15.20	14.42	11.01	6.00	0.69	1.22	1.88	2.25
	Residues	B	B	B	B	R	R	R	R
Henderson	A	0.06	0.06	0.10	0.14	0.01	0.01	0.02	0.05
	B	0.82	0.82	0.71	0.62	1.72	1.63	1.41	1.24
	R^2	0.946	0.949	0.977	0.989	0.982	0.975	0.973	0.977
	P	26.77	26.44	26.27	27.09	7.08	8.32	10.66	11.51
	Residues	B	B	B	B	B	B	B	B
Smith	A	0.27	0.14	-4.21	-7.60	5.52	4.83	3.50	2.21
	B	38.08	34.47	36.55	36.84	9.71	9.51	9.73	9.44
	R^2	0.928	0.932	0.939	0.937	0.996	0.994	0.993	0.991
	P	24.35	23.47	31.03	48.60	3.55	4.12	4.23	5.38
	Residues	B	B	B	B	R	R	R	B
Peleg	k_1	156.39	26.15	145.32	36.07	17.55	24.95	27.52	14.66
	n_1	7.01	0.43	7.53	1.27	0.51	7.91	7.92	0.72
	k_2	26.33	142.70	26.68	162.56	21.66	17.28	15.72	28.86
	n_2	0.36	7.42	0.69	9.89	6.28	0.56	0.60	8.87
	R^2	0.997	0.997	0.998	0.999	0.999	0.998	0.999	0.999
	P	5.94	6.53	7.41	8.55	1.20	2.18	1.94	2.01
	Residues	R	R	R	R	R	R	R	R

R^2: coefficient of determination; P: standard error deviation; B: biased; R: random.

GAB equation has a theoretical background, and it is a refinement of the Langmuir and BET theories of physical adsorption. This model is suitable for analyzing many pulp fruits, including jackfruit pulp (PRETTE et al., 2013), murtilla berry (AH-HEN et al., 2014), jambolan fruits (BISWALL et al., 2017), and Italia grapes (KHIARI et al., 2020). Otherwise, Oswin's model has been widely fitted to foodstuffs, displaying good suitability in seeds and grains, such as jackfruit seeds (PRETTE et al., 2013) and prickly pear seeds (HASSINI et al., 2015).

3.4 THERMODYNAMIC PROPERTIES

The most suitable models (GAB - JP, Oswin - JS) (Table 2) were used to calculate the thermodynamic properties of moisture desorption for JP and JS. The isosteric heat of desorption (Q_{st}) for JP and JS are presented in Figure 2A. The Q_{st} values decreased continually with EMC increasing until 75 g H_2O/100 g db for JP and 30 g H_2O/100 g db for JS, from which the Q_{st} values were close to the latent heat of vaporization of pure water at 40°C (2,405.1 kJ/kg). Still, it can be assumed that Q_{st} values were higher for JP than JS, at a same EMC. Such differences were in the order of 250.82 kJ/kg at 25 g H_2O/100 g db, and increased as EMC regressed; reaching variations around 870.37 kJ/kg, at 15 g H_2O/100 g db.

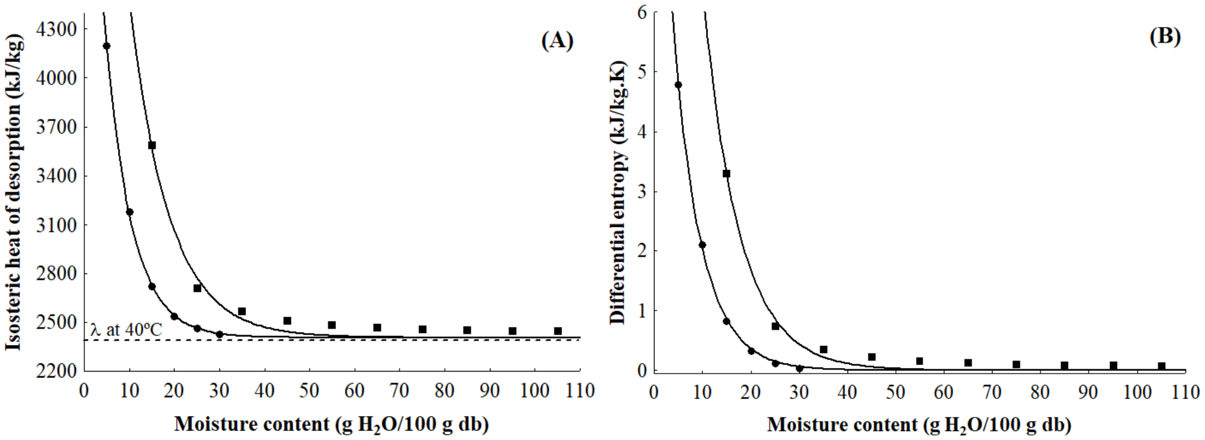

Figure 2. Isosteric heat of sorption (A) and differential entropy (B) for the desorption process of JP (■) and JS (●). Model fitting (——).

The highest Q_{st} values were observed at the lowest EMC values, in which the monolayer values are situated. In this region, the water molecules and the food constituents are more strongly bound, and it corresponds to the highest binding energies for water removal, since water occupies the most active available sites (KHIARI et al., 2020). The increasing trend of Q_{st} with EMC decreasing was also evidenced in fresh pulp and seed of jackfruit (PRETTE et al., 2013), in prickly pear seeds (HASSINI et al., 2015) and in ryegrass seeds (ZEYMER et al., 2020).

The Q_{st} expresses the binding strength between the moisture-sorbent, and it is required in the designing of equipment for dehydration processes (CHOWDHURY; DAS, 2010). In the drying industry, reaching the highest Q_{st} values are undesirable in most operations as it increases the cost within the processes (ASLAN-TONTUL, 2020).

The variation of Q_{st} with EMC was fitted by the previous equation (Eq. 14), and resulted in the equations below (Eq. 16 for JP and Eq. 17 for JS), with a confidence level of 95% ($p \leq 0.05$). In agreement with the good fits appointed by the modeling (lines) in Figure 2A, other authors observed good fits for the cassava flour data (ARAÚJO; PENA, 2020).

$$Q_{st} = 6{,}668.2 \text{ x } \exp(-0.116 \text{ x } m) + 2{,}405.1 \quad (R^2 = 0.975) \tag{16}$$

$$Q_{st} = 4{,}231.1 \text{ x } \exp(-0.172 \text{ x } m) + 2{,}405.1 \quad (R^2 = 0.999) \tag{17}$$

Differential entropy (ΔS) for desorption process of JP and JS increases with EMC decreasing (Figure 2B). Once again, the ΔS data displayed a strong dependence on EMC. It ranged from 0.08 to 3.30 kJ/kg.K for JP and from 0.04 to 4.78 kJ/kg.K for JS. These results are in agreement with the expected for ΔS, which is a measure of the ordering change, and presents lower results when the molecular movement is more restricted, that is, when the product has higher EMC. Besides, at higher a_w, sites are covered with water molecules, implying in less mobility for the water molecules (EIM et al., 2011); also, ΔS of a material is proportional to the number of available sorption sites at a specific energy level (NASCIMENTO et al., 2019), and as desorption progresses, more sites are exposed and become available. Similar results were found by Zeymer at al. (2018), in the desorption process of rice, which ΔS values varied from 0.22 to 4.64 kJ/kg.K, for an EMC variation from 21.5 to 2.6 g H_2O/100 g db.

At the same EMC, ΔS values were higher for JP than JS (Figure 2B). These differences were equal to 0.63 kJ/kg.K (at 25 g H_2O/100 g db), and increased to 2.48 kJ/kg.K (at 15 g H_2O/100 g db). According to the study, the variation of desorption ΔS with EMC was best fitted by the exponential model, which can be visualized in Eq. 18 for JP and Eq. 19 for JS, with a confidence level of 95% ($p \leq 0.05$). These models displayed good suitability to the ΔS data, with a slightly better adjustment for JS over JP (Figure 2B). Hassini et al. (2015) also proposed an exponential model to represent the desorption ΔS as function of EMC in prickly pear seeds.

$$\Delta S = 24.52 \text{ x } \exp(-0.13 \text{ x } m) \quad (R^2 = 0.986) \tag{18}$$

$$\Delta S = 11.34 \text{ x } \exp(-0.17 \text{ x } m) \quad (R^2 = 0.999) \tag{19}$$

From a thermodynamic point of view, Gibbs free energy (ΔG) may be used as indicative of the water and sorbent affinity, and further provide a criterion whether water sorption is a spontaneous or non-spontaneous process, depending on the sign (negative or positive) of the ΔG values (ALPIZAR-REYES, 2017). The values of ΔG for JP (Figure 3A) and JS (Figure 3B) suggest that the moisture desorption processes for both products are non-spontaneous (positive ΔG). This behavior is already expected for desorption processes, since it requires the addition of energy to occur (endergonic reaction) (OULAHNA et al., 2012). Non-spontaneous desorption processes were also found in rice (ZEYMER et al., 2018) and in crambe fruits (OLIVEIRA et al., 2017).

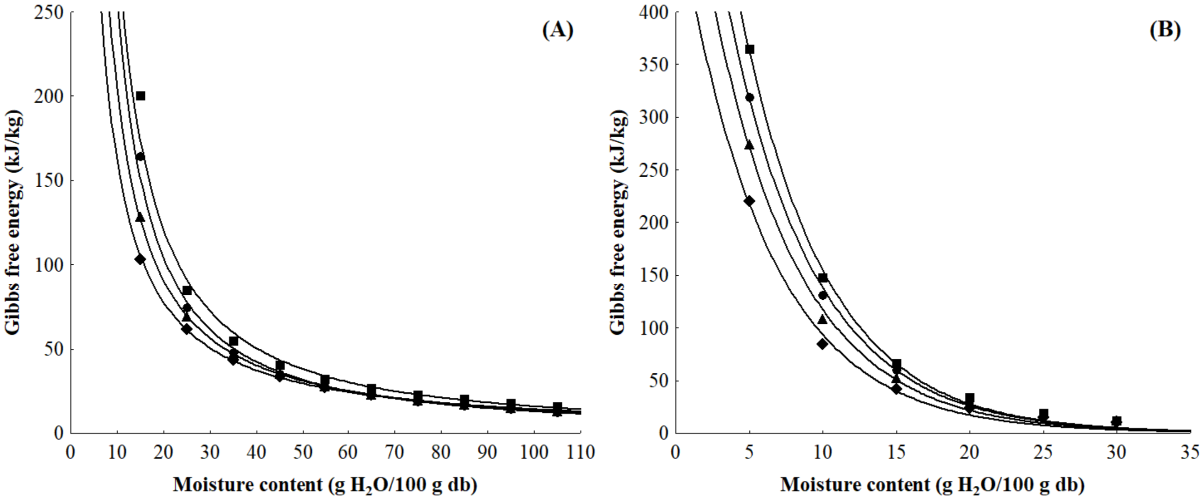

Figure 3. Gibbs free energy for the desorption process of JP (A) and JS (B), at 25°C (■), 35ºC (●), 45ºC (▲), and 55ºC (◆). Model fitting (——).

In general, it was observed a decrease in the ΔG values as temperature increases (Figure 3), for a same EMC value, which indicate that the desorption processes of JP and JS are less viable at higher temperatures. This behavior was more evident for JP than JS, since JP is more hygroscopic (Figure 1). Also, the results showed an increasing of ΔG while EMC decreases, and the effect of temperature was more expressive in the regions below 35 g H_2O/100 g db for JP (Figure 3A) and 15 H_2O/100 g db for JS (Figure 3B). This increasing of ΔG while EMC decreased also occurred in chinoa grains, in the range of 15-35ºC (ASLAN-TONTUL, 2020).

Furthermore, the variation of desorption ΔG followed a power model for JP and an exponential model for JS, at 95% confidence level ($p \leq 0.05$). The lines presented in Figures 3 represent the fits of the referred models to the ΔG data. These good adjustments ($R^2 > 0.99$) generated the models present in Eq. 20-23, for JP; and Eq. 24-27, for JS.

$$\Delta G = 5{,}270.7 \times m^{-1.26} \qquad (25^\circ C) \quad (R^2 = 0.992) \qquad (20)$$

$$\Delta G = 5{,}058.5 \times m^{-1.30} \qquad (35^{\circ}C) \qquad (R^2 = 0.998) \tag{21}$$

$$\Delta G = 2{,}988.9 \times m^{-1.17} \qquad (45^{\circ}C) \qquad (R^2 = 1) \tag{22}$$

$$\Delta G = 1{,}839.2 \times m^{-1.06} \qquad (55^{\circ}C) \qquad (R^2 = 1) \tag{23}$$

$$\Delta G = 850.70 \times \exp(-0.17 \times m) \qquad (25^{\circ}C) \qquad (R^2 = 0.998) \tag{24}$$

$$\Delta G = 729.19 \times \exp(-0.17 \times m) \qquad (35^{\circ}C) \qquad (R^2 = 0.998) \tag{25}$$

$$\Delta G = 627.40 \times \exp(-0.17 \times m) \qquad (45^{\circ}C) \qquad (R^2 = 0.995) \tag{26}$$

$$\Delta G = 505.81 \times \exp(-0.17 \times m) \qquad (55^{\circ}C) \qquad (R^2 = 0.993) \tag{27}$$

The enthalpy-entropy compensation theory was assessed to verify the relationship between physical and chemical phenomena that might occur in the moisture desorption process of JP and JS (Figure 4). This theory states that, for minimizing changes in the free energy of these phenomena, the compensation arises with a change in the ΔH or ΔS values, through the interaction nature between solvent and solute. Thus, the ΔH and ΔS relationship follows a linear behavior, for a specific reaction (HERCIGONJA; RAKIC, 2015).

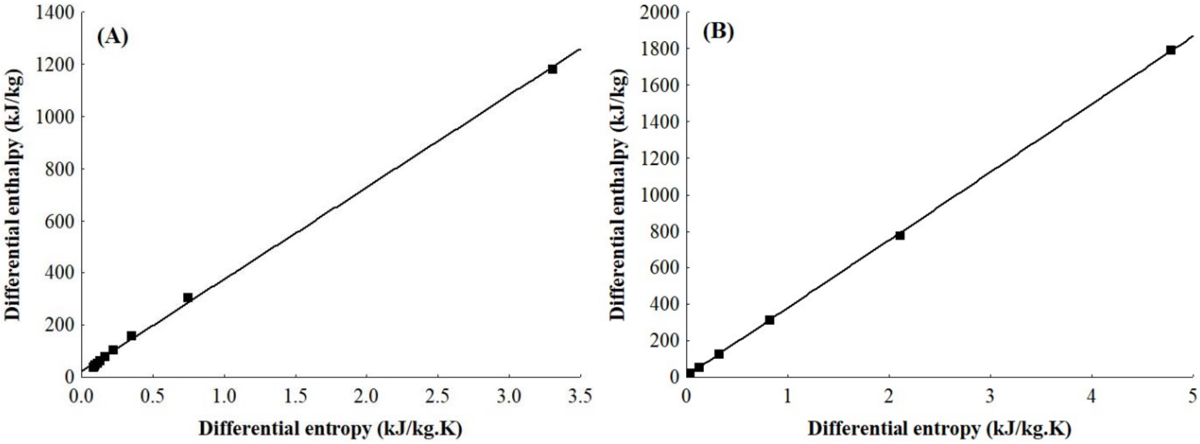

Figure 4. Correlation between differential enthalpy and differential entropy for the desorption process of JP (A) and JS (B). Model fitting (——).

The isokinetic temperature (T_β) was 353.5 K for JP ($\Delta H = 21.3 + 353.5\Delta S$; $R^2 = 0.999$) (Figure 4A), and 371.9 K for JS ($\Delta H = 7.41 + 371.9\Delta S$; $R^2 = 0.999$) (Figure 4B). The harmonic mean temperature (T_{hm}) (calculated by Eq. 13) for both JP and JS was found to be $T_{hm} = 312.8$ K. Then, it was observed that $T_\beta \neq T_{hm}$, and this difference corroborates to the enthalpy–entropy compensation theory. The results showed that $T_\beta > T_{hm}$, and it can be assumed that the desorption processes of both JP and JS are, therefore, enthalpy-driven mechanisms (KRUG et a., 1976). Different authors have applied the compensation theory to assess moisture sorption processes of prickly pear seeds (HASSINI et al., 2015) and figs (HSSAINI et al., 2020), observing that these processes were also controlled by enthalpy.

4 CONCLUSION

The desorption processes of fresh jambolan pulp (JP) and jambolan seed (JS) were studied, and the latter one for the first time. JP isotherms were classified as type II - more to the solution-like, while JS isotherms followed the behavior of type II - more to the Langmuir-like, in the temperature range used (25-55°C). The equilibrium moisture content (EMC) of both JP and JS were influenced by temperature, but JP is a product much more hygroscopic than JS. According to the monolayer moisture content (m_o), to avoid an unnecessary energy consumption in the drying process, the moisture content should not reach values below 7.8 g H_2O/100 g db for JP and 5.1 g H_2O/100 g db for JS. GAB and Oswin equations described with the best accuracy the desorption isotherms of JP and JS, respectively. The energies involved in the desorption process are higher for JP than JS, indicating that it is easier to dry JS. Still, the compensation theory has proven that the desorption phenomena for both pulp and seed of jambolan are enthalpy-driven mechanisms.

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Capítulo IV

ARTIGO 2 - INFLUENCE OF PROCESS CONDITIONS ON THE MASS TRANSFER OF OSMOTICALLY DEHYDRATED JAMBOLAN FRUITS

PUBLISHED IN THE JOURNAL ENTITLED **FOOD SCIENCE AND TECHNOLOGY (CAMPINAS)**

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Submitted in 09/08/2020. Accepted in 15/09/2020. Published online in: 11/06/2021.

ABSTRACT

Jambolan (*Syzygium cumini*) is a tropical fruit rich in anthocyanin pigments, but its fragile skin and pulp present low protection against physical damages and microorganisms. In this sense, a preservative technology, as osmotic dehydration (OD), was studied to investigate the impact of some variables over this process. At first, fruits of jambolan were submitted to physical and physical-chemical analysis. Furthermore, whole fruits underwent OD following a fractional factorial design. The influence of the process variables: temperature (20-50ºC), sucrose concentration (30-60%), pressure (10-90 kPa), vacuum pulse time (5-15 min), calcium lactate concentration (0-4%) and number of vacuum pulse (1-3) was assessed on water loss (WL), solid gain (SG) and weight reduction (WR). In general, biometric analysis showed positive and significant correlation among the physical characteristics of jambolan. Physical-chemical assay demonstrated that the fresh fruit presents a potential source of bioactive compounds. The screening design showed that temperature, sucrose concentration, calcium lactate concentration and pressure affected the WL in jambolan. On the other hand, vacuum pulse time and number of vacuum pulse showed no influence on the WL, SG and WR; therefore, these variables must be fixed at the most economically viable level for any further trials.

Practical Application: The present work can improve the control of the unitary operation (dehydration) in OD of jambolan.

Keywords: dehydration, morphometric characteristic, design of experiments.

1 INTRODUCTION

The Amazon hosts a large biological diversity, including great number of non-native plant species, which have been successfully adapted to the Amazonian environmental conditions (BRITO et al., 2017). Native from India, jambolan, janun or black plum (*Syzygium cumini*) fruits are ellipsoid berries from the *Myrtaceae* family, which present themselves as purplish black, with a sour taste at the ripe stages of maturation (SERAGLIO et al., 2018). This tropical fruit is also rich in anthocyanin pigments, especially in its peel (SARI et al., 2012).

However, the relatively short shelf life of fresh fruits after harvesting indicates the need for developing an efficient and cheap preservation process. Besides, the rising search for products with similar sensory and nutritional properties to fresh fruits also stimulates the food industry to seek for preservation methods in foods (SRIDEVI; GENITHA, 2012). In this scenario, osmotic dehydration (OD) is growing as a popular technique for obtaining processed fruits, improving their quality and stability (AHMED; QAZI; JAMAL, 2016).

Commonly, OD is a pre-treatment prior to air-drying. This technique is based on the immersion of fruits in hypertonic solution for partial water removal. The driving force for its removal is the difference in osmotic pressure between the fruit and the hypertonic solution. The fruit's complex cell structure acts as a semi-permeable membrane, generating extra resistance to water diffusion within the fruit. This mass transfer depends on some factors, such as the product's geometry, temperature, addition of salts and concentration of the osmotic solution, among others (TORREGIANI, 1993).

Traditionally, the osmotic process is employed at atmospheric pressure; however, in recent decades, it has been combined with other innovative techniques, such as vacuum application, which can enhance its efficiency and improve the quality of the product (AHMED; QAZI; JAMAL, 2016). Some researchers have studied the use of vacuum in OD to accelerate mass transfer in products such as guavas (CORRÊA et al., 2010) and tomato (CORRÊA et al., 2015), among other works involving the study of vacuum pulse time (ITO et al., 2007) and number of vacuum pulse (ZAPATA; CIRO; MARULANDA, 2016).

Due to the perishability of jambolan, especially by the fragility of its skin and pulp, that offer low protection against mechanical damages and infectious agents, harvest and post-harvest management are essential for extending the fruit's shelf life. In this sense, some studies have assessed the drying of jambolan fruit using different techniques, including air forced drying (KAPOOR; RANOTE; SHARMA, 2015), foam mat drying (CARVALHO et al., 2017) and spray-drying (SINGH; PASWAN; RAI, 2019). However, no studies have been reported regarding OD techniques for jambolan fruits.

In this context, the objectives of this work were to assess the physical and physical-chemical characterization of jambolan, as well as to use a fractional factorial design for investigating the influence of variables as temperature, sucrose concentration, pressure, vacuum pulse time, calcium lactate concentration and number of vacuum pulse on the responses water loss, solid gain and weight reduction, during the osmotic dehydration of this fruit.

2 MATERIAL AND METHODS

2.1 RAW MATERIAL

Mature fruits of jambolan (*Syzygium cumini*) were collected from a tree located at the Federal University of Para, in the city of Belem, state of Para, Brazil (latitude 1º28’ S, longitude 48º29' W), during the rainy season, in December 2019. The regional climate is tropical monsoon and the average annual air temperature is 26°C (ranging from 22ºC to 31.5ºC). The average annual precipitation is 3000 mm, including daily precipitations above 25 mm (Instituto Nacional de Meteorologia, 1992).

The fruits were transported in polystyrene isothermal boxes to the Laboratory of fruits, at the Federal University of Para. The jambolan fruits were selected based on the maturation stage, absence of physical damages and rot; then, the selected fruits were rinsed in running water, sanitized by immersion in hypochlorite solution at 20 mg/L, during 15 minutes, followed by rinsing. One hundred units of these fruits were separated for biometric analysis, shortly after harvesting. In addition, the sanitized fruits were packaged in small quantities (≈ 100 g) in polypropylene pots coated with aluminum foil, which were frozen in a vertical ultra-freezer (CL374-80, Coldlab, Brazil) at -70°C. Then, the frozen fruits were stored at -18°C, until physical-chemical and osmotic dehydration analysis.

2.2 BIOMETRIC ANALYSIS

The biometric analysis was performed individually with 100 fresh jambolan fruits. The morphometric characteristics (longitudinal and equatorial diameter) of the whole fruit were determined using a caliper (235, WESTERN, China) with 0.05 mm resolution and the results were expressed in mm. The whole fruit, seed and pulp weight (in grams) were obtained by individual weighing in a digital precision balance (M214AIH, BEL, Brazil), with 0.0001 g accuracy. The yield, expressed as percentage, was calculated by the Equation 1.

$$Y = \left(\frac{W_c \times 100}{W_f} \right) \tag{1}$$

where Y = yield (%); W_c = weight of the pulp (g); W_f = weight of the whole fruit (g).

2.3 PHYSICAL-CHEMICAL, CHEMICAL AND COLOR ANALYSIS

Jambolan fruits (epicarp + mesocarp) were submitted to the following analyzes: moisture (n. 920.151), ashes (n. 940.26), total proteins (n. 920.152), total lipids (n. 922.06), pH (n. 943.15), and total titratable acidity (n. 942.15). All measurements were performed according to the Association of Official Analytical Chemists (2002) methodology. The carbohydrates were calculated by the difference between 100 and the sum of the percentage of moisture, total proteins, total lipids and ashes (Food and Agriculture Organization of the United Nations, 2003). Meanwhile, reducing sugars was determined by dinitrosalicylic acid reagent (MILLER, 1959); monomeric anthocyanins were determined according to the method of Lees and Francis (1972) and total phenolics using the Folin–Ciocalteu method (SINGLETON; ROSSI, 1965). The monomeric anthocyanins and total phenolics were expressed as $mg.100^{-1}g$ and as gallic acid equivalents ($mg\ GAE.100^{-1}g$), respectively. Water activity (a_w) was measured at 25ºC using a thermohygrometer (Aqualab 3TE, Decagon, USA). Color parameters for L* (lightness), a* (redness\greenness), b* (yellowness\blueness), C* (chroma – color intensity) and h° (hue angle – values of 0°, 90°, 180° and 270° denote pure red, pure yellow, pure green, and pure blue colors, respectively) were measured using a digital colorimeter (Minolta CR-400, Konica, Japan) calibrated with white plate, using the CIE standard D65 illuminant and CIE 1964 observed (10° visual field). All data were obtained by triplicate analyses and expressed as mean ± standard deviation.

2.4 FRACTIONAL FACTORIAL DESIGN

A fractional factorial design was applied to study the main effects of the independent variables temperature (T), sucrose concentration (SC), pressure (P), vacuum pulse time (PT), calcium lactate concentration (CC) and number of vacuum pulse (N) on the dependent variables water loss (WL), solid gain (SG) and weight reduction (WR); in the osmotic dehydration (OD) process of the jambolan. The OD followed a 2^{6-2} design with 19 assays: 16 linear assays (at -1 and +1 levels) and 3 assays in the central point (all independent variables at level 0). The aim of this technique is to reduce the number of experimental runs when several variables are analyzed simultaneously. Thus, fractional designs are efficient to estimate the main effects of each variable studied (ANTONY, 2014). The experimental design is showed in the first seven columns of Table 1.

Table 1. Fractional factorial design matrix with the original and coded values of independent variables and the experimental results of the dependent variables.

Run	Independent variables (coded and original values)[a]						Dependent variables (response values)		
	x_1	x_2	x_3	x_4	x_5	x_6	**WL**	**SG**	**WR**
1	-1 (20)	-1 (30)	-1 (10)	-1 (5)	-1 (0)	-1 (1)	-1.06	-4.51	3.45
2	1 (50)	-1 (30)	-1 (10)	-1 (5)	1 (4)	-1 (1)	21.20	3.87	17.33
3	-1 (20)	1 (60)	-1 (10)	-1 (5)	1 (4)	1 (3)	9.54	4.68	4.86
4	1 (50)	1 (60)	-1 (10)	-1 (5)	-1 (0)	1 (3)	12.28	2.48	9.81
5	-1 (20)	-1 (30)	1 (90)	-1 (5)	1 (4)	1 (3)	4.93	2.31	2.62
6	1 (50)	-1 (30)	1 (90)	-1 (5)	-1 (0)	1 (3)	7.70	-5.11	12.81
7	-1 (20)	1 (60)	1 (90)	-1 (5)	-1 (0)	-1 (1)	5.55	0.70	4.86
8	1 (50)	1 (60)	1 (90)	-1 (5)	1 (4)	-1 (1)	20.53	1.85	18.69
9	-1 (20)	-1 (30)	-1 (10)	1 (15)	-1 (0)	1 (3)	6.38	1.47	4.91
10	1 (50)	-1 (30)	-1 (10)	1 (15)	1 (4)	1 (3)	14.65	3.26	11.39
11	-1 (20)	1 (60)	-1 (10)	1 (15)	1 (4)	-1 (1)	10.20	3.46	6.74
12	1 (50)	1 (60)	-1 (10)	1 (15)	-1 (0)	-1 (1)	20.71	7.09	13.62
13	-1 (20)	-1 (30)	1 (90)	1 (15)	1 (4)	-1 (1)	2.73	-0.68	3.41
14	1 (50)	-1 (30)	1 (90)	1 (15)	-1 (0)	-1 (1)	9.72	0.51	9.21
15	-1 (20)	1 (60)	1 (90)	1 (15)	-1 (0)	1 (3)	1.93	-1.25	3.18
16	1 (50)	1 (60)	1 (90)	1 (15)	1 (4)	1 (3)	21.35	2.85	18.49
17	0 (35)	0 (45)	0 (50)	0 (10)	0 (2)	0 (2)	8.10	0.47	7.63
18	0 (35)	0 (45)	0 (50)	0 (10)	0 (2)	0 (2)	7.16	0.59	6.57
19	0 (35)	0 (45)	0 (50)	0 (10)	0 (2)	0 (2)	7.53	-0.25	7.79

[a] Values between parentheses are the real forms of the variables; x_1 = temperature (°C); x_2 = sucrose concentration (%); x_3 = pressure (kPa); x_4 = vacuum pulse time (min); x_5 = calcium lactate concentration (%); x_6 = number of vacuum pulse; WL = water loss (%); SG = solid gain (%); WR = weight reduction (%).

2.5 OSMOTIC DEHYDRATION

The OD runs were carried out in a system composed by a mini glass reactor that has a jacketed system to maintain the temperature controlled during the process (Figure 1). The apparatus was also sealed to keep sub-atmospheric pressures (vacuum pulse). A vacuum pump (131, Prismatec, Brazil) with a pressure gauge was coupled to the system to ensure pressure control. The temperature of the OD process was controlled using an ultra-thermostatic bath (Q214M2, Quimis, Brazil), which circulated the fluid into the jacketed apparatus. The OD solution circulation was performed using a magnetic bar inside the reactor and placing it over a magnetic stirrer (Q-261, Quimis, Brazil) to guarantee constant stirring. At the circulation speed used, the surface layer around the sample was constantly renewed. The osmotic solutions were prepared according to the fractional factorial design by dissolving sucrose and calcium lactate to distilled water.

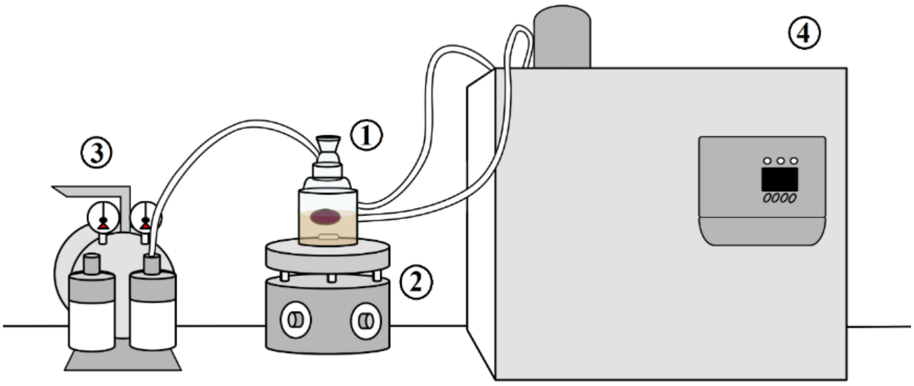

Figure 1. System used in the osmotic dehydration of jambolan composed by a mini glass reactor (1), a magnetic stirrer (2), a vacuum pump (3) and an ultra-thermostatic bath (4).

Whole fruits of jambolan were defrost at 5ºC and weighed in the analytical balance at room temperature (≈ 25ºC). After that, the fruits were immersed in the osmotic solution at working temperature and concentration; then, the system was closed and submitted to vacuum pulse, as defined by the fractional design (Table 1). Each assay was performed using the fruit mass:osmotic solution ratio of 1:10 (m/v) in order to ensure the constant osmotic solution concentration during the process.

Each treatment of the experiment was subjected to specific pressure and vacuum pulse time. A relaxation time of 15 min at atmospheric pressure was done between the numbers of pulses. At the end of pressure conditions, the system's vacuum was interrupted and when the total process time (90 min) was finished, the samples were removed from the osmotic solution, rinsed with 10 mL of distilled water and dried for 10 seconds in absorbent paper to remove water and sucrose/calcium lactate in excess from the surface. The osmo-dehydrated samples were weighed in the analytical balance and the moisture was determined before and after the OD process.

2.6 MASS TRANSFER DETERMINATION

The responses (dependent variables) WL, SG and WR were calculated as described by Sridevi and Genitha (2012), according to Equations 2-4:

$$WL(\%) = \frac{(W_0 - W_t)}{M_0} \times 100 \tag{2}$$

$$SG(\%) = \frac{(S_t - S_0)}{M_0} \times 100 \tag{3}$$

$$WR(\%) = \frac{(M_0 - M_t)}{M_0} \times 100 \tag{4}$$

where, M_0 = initial mass of the sample (g); M_t = mass of the sample (g) after dehydration; W_0 = initial water mass of the sample (g); W_t = water mass of the sample (g) after dehydration; S_0 = initial dry mass of the sample (g) and S_t = dry mass of the sample (g) after dehydration.

2.7 STATISTICAL ANALYSIS

The fruit's biometric data were analyzed by the statistical adjustments of position (mean, minimum and maximum values) and dispersion (standard deviation, coefficient of variation, skewness and kurtosis). The Spearman's correlation coefficients (rS) were estimated at $p \leq 0.01$, for the association between the biometric characteristics of the fruits.

The main effects of the OD process fractional design were estimated for screening variables. The experimental design was performed at a level of significance of 99% ($p \leq 0.01$), using pure error. Both biometric analysis and OD data were analyzed using the software Statistica (version 7.1, StatSoft Inc., USA).

3 RESULTS AND DISCUSSION

3.1 BIOMETRIC ANALYSIS

The descriptive analysis results for the physical characteristics of the jambolan fruits are presented in Table 2. The mean morphometric dimension values of the fruits were 26.7 mm for longitudinal diameter (LD) and 17.87 mm for equatorial diameter (ED). These values are higher than those reported by Albuquerque et al. (2019), who found 22.49 mm for LD and 16.8 mm for ED, for the same fruit.

Table 2. Physical characteristics of jambolan fruits.

Measurement	Result[a]	Minimum	Maximum	Skewness	Kurtosis	CV
LD (mm)	26.7 ± 2.67	19.4	34	-0.24	0.71	10.0
ED (mm)	17.87 ± 1.93	11.1	22.7	-0.48	0.74	10.8
WFW (g)	5.66 ± 1.67	2.1	9.43	0.06	-0.75	29.43
SFW (g)	1.59 ± 0.38	0.64	3.09	0.38	1.64	23.88
PFW (g)	4.07 ± 1.41	1.45	7.08	0.15	-0.81	34.66
Yield (%)	70.74 ± 6.09	53.94	85.51	-0.66	0.26	8.61

[a] Data refer to mean values (n = 100) ± standard deviation; LD = longitudinal diameter (mm); EQ = equatorial diameter (mm); WFW = whole fruit weight (g); SFW = seed fruit weight (g); PFW = pulp fruit weight (g); CV = coefficient of variation (%).

Steiner, Zuffo and Zoz (2017) observed values of 4.83 g, 1.24 g and 72.93% for whole fruit weight (WFW), seed fruit weight (SFW) and yield, respectively. These values are also slightly lower than those found in the present work, except for yield (Table 2). The pulp fruit weight (PFW) observed in the study presented a mean value of 4.07 g, which contributed to a yield higher than 70%. This yield value is much higher than the minimum value (40%) required for products elaboration, mainly for food processing industries (LIRA JÚNIOR et al., 2005).

The values of skewness closest to zero were 0.06, 0.15 and -0.24, for WFW, PFW and LD, respectively (Table 2). These findings indicate an approximately normal distribution for the characteristics evaluated, as shown by the frequency distribution (Figure 2 a, c and e). The negative kurtosis values found in the present study for WFW and PFW suggests a platykurtic distribution (IBE, 2014).

The values of the coefficients of variation (CV) for the physical characteristics of the jambolan ranged from 8.61% (Yield) to 34.66% (PFW). Lower CV values were reported by Steiner, Zuffo and Zoz (2017) for the biometric characteristics of jambolan fruits (*Syzygium cumini*) (5.17-25.94%). In general, the studied fruits presented greater morphometric characteristics when compared to the fruits found by other authors (FERRAZ et al., 2014; STEINER; ZUFFO; ZOZ, 2017; ALBUQUERQUE et al., 2019). These variations observed in the biometric characteristics of jambolan can be attributed to the phenotypic variation, which is influenced by uncontrolled environmental components, such as anthropization conditions, edaphic and climatic factors, plant age and genetic differences (SILVA; CHAVES; NAVES, 2001).

The fruits of jambolan presented themselves as oblong, with 19.4 to 34 mm of LD, 11.1 to 22.7 mm of ED, 2.1 to 9.43 g of WFW, 0.64 to 3.09 g of SFW, 1.45 to 7.08 g of PFW and yield varying from 53.94 to 85.51%. According to Silva et al. (2012), the non-domesticated plant species present great variability between same matrices on the physical characteristics, such as size, color, number of seeds, weight of seed and pulp, among others.

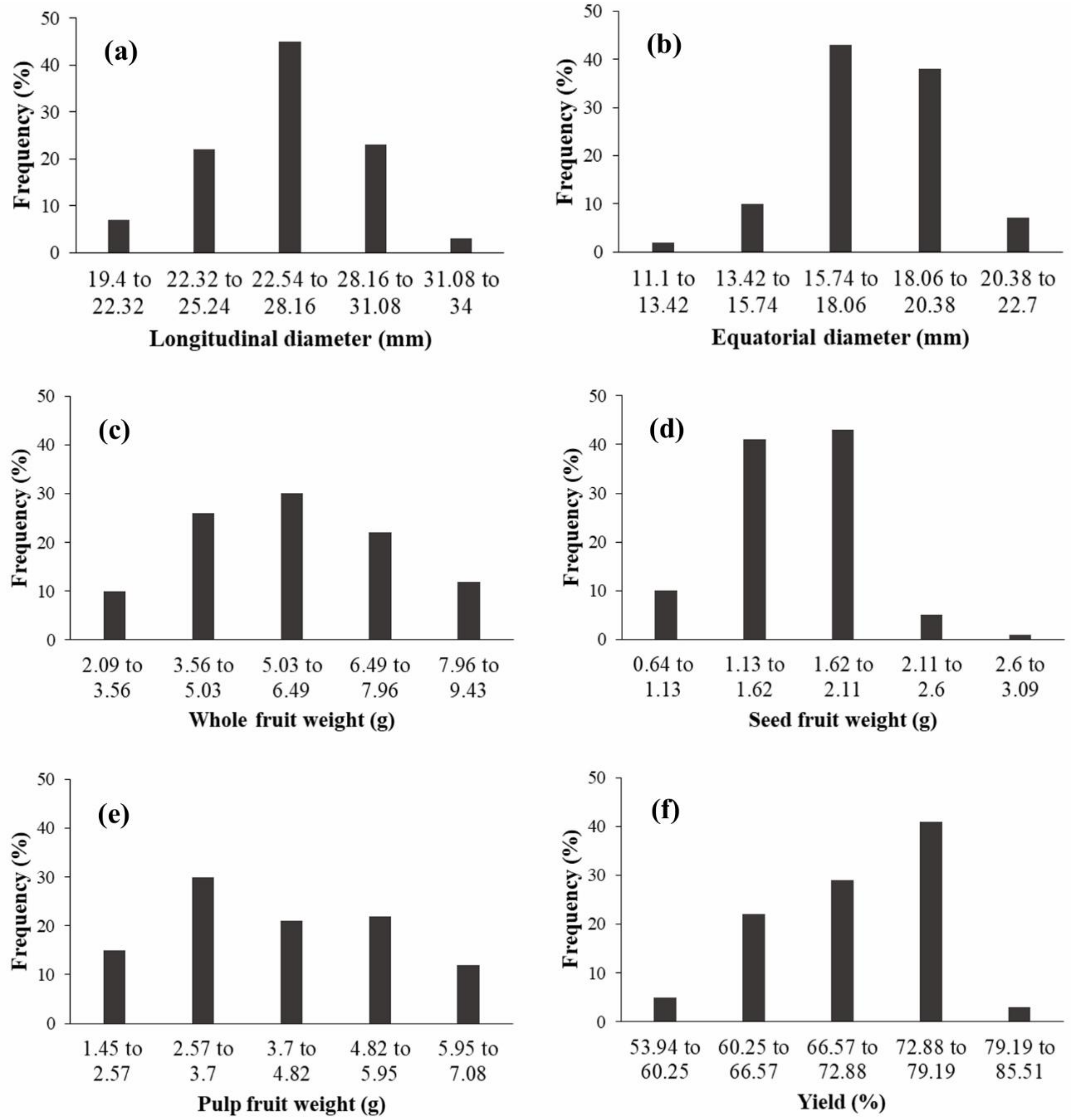

Figure 2. Distribution of frequencies for the different physical characteristics of the fruits measured.

Regarding the frequency distribution, about 45% of the fruits presented LD corresponding to 22.54 to 28.16 mm (Figure 2a) and, approximately, 43% presented ED in the range of 15.74 to 18.06 mm (Figure 2b). Close results were found for fruits of jambolan in the study of Ferraz et al. (2014), who demonstrated that approximately 44% of the LD showed values between 20.90 to 25.40 mm, and around 49% of the ED presented values varying from 14.90 to 16.40 mm.

For the subsequent biometric characteristics, the highest frequency values were in the range of 5.03 to 6.49 g (30%) for WFW (Figure 2c), 1.62 to 2.11 g (43%) for SFW (Figure 2d), 2.57 to 3.70 g (30%) for PFW (Figure 2e) and 72.88 to 79.19% (41%) for yield (Figure 2f). A considerable amount of jambolan fruits (≈ 95%) presented yield above 60% (Figure 2f), which is higher than the results observed by Albuquerque et al. (2019), who found a mean yield value of 57.22%, for the same fruit.

The association among the physical characteristics is also important because it allows verifying the interference between the factors (LIU, 2017). In this sense, the Spearman's rank correlation coefficient (rS) is used to express the degree of association between two numerical characteristics. A positive or negative value corresponds, respectively, to an increasing or decreasing monotonic trend between two variables (SHAW; JHONSON; PROSCHAN, 2017).

Table 3 shows a positive and significant correlation for all physical variables of the jambolan fruits, with the exception of the correlation between yield and SFW. The highest value of the Spearman's rank correlation coefficients (rS) was observed for PFW vs WFW (rS = 0.98; $p \leq 0.01$). In this sense, an increase in WFW is desirable for the economic exploitation of jambolan, since it tends to increase the PFW and, consequently, increases the edible part of the fruit. Thus, the results have shown that it is possible to identify and select jambolan fruits with higher pulp yield based on other biometric characteristics (WFW, LD and ED).

Table 3. Spearman's rank correlation coefficient (rS) between the different physical characteristics of jambolan fruits.

Characteristic	LD	ED	WFW	SFW	PFW
ED	**0.72**				
WFW	**0.82**	**0.85**			
SFW	**0.79**	**0.68**	**0.74**		
PFW	**0.76**	**0.83**	**0.98**	**0.60**	
Yield	**0.31**	**0.48**	**0.62**	-0.03	**0.74**

Bold characters indicate that the corresponding parameter has a significant effect ($p \leq 0.01$). LD = longitudinal diameter (mm); ED = equatorial diameter (mm); WFW = whole fruit weight (g); SFW = seed fruit weight (g); PFW = pulp fruit weight (g).

In this context, the fruits' morphological characterization is fundamental to provide information for the handling, packaging, advanced stages of commercial and industrial exploitation, as well as assisting on the machinery and equipment design (STEINER; ZUFFO;

ZOZ, 2017). Besides, the knowledge of the biometric variation of fruits is important for the creation of germplasm banks and for the improvement of these characteristics, in the sense of fruit's increasing or uniforming. It can also be explored by breeding programs directed to the generation of cultivars that provide fruits with important features in order to improve the commercialization (GONÇALVES et al., 2013).

3.2 PHYSICAL-CHEMICAL, CHEMICAL AND COLOR EVALUATION OF JAMBOLAN

The fresh fruits of jambolan harvested from the Amazon region were characterized as acidic fruits (pH = 3.87 ± 0.04), presenting a total tritatable acidity value of 1.22 ± 0.02 (g citric acid.100^{-1} g). Moreover, the most abundant component of the fruit is moisture (84.62 ± 0.26 g.100^{-1} g), followed by carbohydrates (13.90 ± 0.03 g.100^{-1} g). Although high acidity and low pH (< 4.0) can limit microbial growth in the product (LARA, 2019), the high moisture and a_w (0.98 ± < 0.01) values, as well as the reducing sugars content (8.19 ± 0.17 g.100^{-1} g), favor the degradative processes in the fruits of jambolan. Other minority components in the fruit are ashes (0.43 ± 0.01 g.100^{-1} g), total proteins (0.48 ± 0.05 g.100^{-1} g) and total lipids (0.56 ± 0.03 g.100^{-1} g). These results are similar to those reported by other authors (BRITO et al., 2017; ALBUQUERQUE et al., 2019).

The studied jambolan fruits were shown to be a promising source of total phenolics (391 ± 18.89 mg GAE.100^{-1} g) with levels higher than those found in studies for the same fruit by Rufino et al. (2010) (185 mg GAE.100^{-1} g) and Albuquerque et al. (2019) (182.01 mg GAE.100^{-1} g). The total phenolics present in fruits are one of the main compounds responsible for their antioxidant activity. Thus, as food components, phenolic compounds play roles in imparting the color (especially anthocyanin, a class of flavonoids), having sensory features as well, and acting as food preservatives (SINGH et al., 2018).

Besides their food coloring roles, anthocyanins are also important antioxidant sources and take fundamental part on helping prevent many degenerative diseases (KHOO et al., 2017). Albuquerque et al. (2019), when studying anthocyanin content in fresh jambolan found values of 93.56 mg.100^{-1} g. Rufino et al. (2010) determined the anthocyanins content of several fruits from the *Myrtaceae* family, including camu-camu (42.2 mg.100^{-1} g), jaboticaba (58.1 mg.100^{-1} g), jambolan (93.3 mg.100^{-1} g) and murta (143 mg.100^{-1} g). These findings are slightly below from those observed in the present study (147 ± 1.85 mg.100^{-1} g). Such differences may be explained by several climatic and edaphic factors (BRITO et al., 2017).

The color parameters for the jambolan fruit denote low values of lightness (L^* = 32.34 ± 0.18) and chroma (C^* = 16.80 ± 2.93); the values found for a^* (16.80 ± 2.93, red color), b^*

(-0.29 ± 0.08, blue color) and *h*º (359.02 ± 0.03, pure red) located the fruit in the CIELAB space corresponding to the purple-red color region. These findings corroborate to those observed by Brito et al. (2017), for the same fruit.

3.3 OSMOTIC DEHYDRATION OF JAMBOLAN

The jambolan fruits selected for OD process were chosen based on the interval of the highest frequencies for LD (22.54-28.16 mm) (Figure 2a), ED (15.74-18.06 mm) (Figure 2b) and WFW (5.03-6.49 g) (Figure 2c). The experimental results of water loss (WL), solid gain (SG) and weight reduction (WR) for the OD of jambolan, from the fractional factorial design, are presented in the Table 1 (columns eight to ten). The highest WL values (> 20%) occurred at the maximum temperature (T) and calcium lactate (CC) or sucrose (SC) concentration levels (runs 2, 8, 12 and 16). In contrast, the lowest WL value (run 1) occurred at the minimum levels of all independent variables, in which an undesirable swelling effect (water gain) was observed.

In general, the WL (-1.06-21.35%) was much higher than the SG (-5.11-7.09%), and this phenomenon could be explained by the formation of a dense layer of solutes at the surface of the osmo-dehydrated jambolan, acting as a barrier against solutes penetration into the food, making solutes mass transfer more difficult, which probably has resulted in a lower solids uptake inside the fruit tissue (GIRALDO et al., 2003). According to Torreggiani (1993), in the OD process, the WL must be favored with a minimum SG. Nevertheless, SG should be enough for preservation but not so high to induce sensory and nutritional changes in the product (DELGADO et al., 2018).

In addition, high WR (2.62-18.69%) values were attributed to high WL values, in most of the assays, because there was a greater amount of water getting out from the fruit tissue (mass loss) than solute entrancing (mass gain). This behavior occurs in preserved tissue because the selective permeability of the cell membranes allows the transport of small molecules, such as water. However, it can restricts the transport of larger molecules, such as sucrose, and hence reduce the diffusion of sucrose through the cell tissue (SILVA; FERNANDES; MAURO, 2014).

A useful tool to identify the most important factors affecting an experiment is the Pareto chart. This graph shows the main effects estimate plotted against a horizontal axis. The main effects are ranked according to their significance order and a vertical line is used to indicate the threshold p-value for statistical significance (LOUKAS, 2001). In this sense, the fractional factorial design was employed to analyze the intensity of the effects and its significance instead of model validation. Thus, the Figure 3 shows the Pareto chart of each effect estimate for the dependent variables (responses).

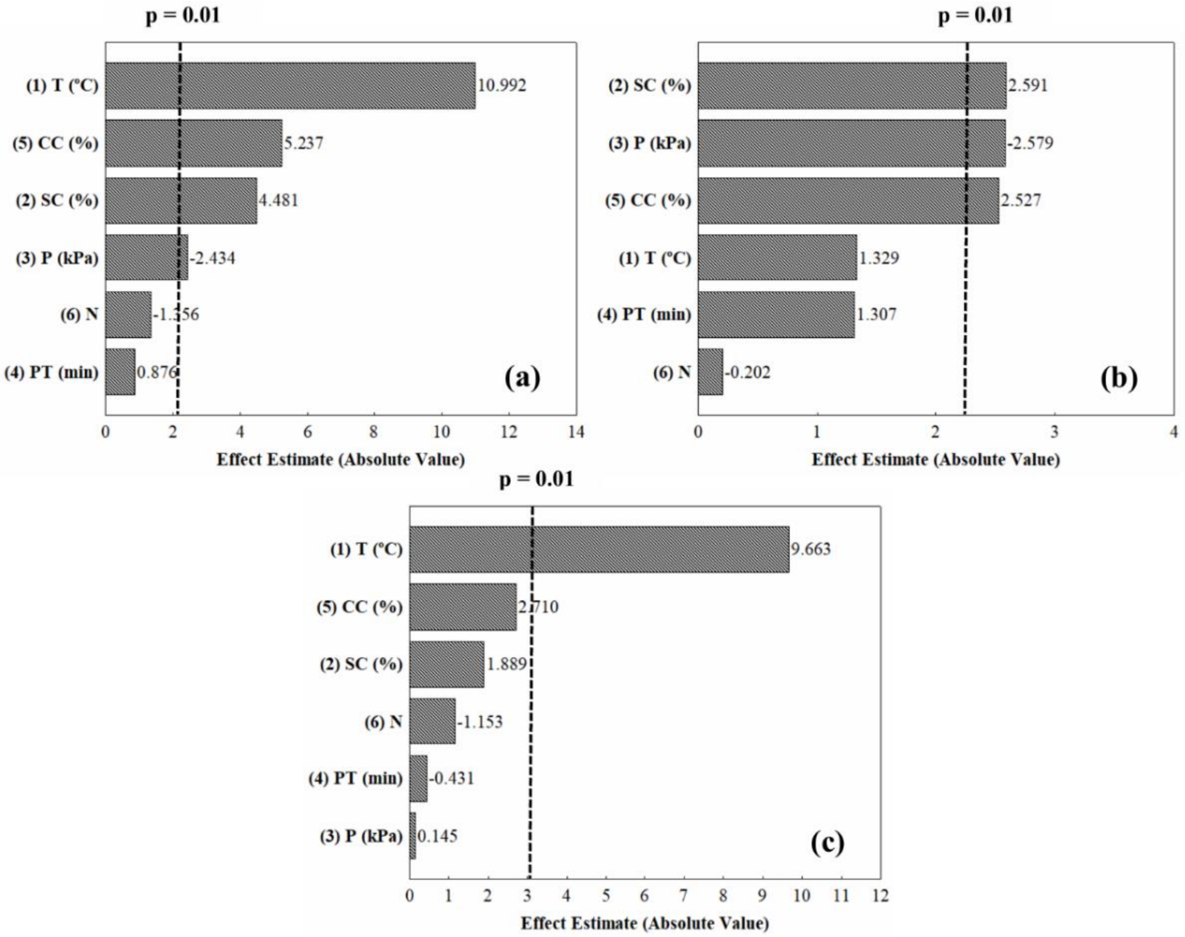

Figure 3. Effect estimate (absolute values) for water loss (a), solid gain (b) and weight reduction (c). T = temperature (ºC); SC = sucrose concentration (%); P = pressure (kPa); PT = vacuum pulse time (min); CC = calcium lactate concentration (%); N = number of vacuum pulse.

3.4 EFFECT ON THE PERCENTAGE OF WATER LOSS

From the Figure 3a, the significant effects that contributed positively with WL were T, CC and SC ($p \leq 0.01$). The T was the main variable affecting WL. These results are in agreement with Alam, Amarjit and Sawhney (2010), who studied the OD of Indian gooseberry and verified that T influenced significantly the WL. Mercali et al. (2010) reported that high temperatures could promote a fast WL due to the plasticizing and swelling effects that occur in cell membranes, as well as the faster water diffusion in the product due to the lower viscosity of the osmotic medium.

The second most important factor in WL of jambolan was CC. OD added calcium has been used in an attempt to increase firmness and enhance the selective effect of sucrose transfer, restricting the SG and increasing WL (MAVROUDIS; GIDLEY; SJÖHOLM, 2012). Close results were found by Silva, Fernandes and Mauro (2014), who observed that the addition of 4% calcium lactate significantly increased the WL of pineapple at all processing times.

High SC also favored the WL, and this fact seems to be based on the increase in osmotic gradient between the food material and the osmotic solution. The existence of a large amount of solute causes a higher osmotic pressure that makes the WL easier (CORRÊA et al., 2010). These results corroborate to those obtained by Porto et al. (2014), who studied the OD of Crimson seedless grape and observed that the SC had a positive influence on the WL content.

The WL increase in jambolan is also attributed to the application of low pressures (P), as explained by the hydrodynamic mechanism (HDM), in which outflow induced in the fruit tissue is the result of the compression of sample volume provoked by the pressure change, implying on the liquid exit from the fruit with subsequent acceleration of WL (CHIRALT; FITO, 2003). Similar behavior was observed Corrêa et al. (2015), who demonstrated that P was the variable with the smallest influence on WL in OD of sliced tomato.

3.5 EFFECT ON THE PERCENTAGE OF SOLID GAIN

The variables SC, P, and CC presented similar influence on SG (Figure 3b), at 99% level of significance. SG was affected positively by SC ($p \leq 0.01$) and, according to Phisut (2012), an increase in SG with SC is due to the high concentration gradient between the sample and the osmotic solution, which increases the diffusion rate of solute. Furthermore, the increased mass transfer of sugar molecules with the increase in concentration is possible due to the membrane swelling effect that increases the permeability of the membrane. Thus, by choosing a higher concentrated medium a much greater SG is observed. These findings are in accordance with those found by Siqueira et al. (2019), for OD of ginger.

The negative effect of P on SG ($p \leq 0.01$) suggests that the use of high pressures decreases the response. Thus, the lower the pressure (high vacuum) the higher the SG. The gain of solid attributed to vacuum pulse pressure can also be explained by the HDM and, therefore, when vacuum conditions are applied, the gas occluded inside the intercellular spaces of vegetable tissues is removed. Once the atmospheric pressure condition is restored, the pores of the food material are filled by osmotic solution, increasing the available mass transfer surface area (CORRÊA et al., 2010).

Shi et al. (1995) reported that osmotic treatments performed with the application of vacuum are effective on WL rates, but its influence on SG is only evidenced in highly porous fruits, once SG is intrinsically related to the micro structural characteristics of the cellular tissue, such as porosity. Corrêa et al. (2010) noticed that the use of 15 min of vacuum pulse (10 kPa) at the beginning of the OD process caused an increase in the SG of guavas.

However, regarding the positive influence of CC on SG observed in the study, the overall SG among the assays (Table 1) was not expressive ($SG < 7.09$). Other studies, as pointed by Silva, Fernandes and Mauro (2014), found that the OD of pineapple in sucrose solutions added calcium lactate significantly reduced the incorporation of sugar in the fruit and denoted that the presence of this salt tends to restrict the gain in sucrose. Similar behavior was noted by Mavroudis, Gidley and Sjöholm (2012) who observed that the SG in apples decreased with the addition of 0.6% calcium lactate to the solution, and attributed the result to a reduction in cell wall porosity.

3.6 EFFECT ON THE PERCENTAGE OF WEIGHT REDUCTION

Figure 3c demonstrates that T was the only significant variable affecting WR ($p \leq 0.01$), which had a positive effect. The WR exhibits a mass relationship among the whole flows involved in the osmotic process, mainly between WL and SG (EL-AOUAR et al., 2006), but in this study WR was more attributed to WL instead of SG (Table 1). The effect of T on WR is because its increments favor the exit of water from the fruit (BEKELE; RAMASWAMY, 2010). Some researchers have observed the same influence of T over WR, involving OD studies in pineapple (SRIDEVI; GENITHA, 2012) and chestnut slices (DELGADO et al., 2018). The WR, which indirectly measures the water reduction in the osmotic dehydrated product, is an important variable in the transportation and storage of great product volumes (ZAPATA; CIRO; MARULANDA, 2016).

Regarding to the variables PT and N, no significant effect was found ($p \leq 0.01$) among all the studied responses (WL, SG and WR). A research involving PT (5-15 min), at the beginning of the process, have pointed its positive effect on SG in 5 h of osmotic process, despite no influence of this variable being found below this period of time for any response, in the OD of mango (ITO et al., 2007). In other research, Zapata, Ciro and Marulanda (2016) studied the influence of N on the OD of Cape gooseberry and have demonstrated no effect of this variable over WL, SG and WR.

The results show that the OD is a suitable technological alternative to reduce post-harvest losses of jambolan fruits. In this sense, this fruit could be dehydrated to save part of the production that is not destined for immediate consumption or exportation, providing its shelf life extension. In addition, to improve the control of this unitary operation, the present work has contributed for the selection of variables that significantly influence the OD process of jambolan.

Thus, the fractional factorial design has collaborated on screening the experiments in which effects of all independent variables were examined simultaneously, identifying efficiently variables that are, in fact, important for the process. Moreover, the present study gives subsides for researches to rearrange the minimum and maximum levels of the variables in further OD experiments and optimization studies can be formulated from the information generated by this preliminary design.

4 CONCLUSION

Osmotic dehydration (OD) of jambolan was studied for the first time. The biometric analysis shows that the pulp yield of jambolan is directly proportional to the dimensions and weight, and inversely proportional to the seed weight of the fruits. The pulp and skin represent more than 70% of the whole fruit weight, also is a potential source of bioactive compounds, which is favorable for its economic exploitation, as in OD process. The screening design shows that temperature, pressure, calcium lactate concentration and sucrose concentration have influence over the osmotic process, considering water loss as the most important property in OD. On the other hand, vacuum pulse time and number of vacuum pulse showed no influence on the OD process; therefore, these variables can be fixed at the most economically viable level for any further trials. The presence of calcium lactate minimized the solute uptake in the cell tissue of the jambolan, controlling the solid gain in the product. Thus, future optimization studies can be formulated from the present fractional factorial design.

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Capítulo V

ARTIGO 3 – COMBINED OSMOTIC DEHYDRATION AND CONVECTIVE AIR-DRYING PROCESS OF JAMBOLAN FRUITS

ABSTRACT

Jambolan (*Syzygium cumini*) is a native fruit from Asia, which has adapted well to the tropical climate of the Amazonian region. This fruit is a promising source of polyphenols (especially anthocyanins) related to health-promoting benefits. Due to its little annual availability and high perishability, the jambolan fruit is still underexploited. Thus, the aim of this study was to preserve the jambolan fruit through a combined process of osmotic dehydration (OD) and convective air-drying, and to monitor the total phenolic contents (TPC) and monomeric anthocyanins (MA) during these processes. For this, jambolan fruits were osmotically pretreated in increasing OD times (1, 4, 8, 16, 24, 48 and 72 hours) and, after continuous monitoring of water loss (WL), solid gain (SG), weight reduction (WR), water activity (a_w), TPC and MA during OD kinetic, the pretreated (PT) and non-pretreated (NPT) fruits underwent convective air-drying at 50, 60 and 70ºC. The osmotic pretreatment reduced half of the water present in the fruit, with little solid impregnation. Parallel to that, OD decreased the TPC and MA through time, which could be induced by the leakage of these compounds into the osmotic solution, or even by chemical and biochemical reactions. The increase in air-drying temperature has shortened the drying time for both NPT and PT jambolan, and osmotic pretreatment has reduced even further the time needed to achieve the equilibrium condition, which emphasizes that PT presented a good capability of saving the energy for subsequent drying. Furthermore, the fruit pretreated and dried at 60ºC has shown promising results, in terms of storage stability, which could be a good alternative to extend the fruit's shelf life and making it available throughout the year. Besides, the TPC and MA values of PT (60ºC) treatment remained as nearly as the NPT (60ºC) one.

Keywords: osmosis; dehydration; preservation; bioactive compounds.

1 INTRODUCTION

Phenolic compounds are secondary metabolites with protective roles in higher plants. They are produced, mainly, using the shikimic acid pathway and can be divided into many subgroups, most likely phenolic acids, tannins and flavonoids (SINGH et al., 2016). Among flavonoids, anthocyanins represent one of the most abundant constituents, corresponding to the red, blue and purple color of fruits and vegetables. These pigments can degrade during thermal processing, which can influence on color quality and affect nutritional properties of the final products (PATRAS et al., 2010).

The ingestion of foods rich in anthocyanins have been associated with some health benefits, which can induce positive effects to the organism because of the bioactivity of these substances (LIMSITTHICHAIKOON et al., 2015). In this scenario, jambolan (*Syzygium cumini*) fruit appears as a potential source of phenolic compounds; being also rich in anthocyanin pigments (LESTARIO et al., 2017). Its potential therapeutic benefits can be explained by the antioxidant and antimicrobial properties (SINGH et al., 2016).

Moreover, jambolan is an ellipsoid berry (3-5 cm length) that presents a whitish/pinkish pulp, reaching full ripening when its peel manifests a purple to black color (SINGH et al., 2018). Native from India, it was first introduced in Brazil by Portuguese settlers and commonly spread in nature. The fruit does not have a high commercial value in Brazil and most of the production is lost (SILVA et al., 2018). Besides, jambolan is a seasonal fruit that is often available in the months of December to February, and its shelf life is no longer than 2 days at room temperature (SABINO; BRITO; SILVA JÚNIOR, 2018).

Throughout post-harvesting, fresh fruits become more susceptible to several changes, which lead them to senescence and deterioration by biochemical reactions and microbial growth. These two variables represent the most common limiting factors that influence the food’s shelf life. Thus, preservation techniques are needed to achieve stability and maintain the moisture content under levels required for fruit conservation (HSSAINI et al., 2022).

Dehydration is a common preservation technique for reducing post-harvest losses and this process can extend the shelf life and availability of fruits during all seasons (KARAM et al., 2016). Hence, to improve the quality of preserved food products, researchers have aimed for alternative paths to process foods, and one of these methods is osmotic dehydration (OD) (MAGUER; SHI; FERNANDEZ, 2003). OD is usually applied as a pretreatment prior to drying operations, since it involves the immersion of foods in hypertonic solutions for partial water removal, due to the pressure difference between the food and the hypertonic solution (RAOULT-WACK, 1994).

This preservation technique is simple and facilitates the processing of fruits, in regards to the retention of initial characteristics, such as color, aroma, texture and nutritional composition. However, OD will not give a safe moisture content to be considered as a shelf stable product, and therefore, the osmotically dehydrated product must be further dried through some complementary process (RAMYA; JAIN, 2016).

Mass transfer during OD depends on some factors, including product's geometry, temperature, agitation, type and concentration of the osmotic agent, among others. Recently, the use of calcium salts have been employed to reduce damages caused by the cell wall structure and improve the mass transfer of the osmo-dehydrated products (SILVA; FERNANDES; MAURO, 2017; TAPPI et al., 2017). In addition, the increment of pulsed vacuum osmotic dehydration (PVOD), at the beginning of the OD process, has been used as a resource to accelerate the mass transfer in plant tissues (SAHIN; ÖZTÜRK, 2016; JUNQUEIRA et al., 2018; MELLO JR et al., 2019).

OD kinetics of jambolan, using ultrasonic vacuum pretreatment, have already been studied (SHARMA; DASH, 2019), and a recent study has selected the variables with significant influence on jambolan's OD (ARAÚJO; PENA, 2022a). However, no studies have contemplated the use of OD pretreatment combined to air-drying conditions for this fruit. Thus, the main goal of this research was to preserve the jambolan fruit through dehydration (pulsed vacuum OD + convective drying) and, at the same time, to monitor the total phenolic contents (TPC) and monomeric anthocyanins (MA) during osmotic pretreatment and air-drying process.

2 MATERIAL AND METHODS

2.1 MATERIAL

Jambolan (*Syzygium cumini*) fruits were collected in November to December 2021, from a local tree at the Federal University of Pará (UFPA) (latitude 1º28' S, longitude 48º29' W), in the city of Belém (Brazil). The harvested fruits were transported in polystyrene isothermal boxes to the Laboratory of fruits, at the same university. After that, mature and integral fruits were chosen as a selection criteria for further analyzes. Then, these fruits were rinsed in running water, and sanitized by immersion in hypochlorite solution at 20 mg/L for 15 minutes, followed by a second rinsing. Furthermore, the sanitized fruits were packaged in low-density polyethylene packages and putted in polypropylene pots coated with aluminum foil. The fruits were stored at -18°C, until next procedures.

2.2 OSMOTIC PRETREATMENT

The following set was chosen for the osmotic pretreatment: 60% sucrose, 4% calcium lactate, and 10 kPa pressure (absolute). These conditions showed influence on jambolan's water loss (ARAÚJO; PENA, 2022a). Additionally, to avoid significant depletion of bioactive compounds during OD (at elevated temperatures) (KUCNER; KLEWICKI; SÓJKA, 2013), the set was fixed at 25ºC. The osmotic solutions were prepared by dissolving sucrose and calcium lactate to distilled water at ambient temperature.

Initially, jambolan fruits underwent defrost at room temperature, followed by weighing on analytical balance (M214AIH, BEL, Brazil). Furthermore, each fruit was putted inside a 100 mL beaker alongside the osmotic solution, using the fruit: solution ratio of 1:10 (m/v). After that, the beakers (quadruplicates) were positioned inside a glass desiccator, at room temperature (≈ 25ºC). Then, the system was sealed and submitted to the specific pressure (vacuum pulse), during five minutes. A vacuum pump (131, Prismatec, Brazil) was used to guarantee the pressure control. At the end of the pressure time, the system's vacuum was interrupted and maintained under atmospheric pressure until the total process time was finished. To keep constant temperature, the beakers (containing the fruits) were coated with aluminum foil, and placed in a BOD incubator (Q315M16, Quimis, Brazil), at 25ºC (±1ºC).

Osmo-dehydrated samples were taken from the osmotic solution in increasing times: 1, 4, 8, 16, 24, 48 and 72 hours (h); rinsed with 20 mL of distilled water and dried for 10 seconds in absorbent paper in order to remove water and sucrose/calcium lactate in excess from the fruit surface. Then, the osmotically dehydrated samples were weighed again in the analytical balance.

2.3 CONVECTIVE AIR-DRYING KINETICS

Non-pretreated and osmotically pretreated (OD = 72 h) samples were submitted to air-drying in a convective tray dryer (Q-316M5, Quimis, Brazil), at 50, 60 and 70ºC. For monitoring purposes, samples were weighed in a semi-analytical balance (S203H, BEL, Italy) (±0.001 g). During the first 60 minutes of drying, samples were weighed every 20 minutes, and after 60 minutes of drying, samples were weighed every 60 minutes until the sample's mass variation was below 0.1% (equilibrium condition). This procedure was performed in three replicates. The drying curves were made based on the correlation between moisture ratio (MR) (Equation 1) and the drying time. The moisture content was assessed before and after drying.

$$MR = \frac{m - m_e}{m_i - m_e} \quad (1)$$

where, MR = moisture ratio (dimensionless); m, m_i and m_e are the moisture contents at a given t time, initial and at equilibrium (g/g db), respectively.

2.4 MONITORING ANALYSES

Monitoring analyses (moisture content, water activity, monomeric anthocyanins and total phenolic contents) were assessed in the non-pretreated sample (control), and in the dried samples with and without osmotic pretreatment. Otherwise, during the osmotic dehydration kinetic, the mentioned analyses were performed plus water loss, solid gain and weight reduction.

2.4.1 Water loss, solid gain and weight reduction

Water loss (WL), solid gain (SG) and weight reduction (WR) were calculated according to Sridevi and Genitha (2012), by the Equations 2-4:

$$WL(\%) = \frac{(W_0 - W_t)}{M_0} \times 100 \quad (2)$$

$$SG(\%) = \frac{(S_t - S_0)}{M_0} \times 100 \quad (3)$$

$$WR(\%) = \frac{(M_0 - M_t)}{M_0} \times 100 \quad (4)$$

where, W_0 = initial water mass of the sample (g); W_t = water mass of the sample (g) after dehydration; S_0 = initial dry mass of the sample (g) and S_t = dry mass of the sample (g) after dehydration; M_0 = initial mass of the sample (g); M_t = mass of the sample (g) after dehydration.

2.4.2 Water activity and moisture content

The water activity (a_w) was determined by direct reading in the water activity meter (Aqua LAB 4TEV, Decagon Devices, USA), at 25ºC. Moisture content (MC) was calculated according to the AOAC (2002) methodology.

2.4.3 Monomeric anthocyanins and total phenolic contents

An extract was prepared for both essays of monomeric anthocyanins (MA) and total phenolic contents (TPC). The extraction followed the procedure from Brito et al. (2017), with modifications. The sample (≈0.3 g) was exhaustively macerated with acidified ethanolic solvent ($EtOH_{95\%}$:$HCl_{1,5N}$, 85:15, v:v), using a mortar and a pestle. Then, the macerated sample was putted inside a conical centrifuge tube Falcon (15 mL), and centrifuged at 1980 *x g*, for five minutes, in a centrifuge (K14-0815C, Kasvi, Brazil). After that, the supernatant was removed from the tube with a graduated pipette; transferred to a volumetric flask (10 mL) and completed with the extraction solvent. The extract concentration was equivalent to 0.03 g/mL, which corresponds to 30 g/L.

TPC was determined through the method described by Singleton and Rossi (1965) based on the reaction with the Folin-Ciocalteau reagent. Quantification was made by reading in UV-Vis spectrophotometer (Evo 60, Thermo-Fischer Scientific, EUA), at 760 nm. Concentrations between 5 to 100 mg Gallic acid/L were used for the construction of the analytical curve. TPC was expressed as mg GAE/100 g (mg Gallic acid equivalent/100 g), in dry basis (db).

MA was determined according to Giusti and Wrolstad (2001), which is based on the quantification of monomeric anthocyanins by the differential pH method. Briefly, aliquots of the extract were added to each buffer: 0.025 M potassium chloride (pH 1.0) and 0.4 M sodium acetate (pH 4.5). The absorbance of the mixture was measured at 514 nm and 700 nm, using the UV-Vis spectrophotometer. Absorbance (A) was calculated as $A = [(A_{514nm} - A_{700nm})$ at pH 1.0] – $[(A_{514nm} - A_{700nm})$ at pH 4.5]. The MA was calculated as delphinidin-3-glucoside equivalents (Equation 5):

$$\mathrm{MA(mg/L)} = \frac{\mathrm{A} \times \mathrm{MW} \times \mathrm{DF} \times 10^3}{\varepsilon \times \lambda} \tag{5}$$

where: MW = molecular weight (465 g/mol of delphinidin-3-glucoside); DF = dilution factor; $\mathcal{E}$ = molar attenuation coefficient (29000 L/mol.cm); λ = length of the cuvette (1 cm); 10^3 = conversion factor from g to mg. At first, the results were converted to mg delphinidin-3-glucoside/g, by dividing the MA value (mg/L) to the extract concentration (30 g/L). The MA results were expressed as mg delphinidin-3-glucoside/100 g, multiplying the value obtained in mg/g by 100. Further, the final MA results were expressed in dry basis (db).

2.5 OSMOTIC DEHYDRATION MODELING

A two-parameter model was used to predict the OD kinetic of jambolan. The values of WL and SG, at equilibrium, and the *k* parameter were calculated by linear regression from the relationship t/GX versus t, according to the linearized form of the model proposed by Azuara et al. (1992) (Equation 6).

$$\frac{t}{GX} = \frac{1}{k \times GX^{\infty}} + \frac{t}{GX^{\infty}} \tag{6}$$

where: GX = water loss (WL) or solid gain (SG) (%), in a time t; GX^{∞} = water loss (WL^{∞}) or solid gain (SG^{∞}) (%), at equilibrium; k = constant (h^{-1}); t = time of the process (h).

The model’s fit to the experimental data was confirmed by the coefficient of determination (R^2) and by the root mean square error (RMSE) (Equation 7).

$$RMSE = \left[\frac{1}{n} \sum_{i-1}^{n} \left(m_{exp,i} - m_{pre,i} \right)^2 \right]^{1/2} \tag{7}$$

where: $m_{exp,i}$ = experimental WL or SG value; $m_{pre,i}$ = predicted WL or SG value; n = number of observations.

2.6 CALCULATION OF EFFECTIVE MOISTURE DIFFUSIVITY

The effective moisture diffusivities (D_{eff}) from the air-drying process for non-pretreated and osmotically pretreated jambolan were estimated according to the analytical solution of Fick's second Law in the unsteady state, for nearly spherical materials (Equation 8) (CRANK, 1975). The calculation is conditioned by assuming that the moisture transfer is predominantly one-directional; the initial moisture content is equally diffused in the product; shrinkage is negligible, and the diffusion coefficient is assumed constant and homogeneous during drying.

$$MR = \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp\left(- n^2 \pi^2 \frac{D_{eff} t}{r^2} \right) \tag{8}$$

where, D_{eff} = effective moisture diffusivity (m^2/s); r = radius of a sphere (m); n = positive integer; t = time (s).

Simplifying Equation 8 and taking only the first term of the series into consideration, it could be rewritten in a logarithm form as Equation 9, which is an acceptable approximation in longer times (setting n =1).

$$\ln MR = \ln\left(\frac{6}{\pi^2} \right) - \left(\frac{\pi^2 D_{eff}}{r^2} \right) t \tag{9}$$

The plot of experimental drying data of ln (MR) *versus* time (t) gave a straight line with a slope (S), and the D_{eff} was calculated in Equation 10 by linear regression (SAHIN; ÖZTÜRK, 2016).

$$D_{eff} = S \left(\frac{d_e}{2\pi} \right)^2 \tag{10}$$

where, S = slope of ln (MR) *versus* drying time (t) and d_e = geometric mean diameter (m) of the fruit.

The shapes of non-pretreated jambolan and osmotically pretreated jambolan were assumed as ellipsoidal, and the mean geometric diameters (d_e) were obtained from the three measured diameters, as presented in Equation (11) (XANTHOPOULOS; YANNIOTIS; LAMBRINOS, 2009).

$$d_e = \sqrt[3]{d_x d_y d_z} \tag{11}$$

The effect of air-drying temperature on the D_{eff} of non-pretreated and osmotically pretreated jambolan were estimated by an Arrhenius-like equation (Equation 12) (SACILIK; ELICIN; UNAL, 2006).

$$D_{eff} = D_0 \exp\left(-\frac{E_a}{RT}\right) \tag{12}$$

where, E_a = activation energy (kJ/mol), D_0 = pre-exponential factor of the Arrhenius equation (m^2/s); R = universal gas constant (8.3143 kJ/mol), and T = absolute air temperature (K).

2.7 DRYING KINETIC MODELING

The following drying classic models (AKPINAR; BICER; YILDIZ, 2003): Newton (Equation 13), Page (Equation 14), modified Page (Equation 15), Henderson and Pabis (Equation 16), logarithm (Equation 17), Midilli (Equation 18), two-term (Equation 19), diffusion approximation (Equation 20), and two-term exponential (Equation 21) were used to predict the air-drying kinetics for non-pretreated and osmotically pretreated jambolan.

$$MR = \exp(-kt) \tag{13}$$

$$MR = \exp(-kt^n) \tag{14}$$

$$MR = \exp[-(kt)^n] \tag{15}$$

$$MR = a\exp(-kt) \tag{16}$$

$$MR = a\exp(-kt) + c \tag{17}$$

$$MR = a\exp(-kt^n) + bt \tag{18}$$

$$MR = a\exp(-k_0 t) + b\exp(-k_1 t) \tag{19}$$

$$MR = a\exp(-kt) + (1-a)\exp(-kbt) \tag{20}$$

$$MR = a\exp(-kt) + (1-a)\exp(-kat) \tag{21}$$

where, MR = moisture ratio (dimensionless); t = drying time (min); k, k_0 and k_1 are drying coefficients; a, b and n = equation fit parameters.

Mathematical modeling of the drying curves were fitted by non-linear regression, using the Statistica software. The Levenberg-Maquardt algorithm was employed with a convergence criterion of 10^{-6}. The values of R^2, RMSE (Equation 7), reduced Chi square (χ^2) (Equation 22), and residues dispersion (RD) were used to select the best drying fits.

$$\chi^2 = \frac{\sum_{i=1}^{N}(MR_{exp,i} - MR_{pre,i})^2}{N - n} \tag{22}$$

where, $MR_{exp,i}$ and $MR_{pre,i}$ = moisture ratio determined from the experimental data and moisture ratio predicted by the fitted models, respectively; N = number of experimental measurements; and n = number of model parameters.

3 RESULTS AND DISCUSSION

3.1 OSMOTIC DEHYDRATION KINETIC

3.1.1 Changes in water loss, solid gain, weight reduction and water activity

The progressing of water loss (WL), solids gain (SG), weight reduction (WR) and water activity (a_w), during the osmotic dehydration (OD) kinetic of jambolan, can be visualized in Figure 1. As can be seen, WL and WR presented a sharp increase in the first 16 hours of process, which represented 64.5% and 60% of the total WL and WR accounted for jambolan, respectively, during its OD kinetic. After that period, WL and WR evolution became less intense, until no big changes were observed. The osmotic pressure is the driving force responsible for water diffusion from the food to the solution, which is higher in the beginning of the process and promotes higher WL rates, due to the difference between concentrations of the dilute solute (from the fruit) and its surrounding hypertonic solution. In contrast, SG rates were not prominent at the initial stages of osmotic treatment (first 8 hours). However, SG slightly increases into the material after 8 hours. Thus, the pressure and concentration gradients gradually decrease as time progresses, until the equilibrium condition is achieved (RASTOGI; RAGHAVARAO, 1997; RASTOGI et al., 2002; SHARMA; DAS, 2019).

WL features a key role in OD processes, because it generates osmotic shock in microbial cells, which might affect their osmoregulation. Microorganisms keep the pressure turgor by retaining slightly lower moisture content (MC) inside their cells, in comparison to the outside environment. When OD is applied by either removing water or adding solutes or hydrophilic colloids, the environment's free water is reduced, which causes the free water of microorganism cells (hypotonic) to flow from the inside to the exterior medium (hypertonic environment), and may result in plasmolysis of microorganisms cells (ERKMEN; BOZOGLU, 2016).

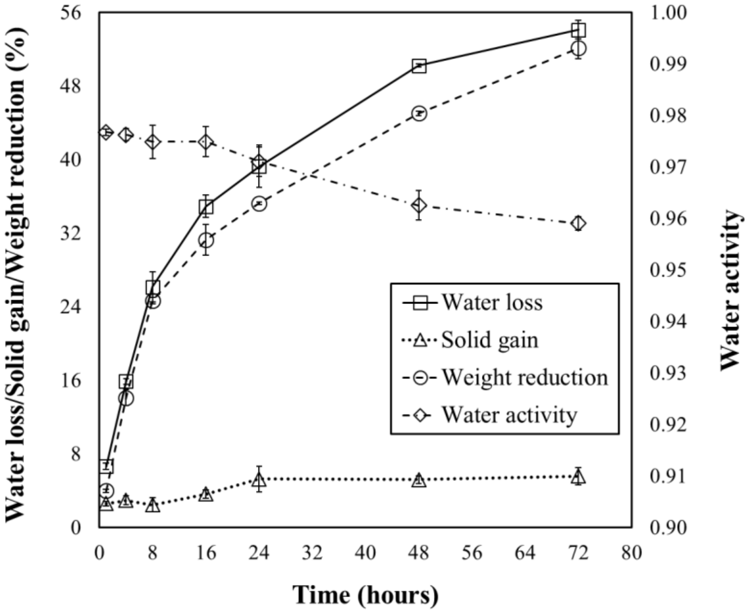

Figure 1. Progressing of water loss, solid gain, weight reduction and water activity during the osmotic dehydration kinetic of jambolan fruits.

As expected in OD processes, WL was much higher than SG. After 72 hours of process at 25 °C, WL and SG were around 54.10 ± 1.02% and 5.56 ± 0.96%, respectively. Sharma and Das (2019) have reached WL values around 35.2%, 47.1% and 54.5%, under atmospheric pressure (AP), vacuum pretreatment (VP), and ultrasound vacuum pretreatment (USVP), respectively, during the OD of jambolan. For SG, the same authors have observed the following values: 4.6% (AP), 7.6% (VP), and 9.3% (USVP). These WL and SG findings were reached after 5 hours of process, at 30ºC. On the other hand, the WL and SG values found by Araújo and Pena (2022a) ranged from -1.06 to 21.35% and from -5.11 to 7.09%, respectively, in osmotically dehydrated jambolan fruits, after 1.5 hours of process, in treatments with different combinations of temperature (20-50ºC), sucrose concentration (30-60%), pressure (10-90 kPa), and calcium lactate concentration (0-4%). Araújo and Pena (2022a) verified that the highest WL values (> 20%) were noticed at the maximum levels of temperature and calcium lactate/sucrose concentration.

By increasing the OD time, a_w presented an important reduction (Figure 1). In the first 16 hours of OD, the a_w values almost remained steady and, after that period, the a_w continually dropped until it reached nearly an equilibrium condition (72 hours). Although the a_w reduction, from the numerical point of view, presented a slight decrease during the OD kinetic, a considerable change has occurred in the fruit. Since jambolan is very hygroscopic, small a_w changes provoke considerable differences in the MC values at elevated a_w ranges, as shown by Araújo and Pena (2022b), during the moisture desorption process of jambolan pulp, at 25ºC. These authors noticed that great amounts of water were demanded to reduce the fruit's initial a_w. Thus, to decrease the a_w of jambolan from 0.98 to 0.90, the fruit requires a MC reduction superior to 40%.

In addition, a reduction in a_w decreases the lag phase of microorganisms and, thus, decreases their growth rate. Even a slight 0.005 reduction in the product's a_w (e.g. from 0.955 to 0.950) can decrease their intracellular MC in 50% and cell volume in 45%, which evidences the sensitivity of microorganisms to minimal a_w changes. As a result, microbial cells will keep alive or parish in a product with a_w change. However, some microorganisms present effective mechanisms to breakthrough plasmolysis, regain turgor by activating osmoregulation capacities, and maintain homeostasis, which varies from bacteria to fungi (ERKMEN; BOZOGLU, 2016). Due to this fact, it is important to avoid the microbial growth by lowering even further the a_w with another complementary process (e.g. drying), in order to stabilize jambolan under the required limits for food safety. Still, in Figure 1, the a_w variations were proximate to those found in OD kinetics (30ºC) with sweet potato, using sucrose (51% concentration) as osmotic agent (JUNQUEIRA; CORRÉA; MENDONÇA, 2016).

The extensive OD times observed in our present experiment (Figure 1) may be due to the application of a static osmotic treatment, since agitation of the osmotic solution can increase the mass transfer rates and improve the OD demanding time. However, this experiment choice was conditioned by limitations in the agitation of our OD system. Additionally, the temperature used to dehydrate jambolan osmotically was set at 25 ºC (room temperature), which could delay the water and solids diffusion through the material, in comparison to higher temperatures. Once again, this last choice was based on the degradative rates of bioactive compounds when subjected to higher OD temperatures. Furthermore, the essays were performed in whole fruits, and it could be assumed that the jambolan skin acts as a barrier against mass exchange during OD, due to its low permeability (KUCNER; KLEWICKI; SÓJKA, 2013). This same evidence was demonstrated in sour cherries with seeds (NOWICKA et al., 2015), in which experiments showed that the highest WL and SG rates occurred when the seeds were removed before the OD tests; since pitting of sour cherries enhanced the process, causing damages to its natural barrier.

3.1.2 Monomeric anthocyanins and total phenolic contents

In Figure 2, the monomeric anthocyanins (MA) and total phenolic contents (TPC) modifications are evidenced during the OD kinetic of jambolan. During the first 4 hours of process, the MA and TPC suffered a significant decrease and, after this process time, the values remained almost stabilized until it reached the end of the operation (72 hours). At the final OD time, MA retained 53.43% and TPC retained 73.11%, concerning the initial contents.

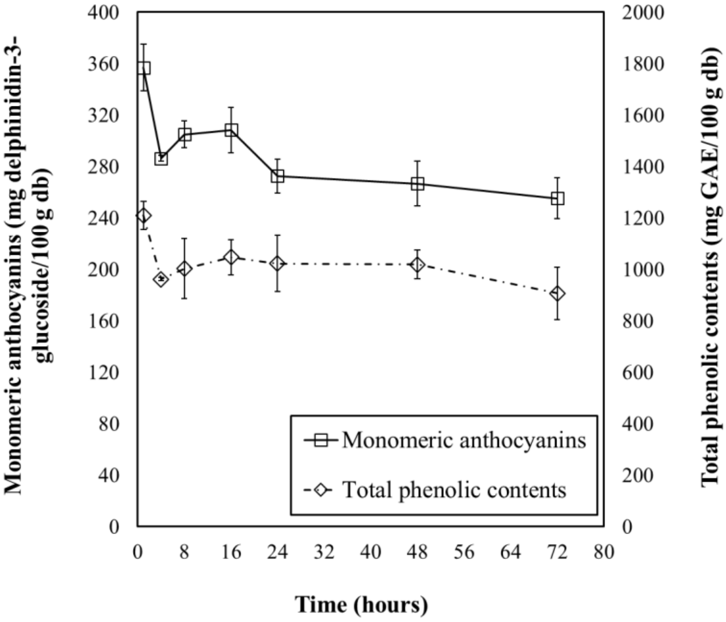

Figure 2. Changes of monomeric anthocyanins (MA) and total phenolic contents (TPC) during the osmotic dehydration kinetic of jambolan fruits.

Nowicka et al. (2015) noticed a similar behavior throughout the OD kinetic of sour cherries at 40ºC. During the first 60 minutes, significant losses of TPC occurred and the authors associated that to an intensive mass exchange during the first phase of OD, which was followed by a slight TPC increase in the second hour of OD, and further, it resulted in the TPC degradation over time, by the loss of cell membrane selective transport of substances. Araya-Farias, Macaigme and Ratti (2014) figured out that OD (40ºC/6 h) caused an 87.99% reduction in TPC of seabuckthorn fruit. Sójka, Karlinska and Klewicki (2017) evidenced substantial decreases in the MA contents of blackberries after 1 hour of OD (30ºC), in which retentions ranged from 60.8-68.6%. These researchers realized that further OD time had a less dramatic effect on MA, which also translated to the retention levels (50.5-55.9% and 46.8-53.6%, after 3 h and 5 h, respectively), at different osmotic solution concentrations (50, 57.5, and 65%).

During OD, the osmotic pressure difference results in three simultaneous flows: water flow from the food matrix to the solution, reverse flow of solutes from the concentrated solution to the product and removal of soluble solids from the product (e.g. vitamins, minerals and other water-soluble nutrients) from the food into the solution (RAOULT-WACK, 1994). Thus, chemical compounds present in fruits can be influenced by different variables of OD, and the content these compounds might change through biochemical or chemical transformations (e.g. enzymatic, hydrolytic, polymerization, biosynthesis, etc.) or even by their leaching into the osmotic solution. Therefore, a further analysis of individual compounds can shed light on this question. Anyway, the WL phenomenon promotes the concentration of chemical compounds available in the raw material. On the contrary, SG increases the sample's weight with an apparent decrease in chemical compounds (BLANDA et al., 2009). Thus, these different pathways can affect the product's final constituents. During OD (40ºC) of blueberries, Yu et al. (2018) observed both physical migration of bioactive compounds to the syrup medium as well as degradation of these nutrients by biochemical reactions.

3.1.3 Overall appearance of jambolan during osmotic dehydration

Figure 3 shows the aspects of jambolan fruits in different osmotic dehydration times. During osmotic pretreatment, the shrinkage effect becomes more evident as time progresses. These modifications were observed in osmo-dehydrated cranberries as well, and became more intense as the power of the microwave vacuum pretreatment increased from 100 to 800 W (ZIELINSKA, ZIELINSKA; MARKOWSKI, 2018). Yu et al. (2018) have noticed similar behavior after submitting blueberries to osmotic dehydration (40ºC), but the highest shrinkage effect was evidenced in samples pretreated with pulsed electric fields, in regards to those thermally pretreated. The shrinkage occurs when a plant cell is placed in an environment with low water potential (e.g. sucrose solution). As a result, the vacuole and the rest of the protoplast retract and cause the detachment between the plasma membrane and the cell wall, which is known as plasmolysis. The mass transfer and shrinkage phenomena are diffused from the surface of the material to its center, in function of the operation time (MAGUER; SHI; FERNANDEZ, 2003; EVERT; EICHHORN, 2014).

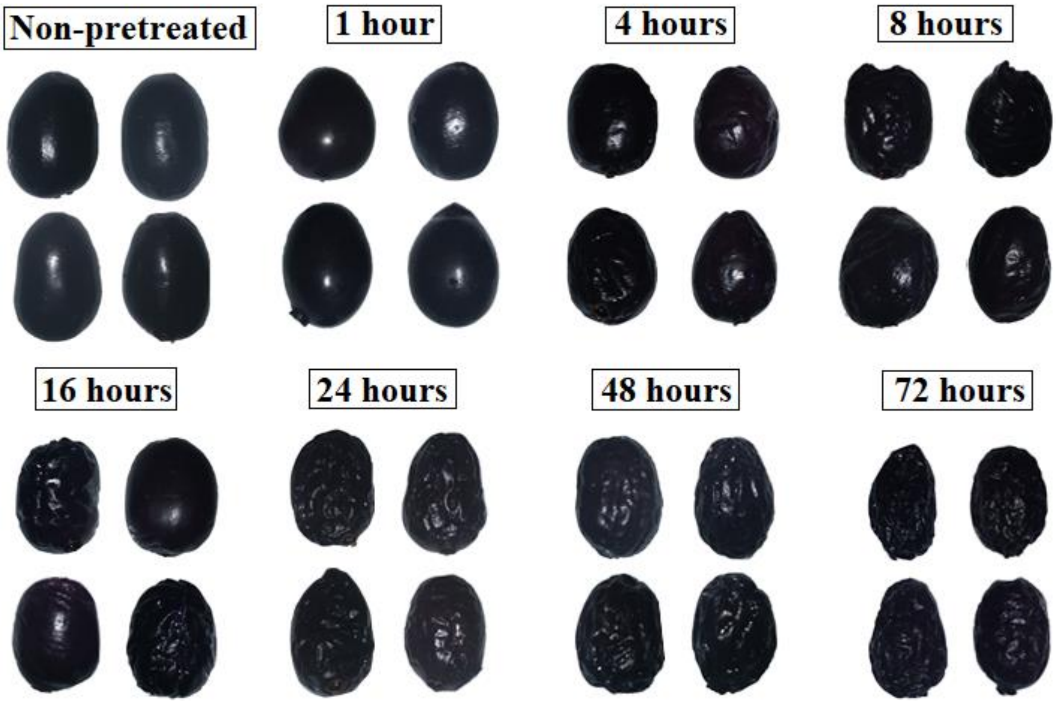

Figure 3. Overall appearance of jambolan fruits in different osmotic dehydration times.

3.1.4 Osmotic dehydration modeling

In the present study, the Azuara model (Equation 6), mathematically described the changes in WL and SG, during OD of jambolan. The values of R^2 (0.99 for WL and 0.98 for SG) and RMSE (0.66 for WL and 0.23 for SG) indicated that the Azuara model, fitted to the experimental data, can be used with good accuracy to predict the WL and SG kinetic of jambolan fruits, at the experimental domain. This model also displayed satisfactory fits ($R^2 > 0.96$ and $RMSE < 0.08$) for WL and SG in osmotically dehydrated cherry tomatoes, at different salt concentrations (10-25%) and temperatures (30-40ºC) (SOURAKI; GHAVAMI; TONDRO, 2014). The *k* value represents the process velocity when it reaches the equilibrium state, therefore, the high the *k* value the high is the water or solid diffusion by time unity. Thus, the *k* values showed that SG ($k_2 = 0.17$ h^{-1}) reached the equilibrium faster than the WL ($k_1 = 0.09$ h^{-1}). In addition, WL^{∞} and SG^{∞} parameters manifest WL and SG, at equilibrium state, respectively. The values of these parameters showed that times beyond 72 hours can contribute to higher water removal ($WL^{\infty} = 61.7\%$) and little solids impregnation rates ($SG^{\infty} = 5.95\%$) during OD of jambolan fruits.

3.2 DRYING KINETICS

3.2.1 Drying kinetics of osmotically dehydrated jambolan

For the next discussions, osmotically pretreated sample was defined based on the highest WL value and the lowest a_w level, which matches with 72 hours of OD (Figure 1). Curves of moisture ratio (MR) versus drying time for non-pretreated and pretreated dried jambolan are shown in Figure 4, along with the temperature effect. The drying curves show that the MR decreased with the drying time, for all treatments. At the early stages of drying, the MR decreased relatively fast and, after reaching a certain time, it decreased slowly until it reached an equilibrium condition. For non-pretreated samples, the time needed to reach the equilibrium moisture content was equivalent to 36 h at 50°C, 21 h at 60°C and 16 h at 70°C. In turn, for the osmotically dehydrated jambolan fruits, the equilibrium times were equal to 27 h at 50°C, 15 h at 60°C and 11 h at 70°C.

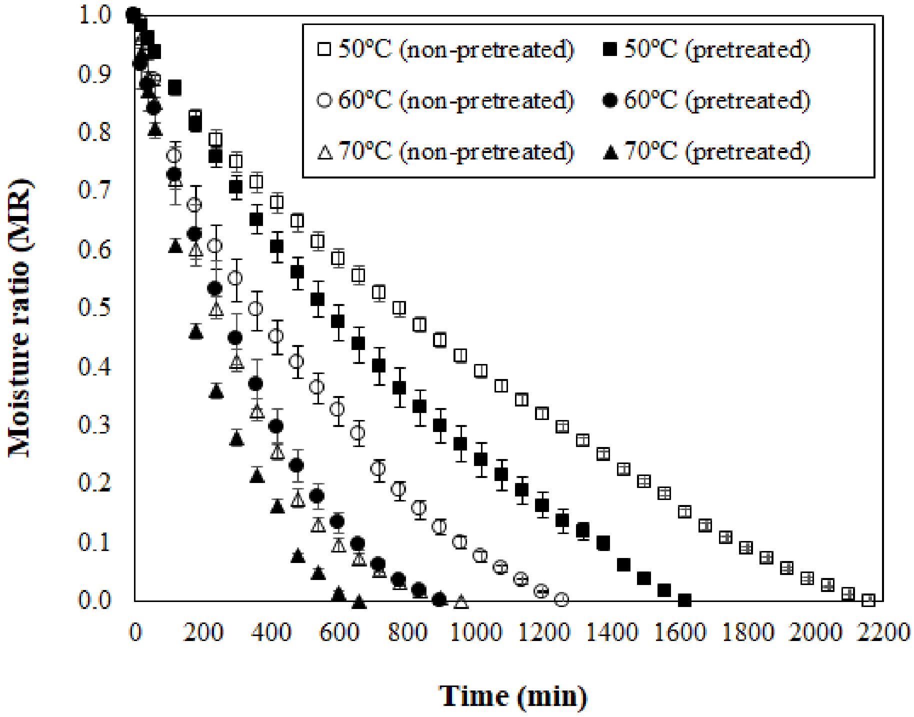

Figure 4. Moisture ratio (MR) versus drying time for non-pretreated and pretreated jambolan fruits submitted to drying at 50, 60 and 70°C.

The long drying periods noticed in the present research are awaited, since fruits are regularly difficult to dehydrate during hot air, due to their high water content, which results in extensive drying times and subsequently leads to structural and color changes (KROKIDA; MAROULIS, 2001). It is worth mentioning that the present study was set in whole jambolan fruits, and it is more challenging to conduct drying experiments in these conditions, considering that the fruit still contains its pericarp, whose natural feature is to protect the fruit from exterior influences. Therefore, it could difficult the water exit from the material, in comparison to sliced or cut fruits.

Furthermore, an increase in temperature has shortened the drying time for both non-pretreated and pretreated jambolan. This increase is expected, since a higher air-drying temperature promotes higher moisture evaporation rates at the interface between the food and the surrounding air. The greater moisture evaporation leads the moisture to diffuse at elevated rates, from the internal regions of the material to the surface (GARBA et al., 2015). Potosí-Calvache, Vanegas-Mahecha and Martínez-Correa (2017) have inferred that dehydration at higher temperatures was more advantageous, in regards to energy savings for squash.

Great amounts of WL during OD (Figure 1) led to significant decreases in the drying time of pretreated samples, when compared to the non-pretreated ones (Figure 4) and it took about 25%, 29% and 31% less time to dry jambolan, at 50°C, 60°C and 70ºC, respectively. Thus, much less moisture content is demanded for its drying, which results in less required drying time to remove water from the material, when compared to non-pretreated jambolan. The effect of osmotic pretreatment on the shortening of the drying time was already evidenced in the works of Sahin and Öztürk (2016), and An et al. (2013), during the drying of figs and cherry tomatoes, respectively. Fito et al. (2001) stated that vacuum impregnation during OD could cause modifications in certain food’s structure and composition, which might increase the heat and mass transfer rates during air-drying operations.

3.2.2 Effective moisture diffusivities and activation energy for drying process

The effective moisture diffusivities (D_{eff}) for the air-drying process of non-pretreated and osmotically pretreated jambolan fruits are presented in Table 1. These values lay within the range of food materials, which is from 10^{-11} to 10^{-9} m^2/s (MADAMBA; GRISCOLL; BUCKLE, 1996). Comparable results have been reported in the drying kinetics of fruits with spherical dimensions, as Turkish berries (3.73 to 7.56 x 10^{-10} m^2/s) (BARATHIRAJA et al., 2021), blueberries (2.58 to 4.58 x 10^{-11} m^2/s) (MARTÍN-GÓMEZ et al., 2020), and cranberries (0.92 to 2.37 x 10^{-9} m^2/s) (ZIELINSKA; ZIELINSKA; MARKOWSKI, 2018).

From data, it is possible to infer that an increase in drying temperature had increased the D_{eff} in non-pretreated and osmotically pretreated samples. According to Falade and Oyedele (2010), this behavior is due to a higher driving force, which accelerates the transfer of water vapor from the inside to the surface of the product, promoting higher D_{eff} values. Hence, it indicates that higher temperatures can promote faster drying speed, which could be attributed to an increase in the heating energy. Thus, increasing the activity of water molecules, and resulting in a higher water diffusion rate (XIAO et al., 2010). In addition, the osmotic pretreatment resulted in higher D_{eff} values when compared to each respective temperature of the non-pretreated samples. This same behavior was already evidenced in figs (SAHIN; ÖZTÜRK, 2016), as well as in cherry tomatoes (AN et al., 2013), both submitted to previous OD treatments.

Table 1. Effective moisture diffusivities (D_{eff}) and activation energy (E_a) for the drying processes of non-pretreated and osmotically pretreated jambolan fruits.

Temperature (ºC)	D_{eff} (m²/s)	E_a (kJ/mol)
	Non-pretreated	
50	1.45×10^{-10}	52.0
60	3.85×10^{-10}	
70	4.46×10^{-10}	
	Pretreated	
50	2.56×10^{-10}	30.2
60	3.99×10^{-10}	
70	4.93×10^{-10}	

In regards to the activation energy (E_a) (Table 1), non-pretreated samples presented higher values than the osmotically pretreated ones. In the literature, similar results have been observed for non-pretreated (44.4 kJ/mol) and pretreated (32.02 kJ/mol) strawberries (AMAMI et al., 2017). The E_a can be defined as the energy needed to displace one mol of water from the sample that is dried (MARTÍN-GÓMEZ et al., 2020). In this sense, the osmotic pretreatment has reduced the E_a demanded to dry jambolan fruits. Still, some researchers have claimed that the lower the E_a in the drying process, the greater is the D_{eff} of water in the product, which demands less thermal energy to enable the physical transformation of liquid water (PURLIS, 2019). The E_a values observed lie within the general range for drying of food products, which is comprehended between 12.7 and 110 kJ/mol (BABALIS; BELESSIOTIS, 2004).

3.2.3 Drying kinetics modeling

Mathematical modelling is the process of creating a mathematical representation of a real-world scenario, which can be used for scaling up the processing capacity or even predicting its outcome. An equation with a sufficient approximation level could be employed to predict the drying time needed to get a desired moisture content (WIKTOR et al., 2013). The optimal criteria to analyze the quality of the models was based on the highest values for R^2, lowest values for χ^2 and RMSE, followed by a random distribution of residues. The data obtained for these parameters are set in Table 2, and describe the fits to the drying processes for non-pretreated and osmotically pretreated jambolan, at 50ºC, 60ºC and 70ºC. Overall, the R^2 values were higher than 0.98, the χ^2 ranged from 1 x 10^{-5} to 2.5 x 10^{-3}, and the RMSE values were between 4 x 10^{-3} and 5 x 10^{-2}.

Table 2. Values of the coefficient of determination (R^2), reduced Chi square (χ^2), root mean square error (RMSE), and residues dispersion (RD) for the non-linear regression of the models fitted to the drying kinetic data of jambolan fruits.

Model	**Statistics**											
	50ºC				**60ºC**				**70ºC**			
	R^2	**χ^2 ($\times 10^{-4}$)**	**RMSE ($\times 10^{-2}$)**	**RD**	**R^2**	**χ^2 ($\times 10^{-5}$)**	**RMSE ($\times 10^{-2}$)**	**RD**	**R^2**	**χ^2 ($\times 10^{-5}$)**	**RMSE ($\times 10^{-2}$)**	**RD**
Non-pretreated												
1	0.97	25	5	B	0.99	13	3	B	0.99	15	4	B
2	0.99	14	4	B	0.99	10	3	B	1	3	4	R
3	0.99	14	4	B	0.99	9	3	B	1	3	2	R
4	0.98	24	5	B	0.99	13	3	B	0.99	12	3	B
5	1	1	1	B	1	4	2	R	1	3	1	B
6	1	0.4	1	R	1	2	1	R	1	1	1	R
7	0.98	25	5	B	0.99	14	3	B	0.99	14	3	B
8	1	3	2	B	1	3	2	R	1	2	1	B
9	0.99	14	4	B	0.99	10	3	B	1	3	2	R
Pretreated												
1	0.98	21	5	B	0.99	16	4	B	0.99	11	3	R
2	0.99	7	3	B	0.99	9	3	B	1	5	2	R
3	0.99	7	3	B	0.99	9	3	B	1	5	2	R
4	0.98	18	4	B	0.99	16	4	B	0.99	9	3	R
5	1	0.3	1	R	1	1	1	R	1	2	1	R
6	1	0.2	0.4	R	1	1	1	R	1	2	1	R
7	0.99	7	2	B	0.99	18	4	B	1	2	1	R
8	1	0.2	0.4	R	1	2	1	B	1	5	2	R
9	0.99	8	3	B	0.99	8	3	B	1	5	2	R

Model: (1) Newton; (2) Page; (3) Modified Page; (4) Henderson and Pabis; (5) Logarithm; (6) Midilli; (7) Two-term; (8) Diffusion approximation; (9) Two-term exponential. RD: residues dispersion. R: random. B: biased.

Although these model adjustments were within the previous optimal criteria defined previously above, the residues dispersion must be verified as well. Through a visual analysis of scatterplots from residuals versus the predicted values, it can be observed if the residuals are randomly distributed around zero with no systematic patterns, at the significance level analyzed (WISNIAK; POLISHUK, 1999; ASSIS; MORAIS; MORAIS, 2017). By these means, only the Midilli model presented a randomly distribution of residues, in all treatments studied, which indicates the goodness of fit, besides being able to predict the drying data. Still, this model has evidenced the most elevated R^2 values ($R^2 = 1$), the lowest χ^2 (< 0.00004) and RMSE (< 0.01) values, among all equations tested (Table 2). In the literature, the Midilli model displayed the best suitability on fitting the observed data of murtilla berries (PUENTE-DÍAZ et al., 2013) and kiwi berries (BIALIK et al., 2020) during drying.

3.2.4 Overall appearance of jambolan after drying

Figure 5 shows the overall appearance of non-pretreated and osmotically pretreated jambolan fruits at the final of the drying process. In terms of fruit aspects, significant changes have occurred at surface level among all different treatments, in comparison with the non-pretreated and osmotically pretreated jambolan (Figure 3). The great number of fissures and cracks at the final drying time was already reported in cranberries subjected to microwave vacuum pretreatment (100-800 W), followed by osmotic dehydration and microwave vacuum drying at microwave power of 100 W (ZIELINSKA; ZIELINSKA; MARKOWSKI, 2018).

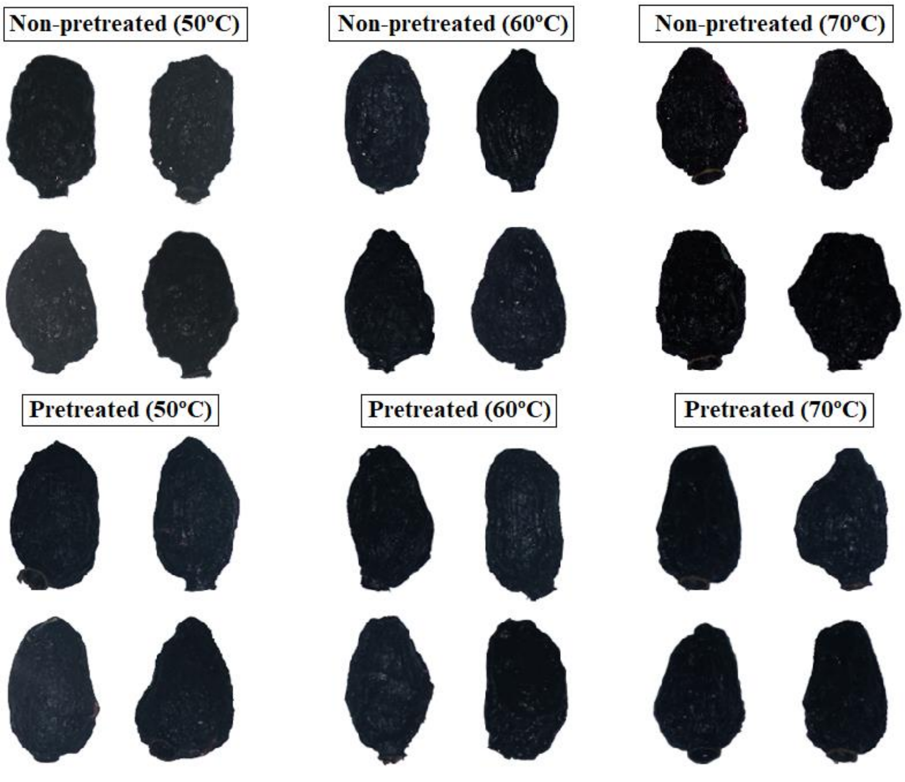

Figure 5. Overall appearance of non-pretreated and pretreated jambolan at final drying time.

Liu et al. (2022) also observed these changes on the surface of blueberries during drying; using innovative far-infrared radiation heating assisted pulsed vacuum drying. Although modifications had been perceptive, at the macroscopic level, between the osmotically dehydrated jambolan (Figure 3) and the jambolan after drying (Figure 5), it is hard to differentiate those changes among the dried treatments (Figure 5).

3.3 MONITORING ANALYSES OF JAMBOLAN AFTER DRYING

Table 3 shows the results for moisture content (MC), water activity (a_w), monomeric anthocyanins, and total phenolic contents (TPC) performed in the jambolan after the drying process at different temperatures, for the non-pretreated and osmotically pretreated jambolan.

Table 3. Moisture content (MC), water activity (a_w), monomeric anthocyanins (MA), and total phenolic contents (TPC) at the final drying time, for the non-pretreated and osmotically pretreated jambolan fruits.

Treatment	**MC**	**a_w**	**MA**	**TPC**
NPT	87.32 ± 0.13^{a}	0.980 ± < 0.001^{a}	477.89 ± 16.83^{a}	1236.13 ± 57.56^{a}
NPT (50ºC)	29.78 ± 0.26^{c}	0.712 ± 0.001^{d}	81.24 ± 5.33^{c}	1230.30 ± 34.42^{ab}
NPT (60ºC)	28.34 ± 0.77^{c}	0.758 ± 0.001^{c}	80.02 ± 0.10^{c}	952.86 ± 43.38^{c}
NPT (70ºC)	20.88 ± 0.26^{e}	0.582 ± 0.001^{g}	68.31 ± 7.97^{cd}	1041.30 ± 5.67^{bc}
PT	69.38 ± 0.56^{b}	0.959 ± 0.001^{b}	255.35 ± 15.90^{b}	906.80 ± 101.09^{c}
PT (50ºC)	24.21 ± 0.46^{d}	0.672 ±< 0.001^{e}	23.21 ± 2.38^{d}	557.88 ± 36.92^{d}
PT (60ºC)	17.34 ± 0.33^{f}	0.599 ± 0.001^{f}	46.06 ± 1.41^{cd}	1056.22 ± 15.19^{abc}
PT (70ºC)	17.79 ± 0.76^{f}	0.555 ± 0.001^{h}	40.08 ± 1.57^{cd}	854.55 ± 20.78^{c}

MC: moisture content (g/100 g wb); a_w: water activity (dimensionless); MA: monomeric anthocyanins (mg delphinidin-3-glucoside/100 g db); TPC: total phenolic contents (mg GAE/100 g db); NPT: non-pretreated; PT: pretreated. Values at the same column with different superscript letters are significantly different by the Tukey's test ($p \leq 0.05$).

3.3.1 Effect of osmotic pretreatment and drying on moisture contents and water activities

The osmotic pretreatment combined to the air-drying showed that both processes are useful tools to reduce even further the moisture content (MC) and a_w of jambolan, in comparison to dried jambolan with no pretreatment, at each temperature studied. In general, an increase in temperature has promoted a decrease in the MC and a_w values for non-pretreated (NPT) and pretreated (PT) jambolan. Regarding MC, no statistical differences were noted between NPT (50ºC) and NPT (60ºC), and between PT (60ºC) and PT (70ºC) treatments, at 95% confidence level. For a_w, all treatments were statistically different among each other ($p \leq 0.05$). The fruit's initial MC and a_w are comparable to the results found in jambolan from the Amazonian region

(ARAÚJO; PENA, 2022a; ARAÚJO; PENA, 2022b). For dried papaya slices, Chandra et al. (2020) found MC and a_w variations around 7.6-12.5% and 0.39-0.45, respectively, using ultrasonic and osmotic pretreatments, followed by convective and vacuum drying.

There are several reasons for food products to undergo OD and drying techniques, one of them is the need to preserve them by lowering the a_w, since microorganisms grow, sporulate, and germinate at different a_w levels. Pretreatment of foods before drying stages may be responsible for inactivating microorganisms and enzymes. Removal of water by drying reduces the a_w as it tends to exhibit a negative effect on the growth and survival of different microorganisms, retarding the deterioration in foods, which represents a critical step in producing safe food products. Depending on the temperature and drying exposure time, a certain amount of microbial cells can die or get sub-lethal injuries (ERKMEN; BOZOGLU, 2016; KOWALSKA et al., 2020).

The high a_w values observed in NPT and PT jambolan fruits (Table 4) are commonly noticed in high perishable foods. At this region ($a_w > 0.95$), microorganisms as *Pseudomonas, Escherichia, Proteus, Shigella, Klebisiella, Bacillus, Clostridium perfringens* and some yeasts are frequently found in fruit and vegetables. For NPT (60ºC) product, most of the halophilic bacteria and mycotoxigenic *Aspergillus* can be found ($0.75 < a_w < 0.8$). Additionally, xerophilic fungi (*Aspergillus chevalieri, candidus, Wallemia Sebi*) and *Saccharomyces bisporus* can represent a potential growth in NPT (50ºC) and PT (50ºC) products, at the a_w region between 0.65-0.75 (REID; FENNEMA, 2008). Microbial growth does not occur at $a_w < 0.60$ (SCOTT, 1957). Therefore, only NPT (70ºC) and PT (60ºC, 70ºC) samples reached values lower than the a_w limit for stablishing microbiological food safety. Brazilian legislation requires a MC limit for dehydrated fruits, which should be no longer than 25 g/100 g (BRASIL, 2005). In this sense, NPT (70ºC) and PT (50, 60, and 70ºC) samples were within the permitted limit.

3.3.2 Effect of osmotic pretreatment and drying on monomeric anthocyanins and total phenolic contents

NPT samples suffered decreases in the monomeric anthocyanins (MA) and total phenolic contents (TPC) throughout the hot air-drying, at different temperature conditions. In general, these bioactive compound losses were continually increasing as the temperature increased (Table 4). For NPT jambolan, MA and TPC retention were 17% and 99.5%, 16.8% and 77.1%, and 14.3% and 84.2%, at 50ºC, 60ºC and 70ºC, respectively. Other studies demonstrated that drying at high temperatures promotes a reduction in the MA and TPC levels, as observed in strawberries (MÉNDEZ-LAGUNAS et al., 2017), in which 26% MA and 60.9%

TPC had been lost at 50°C, while at 60°C, the losses reached 45% and 78.1%, respectively. Liu et al. (2022) reported this same phenomenon in blueberries during far-infrared radiation heating assisted pulsed vacuum drying. These researchers noticed that the MA and TPC presented a continuous decline with the constant rise of the drying temperature used (65, 70 and 75ºC).

Temperature is one of the main variables affecting anthocyanins' stability. Degradation by heat is not only dependent on temperature, but also depends on processing time, which is mainly caused by oxidation and cleavage of the covalent bonds. The flavylium cation can be transformed in quinoidal base and, later, into several intermediates, resulting in coumarin derivatives and in compounds equivalent to the B ring. Alternatively, flavylium cation could be transformed in colorless carbinol base and further into chalcone, which results in brown degradation products. Other possible explanation of anthocyanins degradation occurs similarly to the last mentioned mechanism; however, the molecule deglycolysis occurs first, leading to the loss of glycosidic bond and the formation of unstable anthocyanins, whose degradation products express brown and yellow colors (SCHWARTZ et al., 2017).

Furthermore, at higher temperatures, a reduction in the TPC during drying can be described by the release of bound phenolic compounds and partial degradation of lignin, which promotes the release of phenolic acid derivatives (MAILLARD; BERSET, 1995). Parallel to the chemical changes, physical alterations, such as collapse, shrinkage and porosity can favor the exposure of the compounds to oxygen and light. Therefore, the overall effect of thermal processing on the bioactive compounds retention is dependent by the complexity of physical and chemical phenomena (MÉNDEZ-LAGUNAS et al., 2017).

Concerning PT samples, the lowest retention of bioactive compounds were found at 50ºC, which corresponded to 9.1% and 61.5%, for MA and TPC, respectively. Thus, the prolonged drying time and the lowest temperature produced the strongest degradation. The highest MA and TPC retentions were recorded at 60ºC, which were equivalent to 18.0% and 116.5%, respectively. Garba et al. (2015) also recorded the highest MA values for black carrots during drying at 60°C, in both control and blanched samples (98ºC/3 min). Still in Table 4, when the temperature increased from 60ºC to 70ºC, the bioactive compounds retention decreased again, and accounted with 15.7% and 94.2%, for MA and TPC, respectively, in regards to the initial PT contents.

Tan et al. (2022) state that drying temperature and time can affect the quality of dried fruits and vegetables. When drying temperature is too low, enzymatic browning increases and if the temperature is too high, most of the nutrients can be destroyed. In PT samples, it is possible that WL phenomenon had promoted a change in cell membrane integrity, and resulted in loss of compartmentation of enzymes and substrates, permitting their interaction. Nunes et al. (2005) have attributed the MA and TPC losses to the accelerated WL during storage of strawberries, in which the oxidation of phenolic compounds, by polyphenol oxidase (PPO), might have induced the formation of *o*-quinones and stimulated the anthocyanin degradation by coupled oxidation mechanisms. According to Maghsoudlou, Guajari and Tavasoli (2019), phenolic compounds (as chlorogenic acid) are one the main substrates of the catecholase activity from PPO, and the oxidation process can alter the concentration of these phenolic compounds.

By the perspective of presenting a shelf-stable product during storage, PT (60ºC) jambolan appears as a good option, since the a_w level lays within the permitted limit for microbial safety. Therefore, the osmotic pretreatment, although it decreased the bioactive compound contents during OD, it represented a promising path to preserve these nutrients, since MA and TPC values remained very similar at the final drying stage. Still, PT (60ºC) product demands less drying time in comparison to the NPT dried products (Figure 4), and thus, it favors the reduction of drying costs because of energy savings.

In the end, jambolan fruit represents a formidable potential for the food and pharmaceutical industrial sectors. On the other hand, the lack of investments in technologies for its post harvesting increases the rate of residues, and results in a non-stimulating scenario for cultivation and commercialization of the fruit (SABINO et al., 2018). The present work, however, has given a promising contribution to avoid the fruit’s waste during the seasonal period, making it available for much longer by presenting a quality product under the levels required for avoiding microbial growth.

4 CONCLUSION

Combination of osmotic dehydration (OD) and hot air-drying process was studied for the first time in jambolan. OD resulted in partial water removal from the fruit, with little solid uptake. OD decreased the anthocyanins and total phenolic contents throughout time, which could be induced by its migration into the osmotic solution or either by chemical and biochemical reactions. The rise of air-drying temperature has shortened the drying time for both non-pretreated and osmotically pretreated jambolan. In addition, osmotic pretreatment has lowered the energy demands for posterior drying of jambolan, which is interesting for economic issues. Osmotically pretreated jambolan fruits submitted to drying were within the moisture content limits stablished by the Brazilian legislation. Furthermore, the fruit pretreated and dried at 60ºC has shown promising results, due to its low moisture content and water activity level, as well as its bioactive compounds contents, which were nearly the same as in non-pretreated sample (control), at 60°C. Conclusively, the present study appears as a good alternative to prolong the shelf life of the jambolan fruit and making it available for much longer.

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Capítulo VI

CONCLUSÃO GERAL

Na primeira parte da pesquisa foi possível estabelecer condições de armazenamento para a polpa e para a semente do jambolão *in natura*, na faixa de temperatura de 25°C a 55°C. As regiões que representam a maior estabilidade para os produtos correspondem a níveis de umidades de 8 g H_2O/100 g (base seca – bs) e 5,1 g H_2O/100 g bs, para a polpa e a semente do fruto, respectivamente. As isotermas de dessorção de umidade indicaram que o comportamento higroscópico da polpa do jambolão se assemelhou a alimentos ricos em sólidos solúveis, enquanto as sementes do fruto se comportaram como produto proteico. Ademais, a polpa do jambolão é mais higroscópica que a semente do fruto. O estudo também abordou as demandas energéticas envolvidas no processo de dessorção da polpa e da semente do fruto e indicou que a secagem da polpa do jambolão demanda maior quantidade de energia do que a secagem da semente.

Na segunda parte da pesquisa, a análise biométrica mostrou que a parte comestível do jambolão (polpa + casca) representa mais de 70% do peso do fruto, e a análise química mostrou que a parte comestível do fruto é uma fonte potencial de compostos fenólicos e antocianinas, o que indica o potencial tecnológico e funcional do jambolão. O processo de triagem das variáveis, envolvidas no processo de desidratação osmótica (DO) do jambolão, indicou que a temperatura, a concentração de sacarose, a concentração de lactato de cálcio e a pressão exibem efeitos significativos sobre a resposta perda de água; variável de maior relevância para a DO.

Na terceira e última parte da pesquisa, a cinética de DO do jambolão mostrou a retirada parcial de água do fruto, com pequena incorporação de sólidos. Foi observada a redução de compostos fenólicos e antocianinas durante a cinética de desidratação osmótica (retenção de 53,43% para antocianinas monoméricas e de 73,11%, para compostos fenólicos totais), atribuída à migração destes compostos do produto para a solução osmótica ou a reações químicas e bioquímicas, durante a DO. A pesquisa mostrou ainda que, amostras de jambolão pré-tratadas por DO, apresentam menores demandas de energia durante a secagem convectiva do fruto, por exigirem menores tempos de secagem, quando comparadas com amostras não submetidas a DO. Finalmente, os frutos de jambolão submetidos a DO, por 72 h, em solução osmótica (60% sacarose/ 4% lactato de cálcio), à temperatura ambiente e à secagem convectiva por 15 h a 60°C, apresentam boa estabilidade microbiológica durante o armazenamento, bem como teores de compostos fenólicos e antocianinas próximos aos observados para amostras não submetidas a DO. Assim, a combinação da DO com a secagem convectiva se apresenta como uma importante alternativa tecnológica para a conservação do jambolão.

APÊNDICES

APÊNDICE A

Capa do artigo 1: Moisture desorption behavior and thermodynamic properties of pulp and seed of jambolan (*Syzygium cumini*)

Contents lists available at ScienceDirect

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journal homepage: www.cell.com/heliyon

Research article

Moisture desorption behavior and thermodynamic properties of pulp and seed of jambolan (*Syzygium cumini*)

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ARTICLE INFO

Keywords:
Myrtaceae
Equilibrium moisture
Mathematical modeling
Thermodynamics

ABSTRACT

The study objectives were to establish isotherms and thermodynamic properties for the moisture desorption process of jambolan pulp (JP) and jambolan seed (JS), harvested in the city of Belém (Brazil). These characteristics can contribute for the proper selection of the operating drying conditions. Thus, the following essays were made for both JP and JS. Firstly, proximate composition was performed, followed by moisture desorption essays at 25, 35, 45 and 55 °C. In addition, six mathematical models were fitted to the experimental data to simulate the desorption behavior; and based on the chosen models, the thermodynamic properties were calculated. The results have shown that JP isotherms followed the typical behavior of products rich soluble solids, and JS isotherms were more influenced by protein components. The influence of temperature was evidenced throughout the entire range of water activity (a_w) studied. The GAB and Oswin models represented the best fitted equations for the JP and JS, respectively. In general, the energies involved in the desorption process of jambolan showed a greater dependence of JP with the equilibrium moisture content (EMC), in comparison with JS. Still, there was an increasing tendency of the thermodynamic properties with EMC decreasing. Besides of being non-spontaneous processes, desorption phenomena of JP and JS were enthalpy-driven mechanisms.

1. Introduction

Syzygium cumini is a tropical fruit from the *Myrtaceae* family, popularly known as jambolan, jamun, black plum or java plum. This berry presents an ovoid form, a skin varying from purple-red to black color in the ripe stages, and a total length of 2–3 cm. It also presents an astringent taste in a freshly pink/white pulp (Faria et al., 2011). All parts of the fruit, besides their nutritional constituents, contain many phytochemicals, and thus, they are widely used for medicinal purposes (Paul and Das, 2019). Some of the fruit's constituents have been attributed to the presence of anthocyanins, flavonoids, and terpenes (Aqil et al., 2014).

Jambolan, however, presents a short seasonal availability and a high perishable nature, which enhances the use of preservative techniques for lowering the moisture content of these fruits and make them available throughout the year (Paul and Das, 2019). There are still few works regarding the use of new processes that can improve the shelf life of jambolan; in this sense, drying can be used as the preservative process and the dried pulp could be used as food ingredient in the preparation of new products. Moreover, due to the large application in the traditional medicine, jambolan pulp and seed have a great potential for commercialization in the dried state, facilitating the transportation and storage stages as well.

In order to optimize the drying processes and guarantee stability of the dried products during storage, the knowledge of the product's moisture sorption isotherms is necessary, which expresses the graphical relationship between the food's equilibrium moisture content (EMC) and its correspondence water activity (a_w), over a range of a_w values and at constant temperature (Damodaran, 2017).

Brunauer et al. (1938) classified sorption isotherms by their shape and processes. Type II is one of the most frequent isotherms in foods, and may be classified into three zones. In general, a hypothetical food system of the first zone is associated with small quantities of water and its molecules are tightly bound to polar sites. In zone II, the water is less bound, occurring as multilayer, right after the monolayer. At this region, chemical and biochemical reactions take place. In zone III, water fills the macro capillaries, exhibiting almost the full properties of bulk water, as microbial growth becomes the major deteriorative reaction (Aguilera and Stanley, 1999).

Furthermore, foodstuffs rich in soluble compounds, such as sugars, usually behave as type III, according to the classification mentioned in

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https://doi.org/10.1016/j.heliyon.2022.e09443
Received 29 October 2021; Received in revised form 3 January 2022; Accepted 11 May 2022

APÊNDICE B

Capa do artigo 2: Influence of process conditions on the mass transfer of osmotically dehydrated jambolan fruits

Original Article
Food Science and Technology

ISSN 0101-2061 (Print)
ISSN 1678-457X (Online)

DOI: https://doi.org/10.1590/fst.37520

Influence of process conditions on the mass transfer of osmotically dehydrated jambolan fruits

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Abstract

Jambolan (*Syzygium cumini*) is a tropical fruit rich in anthocyanin pigments, but its fragile skin and pulp present low protection against physical damages and microorganisms. In this sense, a preservative technology, as osmotic dehydration (OD), was studied to investigate the impact of some variables over this process. At first, fruits of jambolan were submitted to physical and physical-chemical analysis. Furthermore, whole fruits underwent OD following a fractional factorial design. The influence of the process variables: temperature (20-50 °C), sucrose concentration (30-60%), pressure (10-90 kPa), vacuum pulse time (5-15 min), calcium lactate concentration (0-4%) and number of vacuum pulse (1-3) was assessed on water loss (WL), solid gain (SG) and weight reduction (WR). In general, biometric analysis showed positive and significant correlation among the physical characteristics of jambolan. Physical-chemical assay demonstrated that the fresh fruit presents a potential source of bioactive compounds. The screening design showed that temperature, sucrose concentration, calcium lactate concentration and pressure affected the WL in jambolan. On the other hand, vacuum pulse time and number of vacuum pulse showed no influence on the WL, SG and WR; therefore, these variables must be fixed at the most economically viable level for any further trials.

Keywords: dehydration; morphometric characteristic; design of experiments.

Practical Application: The present work can improve the control of the unitary operation (dehydration) in OD of jambolan.

1 Introduction

The Amazon hosts a large biological diversity, including great number of non-native plant species, which have been successfully adapted to the Amazonian environmental conditions (Brito et al., 2017). Native from India, jambolan, janun or black plum (*Syzygium cumini*) fruits are ellipsoid berries from the *Myrtaceae* family, which present themselves as purplish black, with a sour taste at the ripe stages of maturation (Seraglio et al., 2018). This tropical fruit is also rich in anthocyanin pigments, especially in its peel (Sari et al., 2012).

However, the relatively short shelf life of fresh fruits after harvesting indicates the need for developing an efficient and cheap preservation process. Besides, the rising search for products with similar sensory and nutritional properties to fresh fruits also stimulates the food industry to seek for preservation methods in foods (Sridevi & Genitha, 2012). In this scenario, osmotic dehydration (OD) is growing as a popular technique for obtaining processed fruits, improving their quality and stability (Ahmed et al., 2016).

Commonly, OD is a pre-treatment prior to air-drying. This technique is based on the immersion of fruits in hypertonic solution for partial water removal. The driving force for its removal is the difference in osmotic pressure between the fruit and the hypertonic solution. The fruit's complex cell structure acts as a semi-permeable membrane, generating extra resistance to water diffusion within the fruit. This mass transfer depends on some factors, such as the product's geometry, temperature, addition of salts and concentration of the osmotic solution, among others (Torreggiani, 1993).

Traditionally, the osmotic process is employed at atmospheric pressure; however, in recent decades, it has been combined with other innovative techniques, such as vacuum application, which can enhance its efficiency and improve the quality of the product (Ahmed et al., 2016). Some researchers have studied the use of vacuum in OD to accelerate mass transfer in products such as guavas (Corrêa et al., 2010) and tomato (Corrêa et al., 2015), among other works involving the study of vacuum pulse time (Ito et al., 2007) and number of vacuum pulse (Zapata et al., 2016).

Due to the perishability of jambolan, especially by the fragility of its skin and pulp, that offer low protection against mechanical damages and infectious agents, harvest and post-harvest management are essential for extending the fruit's shelf life. In this sense, some studies have assessed the drying of jambolan fruit using different techniques, including air forced drying (Kapoor et al., 2015), foam mat drying (Carvalho et al., 2017) and spray-drying (Singh et al., 2019). However, no studies have been reported regarding OD techniques for jambolan fruits.

In this context, the objectives of this work were to assess the physical and physical-chemical characterization of jambolan, as well as to use a fractional factorial design for investigating the

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Received 09 Aug., 2020
Accepted 15 Sept., 2020

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