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CAROLINA CASTILHO GARCIA

AVALIAÇÃO DA DESIDRATAÇÃO DE MAMÃO UTILIZANDO MÉTODOS COMBINADOS

SÃO JOSÉ DO RIO PRETO
2012

CAROLINA CASTILHO GARCIA

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Tese apresentada ao Instituto de Biociências, Letras e Ciências Exatas da Universidade Estadual Paulista "Júlio de Mesquita Filho", Campus de São José do Rio Preto, para obtenção do título de Doutor em Engenharia e Ciência de Alimentos, área de Engenharia de Alimentos.

Orientadora: Prof[a]. Dr[a]. Maria Aparecida Mauro

Co-orientadora: Prof[a]. Dr[a]. Mieko Kimura

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Prof. Dr. Joel Fernando Nicoleti

São José do Rio Preto
06 de fevereiro de 2012

Aos meus queridos filhos, Matheus, Pedro e Gabriel.
Com todo amor do mundo.
À minha mãe, Myrian, e à grande amiga Cida,
Exemplos a serem seguidos.
À Lica, melhor irmã do mundo.

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Ao meu esposo e aos meus filhos, aos meus queridos avós e aos meus irmãos pelo apoio, compreensão e paciência.

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À todos MUITO OBRIGADA!

“...
O que a gente não pode
mesmo, nunca, de jeito nenhum,
é amar mais ou menos,
é sonhar mais ou menos,
é ser amigo mais ou menos,
é namorar mais ou menos,
é ter fé mais ou menos
e acreditar mais ou menos.

Senão a gente corre o risco de se tornar
uma pessoa mais ou menos.”

Chico Xavier

RESUMO

Com o intuito de agregar valor a frutas nacionais desidratadas, os efeitos de pré-tratamentos sobre a secagem de mamão Formosa foram avaliados. Para tanto, foram utilizados previamente à secagem convectiva o branqueamento, a desidratação osmótica (DO) e a aplicação de coberturas comestíveis na superfície das frutas frescas e osmoticamente desidratadas. Com base em um planejamento fatorial, verificou-se que a DO das fatias de mamão frescas e branqueadas em solução de sacarose a 50% p/p adicionadas de 1% p/p de lactato de cálcio e 0,1 M de ácido lático por 120 min e 105 min, respectivamente, foram as condições que ocasionaram redução na atividade de água das amostras e pequenas variações em sua cor. A cinética de DO das frutas frescas e branqueadas mostrou que o branqueamento resultou em maiores coeficientes de difusão da água e dos solutos. Melhores ajustes para as difusividades das fatias de fruta foram verificados considerando-as como placas infinitas. Os resultados evidenciaram grande atividade de invertase no mamão, o qual se apresentou como uma fonte potencial para a extração da referida enzima. As isotermas de sorção de fatias frescas, branqueadas e desidratadas osmoticamente de mamão Formosa mostraram que mais energia foi necessária para a remoção da água das amostras osmoticamente tratadas e que a dessorção das frutas branqueadas exigiu menor quantidade de energia. O branqueamento danificou a estrutura celular dos mamões resultando em maiores coeficientes de difusão da água que as frutas frescas durante a secagem. A desidratação osmótica dos mamões e a cobertura aplicada na superfície das frutas também resultaram em maiores coeficientes de difusão da água em relação às amostras frescas. A cobertura exerceu efeito protetor sobre o conteúdo de carotenóides das amostras quando aplicada à superfície das frutas frescas. A retenção de vitamina C das frutas frescas cobertas foi maior que a das frescas não cobertas e que a das branqueadas. A desidratação osmótica não exerceu efeito protetor sobre o conteúdo nutricional dos mamões, resultando em frutas secas com teor reduzido de vitamina C.

Palavras-chave: Secagem, Desidratação osmótica, Branqueamento, Cobertura comestível, Mamão Formosa

ABSTRACT

Aiming to add value to national dehydrated fruits, the effects of pre-treatments on convective drying of papaya of Formosa cultivar were investigated. Blanching, osmotic dehydration (OD) and edible coating application at the surface of fresh and osmotically dehydrated fruits were evaluated. Based on a factorial design, it was verified that OD of fresh and blanched papayas sucrose solutions at 50% w/w added with 1% w/w of calcium lactate and 0,1 M of lactic acid for 120 and 105 min, respectively, were the conditions resulting in decreased water activity and small color changes. OD kinetics of fresh and blanched fruits showed that blanching resulted in higher water and solutes diffusion coefficients. The best adjustments for the fruit diffusivities were obtained considering them as infinite slabs. The results revealed the great invertase activity in papaya which presented as a potential source for the referred enzyme extraction. Sorption isotherms of fresh, blanched and osmo-treated papayas showed that more energy was necessary to remove water from osmotically dehydrated samples and the dessorption of blanched fruits needed lower quantity of energy. Blanching damaged the cellular structure of papaya resulting in higher moisture diffusivities than the fresh fruits during drying. Osmotic dehydration and the coating applied at the surface of the fruits also resulted in higher moisture diffusion coefficients in relation to the fresh samples. Coating presented a protective effect on carotenoids content of the samples when applied at fresh fruits surface. Retention of vitamin C of fresh coated fruits was higher than the non-coated fresh and blanched papayas. Osmotic treatment did not have a protective effect on nutritional compounds of papayas, resulting in dried fruits with reduced vitamin C content.

Keywords: Drying, Osmotic dehydration, blanching, Edible coating, papaya of Formosa cultivar

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1. INTRODUÇÃO

O Brasil é um país de ampla extensão territorial cuja área de lavouras ultrapassou 76 milhões de hectares, em 2006 (IBGE, 2011). Em 2009, do total plantado, 2.179.250 de hectares foram ocupados pela fruticultura, ultrapassando 41 milhões de toneladas de frutas produzidas, sendo a laranja e a banana as frutas de maior produção (IBRAF, 2011). Segundo dados da FAO e do Instituto Brasileiro de Frutas, em 2009, o Brasil foi o segundo maior produtor de mamões do mundo, com uma produção de aproximadamente 2 milhões de toneladas, o equivalente a cerca de 509 milhões de dólares.

As exportações brasileiras de frutas cresceram 25% nos últimos cinco anos. Em 2006, o setor movimentou US$ 700 mil, alcançando mais de US$ 875 mil em 2010 (GLOBO RURAL ONLINE, 2011). O mamão está entre as fruta *in natura* mais exportada do país, sendo que o volume de mamão papaia fresco exportado foi de 7,6 mil toneladas em 2010 (PITOMBO, 2011).

O mamão é uma fruta típica de regiões de climas tropicais e subtropicais que possui período de safra relativamente longo. É fonte de carotenóides, vitamina C e sais minerais. Estudos mostram que esse fruto não apresenta alta atividade pró-vitamina A, porém, por ser popular, de baixo custo e disponível quase o ano todo, torna-se importante fonte de pró-vitamina A (RODRIGUEZ-AMAYA, 1996).

Dentre as causas de perdas pós-colheita, destacam-se aquelas devidas à ocorrência de injúrias mecânicas, que podem ser agrupadas em injúrias por impacto, compressão e corte. Tais injúrias ocasionam danos irreparáveis nos produtos, reduzindo sua vida útil e provocando sua desvalorização comercial (DURIGAN et al., 2005).

Segundo Durigan et al. (2005) e Luengo et al. (2001) do instante em que são colhidos até serem consumidos, os produtos hortícolas sofrem uma série de injúrias mecânicas. Dependendo da sensibilidade do produto, essas injúrias ocasionarão danos que comprometerão a qualidade final do produto, ocasionando perdas de cerca de 20 a 25 % do total colhido. As perdas na colheita e pós-colheita são elevadas no Brasil, da ordem de 30% a 45%, justificando medidas para resolver o problema.

Assim, o processamento de partes da fruta que seriam perdidas devido à injúrias, baseado em tecnologias viáveis para pequena escala e que possibilite melhorar a qualidade do produto, contribuiria para a redução das perdas devido a impossibilidade de comercialização da fruta fresca, agregando valor ao produto final.

A apresentação do trabalho foi dividida em introdução, objetivos, revisão bibliográfica geral sobre os temas abordados e cinco capítulos de resultados, na forma de artigos, contendo introdução, materiais e métodos, resultados e discussões, conclusões e referências bibliográficas. Ao final, foram apresentadas as conclusões gerais e as referências bibliográficas gerais.

- Capítulo I: aborda os estudos realizados para a obtenção das melhores condições de desidratação osmótica de fatias frescas e branqueadas de mamão Formosa. Apresenta ainda dados sobre a influência do branqueamento, da desidratação osmótica e da aplicação de cobertura de pectina na cor e atividade de água das frutas após secagem convectiva a 70 °C.

- Capítulo II: apresenta os resultados da cinética de desidratação osmótica dos mamões frescos e branqueados e a caracterização da enzima β-fructofuranosidase (invertase) presente nas frutas.

- Capítulo III: apresenta os dados de sorção das frutas frescas, branqueadas e desidratadas osmoticamente.

- Capítulo IV: aborda os estudos sobre a influência do branqueamento e da aplicação de cobertura de pectina na cinética de secagem, atividade de água, cor e conteúdo de vitamina C de fatias de mamão. As alterações causadas pelos pré-tratamentos e secagem a nível celular foram avaliadas através de Microscopia de Luz e Eletrônica de Transmissão.

- Capítulo V: apresenta os estudos sobre a influência da desidratação osmótica na cinética de secagem, atividade de água, cor e conteúdo de vitamina C de fatias de mamão. As alterações causadas pelo pré-tratamento e secagem a nível celular foram avaliadas através de Microscopia de Luz e Eletrônica de Transmissão.

2. Objetivos

2.1. Objetivos gerais

Estudar a influência de pré-tratamentos sobre a cinética de secagem convectiva de mamão Formosa visando a obtenção da fruta a atividades de água menores que 0,6, para garantir sua segurança microbiológica, e ainda com boas características físicas e químicas.

2.2. Objetivos específicos

- Selecionar as melhores condições de desidratação osmótica de fatias de mamão Formosa frescas e branqueadas com base na manutenção da cor e redução na atividade de água.
- Avaliar a cinética de desidratação osmótica de mamões frescos e branqueados.
- Determinar isotermas de sorção dos mamões frescos, branqueados e desidratados osmoticamente.
- Investigar a influência do branqueamento, da desidratação osmótica, da aplicação de coberturas e combinações dos mesmos sobre a cinética de secagem de fatias da fruta.
- Avaliar o processo de secagem com base em sua cinética e em características físicas e químicas dos produtos: cor, volume, atividade de água e conteúdo de vitamina C.

- Avaliar as alterações causadas pelos pré-tratamentos e secagem na estrutura celular das frutas através de Microscopia de Luz e Eletrônica de Transmissão.

3. REVISÃO BIBLIOGRÁFICA

3.1. Mamão

O mamão (*Carica papaya*) é uma fruta climatérica, originária da América tropical, que se apresenta como uma baga de forma variada (oblonga, arredondada, alongada ou piriforme), de casca fina e lisa e coloração variando do amarelo-claro ao alaranjado. As sementes são numerosas, arredondadas, rugosas e de coloração dependente do tipo da cultivar (DANTAS, 2000; PAULL et al., 1997). Cultivares de mamão de polpa vermelha, Solo e Formosa, apresentam composições semelhantes de β-caroteno (2,5 e 3,7 μg/g, respectivamente), β-criptoxantina (9,1 e 7 μg/g) e licopeno (21 e 22,8 μg/g) (KIMURA et al., 1991). A atividade pró-vitamina A em mamão de polpa vermelha variedade Solo, corresponde a 12 μg/g (GODOY; RODRIGUEZ-AMAYA, 1994).

A vitamina C é um importante antioxidante natural solúvel em água encontrado preferencialmente em frutas e vegetais. Além de prevenir contra o escorbuto, a vitamina C desempenha um importante papel como antioxidante natural. A referida vitamina possui quatro isômeros, porém apenas os ácidos L-ascórbico e araboascórbico (eritórbico) possuem atividade biológica. Em sua forma pura cristalina, o ácido ascórbico é estável quando exposto à luz, ar e temperatura do ambiente por longo período de tempo. Porém, quando em solução aquosa ou nos alimentos sua estabilidade dependerá das condições de armazenamento e composição da matriz alimentícia. Assim, ela será facilmente degradada dependendo de muitos fatores, tais como, pH, temperatura, luz e presença de enzimas, oxigênio e catalisadores metálicos. Por esses motivos a vitamina C é um indicador de qualidade no processamento de alimentos (SANTOS; SILVA, 2008).

Muitos animais são capazes de sintetizar a vitamina C, mas não os humanos, que precisam ingeri-la em sua dieta alimentar. Em 100 g de mamão Formosa são encontrados, em média, 79 μg dessa vitamina (TACO, 2011).

A Tabela 1 apresenta a composição química do mamão Formosa utilizado nos experimentos. Porém, ela pode variar em função da cultivar, práticas culturais, da fertilidade do solo, da época do ano, do grau de maturação, e de outros fatores.

Tabela 1. Composição centesimal do mamão Formosa utilizado nos experimentos.

Umidade	90,74% ± 0,03
Açúcares Totais	4,69% ± 0,02
Açúcares Redutores	4,04% ± 0,07
Proteínas	0,36% ± 0,06
Carboidratos*	8,90%
Cinzas	0,24% ± 0,19
a_w	0,988 ± 0,004
Vitamina C	58,43 ± 7,79 mg/100 g amostra

*calculado por diferença.

Há mais de 57 espécies conhecidas de mamão, mas, no Brasil, são plantados três tipos diferentes: mamão comum, Papaia (“Solo” e “Sunrise-solo”) e, mais recentemente, o Formosa. O Papaia e o Formosa são muito bem aceitos no mercado, pelo sabor e pelos preços. O Formosa, maior, resiste melhor ao transporte, possui teor de açúcar mais elevado, facilidade para obtenção de sementes e grande estabilidade genética (BAHIA, 2011).

Todos os procedimentos pós-colheita do mamão Formosa são realizados sob temperatura ambiente. Na comercialização a granel, a qualidade é comprometida por danos mecânicos, como arranhões, cortes e abrasões, que favorecem a incidência de doenças aumentando, conseqüentemente, as perdas (DURIGAN et al., 2005). Sob temperatura ambiente (27,4 ºC) a vida útil pós-colheita é estimada em seis dias, ocorrendo, posteriormente, murchamento e intensa infestação de patógenos (RIBEIRO, 2002).

O mamão é um fruto muito sensível às variações de temperatura, de maneira que seu consumo de oxigênio aumenta quando submetido ao tratamento térmico (46-47 °C por 20 min), indicando elevação da taxa respiratória e, conseqüentemente, mais rápida maturação. Quando expostos ao mesmo tratamento e posteriormente armazenados sob refrigeração (10 °C) por uma semana, ocorre decréscimo na velocidade da respiração e desaceleração no processo de maturação (MOLINARI, 2007).

Rocha et al. (2007) encontraram a temperatura de 10 ^{o}C como a melhor estimativa para a extensão da vida útil pós colheita, considerando acidez, teor de sólidos solúveis e retenção de vitamina C. Segundo Molinari et al. (2007), quando adequadamente conduzido, o

mamão pode ter uma vida pós-colheita de 4 a 6 dias sob condições ambiente (25 °C a 28 °C), ou de três semanas sob baixas temperaturas (10 °C a 12 °C).

Efeitos sobre a saúde, relacionados ao consumo de frutas e hortaliças, têm sido atribuídos à presença de compostos antioxidantes, especialmente o ácido ascórbico (vitamina C), o α-tocoferol e o β-caroteno (KAUR; KAPOOR, 2001). Porém, esses compostos são muito susceptíveis a degradações, especialmente durante a secagem e posterior armazenamento.

3.2. Secagem

A secagem é um dos métodos mais antigos e utilizados para a conservação de alimentos, pois diminui a disponibilidade de água para reações de deterioração e promove uma considerável redução de custos em transporte e manipulação do produto (DOYMAZ; GÖL, 2011; DOYMAZ, 2010). A secagem é uma operação complexa, pois envolve transferência de massa e de calor simultâneas, em regime transiente, juntamente com transformações bio-físico-químicas (MUJUMDAR, 2004).

A transferência de calor depende da temperatura, umidade e fluxo do ar, da área exposta e espessura dos alimentos e pressão. Os fatores que governam a velocidade dos mecanismos de transferência, tais como, pressão de vapor do material e do ar de secagem, temperatura e velocidade do ar, velocidade de difusão da água no material, espessura e superfície exposta para secagem, determinam a taxa de secagem (PEDRO, 2005; EL-AOUAR, 2005).

O processo de secagem pode ser dividido em três:

1. Período de indução: no qual a temperatura do alimento e a pressão de vapor da água aumentam gradativamente até a temperatura de bulbo úmido do ar.

2. Período de taxa constante: no qual a taxa de secagem é constante. Neste período ocorre a evaporação da água livre do alimento. Enquanto a migração de água do interior até a superfície do produto for suficiente para acompanhar a perda por evaporação de água na superfície, a taxa de secagem será crescente. Segundo El-Aouar (2005) para os materiais biológicos, é difícil a existência deste período, porque, devido às condições operacionais de secagem, as resistências às transferências de massa encontram-se principalmente no interior do produto, fazendo com que a taxa de evaporação da superfície ao ambiente seja superior à taxa de migração da umidade do interior à superfície do material. Em vários estudos de

secagem de outras frutas, não foi detectado o período de taxa constante (DOYMAZ, 2010; GÓNZALEZ-FÉSLER et al., 2008; NIETO et al., 2001; ALVAREZ et al., 1995).

3. Período de taxa decrescente: A partir do momento em que a quantidade de água começa a ser deficiente na superfície do sólido, a velocidade de secagem diminui, o que caracteriza o início do chamado período de taxa decrescente. Ao teor de umidade do sólido intermediário aos períodos de taxa constante e de taxa decrescente, dá-se o nome de umidade crítica. Neste período a troca de calor não é mais compensada, conseqüentemente, a temperatura do produto aumenta e tende assintoticamente à temperatura do ar. Esta redução da taxa de secagem é devido ao abaixamento da pressão parcial de vapor de água na superfície do sólido. (PEDRO, 2005; EL-AOUAR, 2005).

No período de taxa decrescente o mecanismo dominante é a difusão da água do interior do produto até sua superfície, portanto, a principal teoria utilizada para interpretar a secagem de alimentos é baseada na teoria da difusão da umidade como líquido ou vapor, descrita matematicamente pela Segunda Lei de Fick (CRANK, 1975):

$$\frac{\partial X}{\partial t} = \nabla.\left(D_{ef} \nabla X \right) \tag{1}$$

em que: X representa a umidade (b.s.), t o tempo e D_{ef} a difusividade efetiva.

A difusividade calculada com base na Equação 1 é uma difusividade efetiva, que engloba todos os efeitos que podem intervir no fenômeno. As soluções para a Segunda Lei de Fick aplicam-se a sólidos de forma geométrica simples e constante ao longo do processo. Por exemplo, a solução analítica da equação de difusão aplicada a uma placa infinita (Equação 2) está descrita em Crank (1975):

$$\boldsymbol{X} = \left(\frac{\overline{X}(t) - X^{eq}}{X^{0} - X^{eq}} \right) = \frac{8}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{(2n-1)^2} \exp\left[-(2n-1)^2 \frac{\pi^2 D_m t}{z^2} \right] \tag{2}$$

em que: o sobrescrito *eq* indica equilíbrio e o *0*, tempo inicial; $\boldsymbol{X}$ é o adimensional de umidade; X representa o conteúdo de umidade (b.s.); $\overline{X}$ é a umidade (b.s.) no tempo t; D_m é o coeficiente de difusão efetivo da umidade; z é a espessura das amostras.

Há trabalhos que utilizam modelos empíricos como o modelo exponencial e de Page (DOYMAZ; GÖL, 2011; GARCIA et al., 2007; EL-AOUAR et al., 2003) que relacionam a umidade adimensional ($\boldsymbol{X}$) como uma função exponencial do tempo, obtendo resultados aceitáveis.

O efeito da temperatura do ar de secagem no tempo de secagem é característico: aumentando a temperatura de secagem verifica-se diminuição no tempo de secagem (DOYMAZ; GÖL, 2011; DOYMAZ, 2010; SHI et al., 2008; EL-AOUAR et al., 2003). Da mesma maneira, utilizando-se amostras mais espessas obtêm-se maiores tempos de secagem (DOYMAZ; GÖL, 2011).

Dentre os métodos existentes de secagem, a convectiva é responsável por mais de 90% da produção de alimentos desidratados, a despeito do fato de outros tipos de secadores proporcionarem algumas vantagens importantes em termos de eficiência energética, qualidade do produto e impacto ao meio ambiente (MUJUMDAR, 1997).

A despeito das inúmeras vantagens, a natureza não linear dos fenômenos envolvidos na secagem dificulta o controle do processo, o que muitas vezes leva a alterações indesejáveis no produto. Assim, diversos estudos foram realizados a respeito do efeito da secagem em características sensoriais e nutricionais do produto final e da utilização de pré-tratamentos como uma alternativa para melhorar estas características.

O efeito de diferentes umidades relativas, temperaturas e velocidades do ar de secagem no tempo de processamento e conteúdo de vitamina C de fatias de kiwi foi estudado por Kaya et al. (2009). Os pesquisadores fixaram a umidade relativa do ar de secagem e observaram que as maiores temperaturas causaram maior degradação da vitamina C. Menores tempos de secagem foram obtidos por Doymaz (2008) durante a secagem de alho-poró previamente branqueado quando comparado às amostras não branqueadas. Aumento da difusividade efetiva da água na secagem a vácuo de cenouras e abóboras congeladas e branqueadas foi observado por Arévalo-Pinedo e Murr (2007), o que foi atribuído aos danos causados ao tecido vegetal pelos pré-processamentos.

Leeratanarak et al. (2006) estudaram o efeito do branqueamento aplicado previamente à secagem em secador convectivo e de baixa pressão sobre a cor, a textura e o acúmulo de pigmentos escuros em chips de batata. Os autores concluíram que o branqueamento auxiliou na manutenção da cor e diminuição do índice de escurecimento dos vegetais processados em menores tempos de secagem. O branqueamento em água quente ocasionou perdas de açúcares redutores da batata, impossibilitando a ocorrência da reação de Maillard, responsável pelo escurecimento não-enzimático das amostras.

Garcia et al. (2007) estudaram o efeito da desidratação osmótica na secagem de fatias de abóbora. Foi verificado que as amostras pré-tratadas previamente à secagem apresentaram maior conteúdo final de umidade, o que foi atribuído à alta higroscopicidade da sacarose. Os coeficientes de difusão da água das abóboras desidratadas osmoticamente resultaram maiores

que os das amostras frescas durante a secagem, o que foi relacionado à secagem rápida da superfície das amostras *in natura*, formando pontos endurecidos (“crosta”), que diminuiriam as taxas de secagem.

O tratamento osmótico aplicado previamente à secagem é capaz de conferir características desejáveis ao produto final, quando comparado ao produto seco sem pré-tratamento, como maior porosidade (RODRIGUES, 2003), efeito protetor sobre a estrutura do alimento, gerando produtos mais flexíveis e macios (LENART, 1996, MANDALA et al., 2005), melhor aceitação sensorial (SHIGEMATSU et al., 2005) e maior retenção de nutrientes (TONON et al., 2007; NASCIMENTO, 2006; SHI et al., 1999; HENG et al., 1990).

Shi et al. (1999) trabalhando com a desidratação osmótica de tomates seguida de secagem verificaram que durante o processo osmótico não ocorreram perdas de licopeno das amostras, sendo a isomerização da forma *trans* para a *cis* o mecanismo predominante. O processo osmótico, devido à impregnação de açúcares na superfície dos vegetais, protegeu os carotenóides presentes no tomate contra a oxidação durante a secagem.

A aplicação de coberturas comestíveis anteriormente à secagem de abóboras foi avaliada por Lago (2007). Observou-se maior retenção dos carotenóides das amostras cobertas em comparação às frescas, o que foi atribuído à proteção que as coberturas exerceram contra a oxidação dos pigmentos. Resultados semelhantes foram observados por Zhao e Chang (1995) para cubos de cenoura cobertos com amido de milho previamente à secagem.

3.3. Desidratação Osmótica

A desidratação osmótica (DO) é uma técnica que envolve a imersão do produto em soluções aquosas hipertônicas (SERENO et al., 2001) e está diretamente relacionada à remoção de água de frutas e vegetais (RASTOGI et al., 1997). A complexa estrutura da parede celular e das membranas celulares dos vegetais, que são parcialmente seletivas, propicia três tipos de transferência de massa com fluxo contra-corrente: fluxo de água do sólido para solução, transferência de soluto da solução para o sólido e fluxo de solutos do sólido para a solução (RAOULT-WACK, 1994). O resultado desta transferência é uma diminuição do conteúdo de umidade com um incremento simultâneo de sólidos, além de uma modificação na composição química do alimento desidratado (LENART, 1996).

A característica diferencial da desidratação osmótica em relação a outros processos de desidratação de alimentos é a possibilidade de modificar a sua formulação através da

incorporação de solutos na estrutura porosa das frutas e hortaliças. Esses solutos podem ser depressores de pH ou de atividade de água, componentes fisiologicamente ativos, antimicrobianos, entre outros, que possam favorecer a preservação sensorial e nutricional dos produtos, além de formular produtos funcionais, estáveis e mais próximos aos frutos frescos (TORREGGIANI; BERTOLO et al., 2001; FITO et al., 2001). O cálcio tem recebido considerável atenção nos últimos anos devido aos seus efeitos desejáveis na manutenção da firmeza do tecido (GÓNZALEZ-FÉSLER et al., 2008; ANINO et al., 2006; BARRERA et al., 2004), retardamento da senescência e controle das desordens fisiológicas em frutas e hortaliças inteiras (MARTÍN-DIANA et al., 2007).

Goiabas desidratadas osmoticamente em soluções de sacarose e maltose adicionadas de lactato de cálcio apresentaram maiores valores de tensão na ruptura do que as amostras osmo-tratadas sem o cálcio. (PEREIRA et al., 2009; MASTRANTONIO et al., 2005).

A DO é uma técnica normalmente utilizada como tratamento prévio a outros processamentos, tais como secagem a vácuo ou por ar aquecido, liofilização e congelamento. Melhoria nas características sensoriais e nutricionais dos produtos desidratados osmoticamente previamente a outros tratamentos foi observada por vários autores (FERRARI, 2009; OSORIO et al., 2007; HEREDIA et al., 2007; RODRIGUES, 2005; EL-AOUAR, 2005; MASTRANTONIO, 2005; MORENO et al., 2000; SHI et al., 1999; ALZAMORA et al., 1997; VIAL et al., 1991; HENG et al., 1990).

A DO em soluções de sacarose é capaz de prevenir perda de carotenóides durante a secagem, como constatado para abóboras, em que o pré-tratamento osmótico melhorou a retenção de carotenóides durante a secagem (NASCIMENTO, 2006). El-Aouar et al. (2005), avaliou o uso de xarope de milho e de sacarose como solutos para a desidratação osmótica de fatias de mamão, constatando que o primeiro foi menos eficaz na perda de massa e de água, quando comparado a soluções de sacarose. Fernandes et al. (2006) investigaram a DO de cubos de mamão em soluções de sacarose, otimizando o tempo total de processamento (DO seguida de secagem convectiva a 65 ^{o}C). O pré-tratamento osmótico reduziu significativamente o tempo total de processamento e, quando realizado em solução de concentração de 70 ^{o}Brix, a redução foi maior do que quando realizada em solução de 50 °Brix.

Heng et al. (1990) estudaram amplamente a influência da DO de mamão sobre a qualidade do produto final. Utilizaram soluções de sacarose (45 a 72 °Brix) em diferentes temperaturas (30 a 70 °C) com ou sem branqueamento prévio à DO e adição de cloreto de cálcio (0,01 a 0,2 M). O tratamento térmico foi intenso (10 min, 100 °C em água ou na

própria solução osmótica), provocando grande impregnação de sacarose no tecido. Uma concentração de 0,01 M do sal foi suficiente para aumentar a perda de água e melhorar a eficiência do processo de DO. O aumento da temperatura da solução osmótica promoveu maior perda de vitamina C, sendo que nas maiores concentrações a retenção foi maior. A perda de vitamina C após o branqueamento foi significativa, e as amostras branqueadas em água perderam praticamente todo o conteúdo da referida vitamina nos primeiros minutos de DO. Isso não ocorreu nas frutas branqueadas na solução desidratante. Maior conteúdo de carotenóides foi observado nas amostras em que menores temperaturas da solução osmótica foram empregadas.

A adição de ácidos à solução desidratante representa uma alternativa para diminuir o crescimento microbiano, devido à redução no pH do produto, e para aumentar as taxas de desidratação (ARGADOÑA et al., 2002). Rodrigues (1999) (*apud* ARGADOÑA et al., 2002) verificaram que o uso de lactato de cálcio e ácido láctico em soluções osmóticas de sacarose contribuíram por aumentar as taxas de desidratação de mamões, além de proporcionar melhores respostas de redução de atividade de água, de textura e de cor em comparação com os produtos tratados em soluções de sacarose com ácido cítrico e lactato de sódio.

A adição de ácidos cítrico e láctico a soluções de sacarose não influenciou a cromacidade de melões processados em comparação com os frescos. A claridade das amostras submetidas à DO, por outro lado, foi significativamente mais alta que a das frutas *in natura* em todos os processamentos. A deformação na ruptura das amostras processadas com ácido láctico resultou maior que a das frutas frescas, fornecendo produtos mais viscoelásticos (ARGADOÑA et al., 2002).

3.4. Branqueamento

O branqueamento é um pré-processamento aplicado a alimentos, que tem como objetivo principal a inativação de enzimas. O branqueamento térmico antes da secagem é utilizado para inativação de enzimas presentes nas frutas que podem oxidar compostos fenólicos ou vitaminas durante o processo, reduzindo o conteúdo nutricional do alimento, já que as temperaturas de secagem nem sempre são suficientemente altas para inativar enzimas oxidativas.

O efeito do branqueamento na secagem varia com o tipo de vegetal. Morangos tratados termicamente tiveram aumento significativo da difusividade da água durante a secagem, o que foi atribuído a modificação da estrutura celular, facilitando a transferência do

vapor de água (ALVAREZ et al., 1995). Doymaz (2011), trabalhando com berinjela, encontraram maiores coeficientes de difusão da água para as amostras branqueadas e secadas em comparação com as frescas. No entanto, fatias de manga branqueadas e secas apresentaram coeficiente de difusão de água semelhante ao da fruta sem branqueamento, o que foi associado à gelatinização do amido da manga (NIETO et al., 2001). Maçãs tiveram comportamento intermediário aos das amostras citadas, isto é, o coeficiente de difusão foi ligeiramente aumentado pelo branqueamento (NIETO et al., 1998).

Gliemmo et al. (2009) verificaram o efeito do branqueamento com vapor aquecido na cor de purê de abóbora durante o armazenamento por até 40 dias. Os autores constataram que tanto os valores de a* quanto os de b* diminuíram significativamente com o aumento do tempo de armazenamento, e que os valores de a* foram os mais afetados. Os valores de L* não foram afetados pelo armazenamento, o que está relacionado à não ocorrência de reações de escurecimento enzimático e oxidação e isomerização do β-caroteno.

Ndiaye et al. (2009) investigaram o efeito do tempo de branqueamento com vapor aquecido sobre a cor de fatias de manga. Os autores concluíram que a claridade das amostras branqueadas foi maior que a das frutas *in natura*, devido à alta atividade da enzima peroxidase nas frutas frescas. O aumento do tempo de branqueamento de 3 para 7 min, diminuiu os valores de a*.

O branqueamento aplicado previamente à desidratação osmótica de cubos de abóboras mostrou que o pré-tratamento não causou grandes danos ao tecido vegetal, já que a perda de água das amostras branqueadas e frescas submetidas às mesmas condições de DO foram similares. O tratamento térmico foi brando: 80 °C por 1 min, o que pode ter contribuído para estes resultados. Entretanto, a impregnação de açúcares nas amostras branqueadas durante a DO foi significativamente maior que nas frutas frescas (KOWALSKA et al., 2008).

Escobar et al. (2007) estudaram o efeito do branqueamento na DO de fatias de cenoura. Os autores verificaram que os danos celulares diminuíram a resistência à transferência de massa durante a DO, resultando em aumento das taxas de perda de água e de ganho de açúcares. Os coeficientes de difusão da sacarose e da água aumentaram devido ao branqueamento.

3.5. Coberturas comestíveis

Coberturas comestíveis podem ser definidas como finas camadas de material comestível aplicadas à superfície do alimento com o intuito de criar uma barreira seletiva ao

transporte de gases. Muitos componentes podem ser utilizados na formulação de coberturas comestíveis, sendo polissacarídeos, proteínas e lipídeos os mais usuais. Alguns componentes minoritários, tais como polióis (plasticizantes) e ácidos ou bases (reguladores de pH) também podem ser adicionados à formulação (VARGAS et al., 2008).

Segundo Vargas et al. (2008), coberturas de polissacarídeos apresentam boas propriedades de barreira a gases, contudo são altamente hidrofílicas e, em comparação com filmes plásticos comerciais, apresentam alta permeabilidade ao vapor de água, sendo, portanto, adequadas como componente básico para a formulação de coberturas comestíveis aplicadas como tratamento prévio à secagem.

Durante o processo de secagem, coberturas comestíveis a base de polissacarídeos ofereceram proteção contra a oxidação dos carotenóides de abóboras e de cenouras , as quais apresentaram maiores retenções destes pigmentos em comparação com as amostras frescas secas (LAGO, 2007; ZHAO; CHANG, 1995).

O processo de DO pode apresentar o inconveniente de incorporação excessiva de solutos, dependendo das condições em que é realizado. Com o objetivo de reduzir o ganho de solutos, a combinação de coberturas comestíveis com DO tem sido reportada em trabalhos como os de Matuska et al. (2006), Lenart e Piotrowist (2001), Azeredo e Jardine (2000), Lenart e Dabrowska (1999), Camirand et al. (1992) e Camirand et al. (1968).

Matuska et al. (2006) trabalhando com morangos cobertos com alginato, carragena e carragena e goma guar submetidos à DO em soluções de sacarose por 3 h, verificaram que o ganho de sólidos durante o processo foi maior para as amostras frescas do que para as cobertas. A perda de água das amostras cobertas foi menor que a das frutas *in natura*. Contudo, os efeitos das coberturas na eficiência da DO (razão entre a perda de água e o ganho de solutos) foram positivos, sendo que as amostras cobertas apresentaram maior eficiência que as não cobertas quando desidratadas nas mesmas condições.

Lenart e Dabrowska (1999) avaliaram os efeitos da aplicação de cobertura de pectina de baixa metoxilação parcialmente desidratada em fatias de maçã antes de serem submetidas à DO. Os autores observaram que as amostras cobertas apresentaram maior perda de água que as frutas frescas. No geral, as amostras cobertas tiveram menor ganho de sólidos que as não cobertas. Dentre as condições consideradas nos estudos, aquela que apresentou a maior perda de água e o menor ganho de solutos foi: cobertura de solução 2% de pectina e tempo de DO de 10 min.

No entanto, estudos conduzidos por Shigematsu et al. (2005) apresentaram resultados distintos dos observados por Matuska et al. (2006) e Lenart e Dabrowska (1999). Trabalhando

com carambolas, Shigematsu et al. (2005) avaliaram o efeito da aplicação de coberturas de diferentes polissacarídeos na DO em solução de sacarose. As autoras verificaram que as frutas *in natura* apresentaram maior perda de água e menor ganho de solutos do que as amostras cobertas, atribuindo o fato à integridade do tecido das frutas frescas. Assim, a permeabilidade seletiva da membrana plasmática estaria agindo como uma barreira mais eficaz do que as coberturas à entrada de sacarose nas células. Devido ao caráter hidrofílico das coberturas de polissacarídeos é possível que elas tenham sofrido solubilização parcial ou total na solução desidratante durante o processo de DO.

3.6. Isotermas de Sorção

A água é o mais importante componente dos alimentos, sendo o agente controlador de sua deterioração, por ser responsável pelas degradações físicas, químicas e microbiológicas dos produtos alimentícios. A atividade de água (a_w) é uma medida do conteúdo de água livre nos alimentos (PEDRO, 2005; SANJINEZ ARGADOÑA, 2005) e é definida pela razão entre a pressão de vapor exercida pela água no alimento e a pressão de vapor da água pura na mesma temperatura (HELDMAN; LUND, 1992). A atividade de água indica a relação de equilíbrio entre um alimento e a umidade relativa do ambiente que o cerca. Quando a pressão de vapor da água do alimento é igual à pressão de vapor da água no ambiente, a atividade de água é dada pela umidade relativa de equilíbrio (Equação 3).

$$a_w = \frac{P}{P_0} = \frac{UR}{100} \qquad (3)$$

em que P é a pressão de vapor da água no sistema; P_0 é a pressão da água líquida pura; e UR é a umidade relativa do ar.

Em função da atividade de água os alimentos podem ser classificados em:

- Alimentos de baixa a_w: apresentam atividade de água menor que 0,60, sendo microbiologicamente estáveis. Exemplos: mel, frutas secas, açúcar cristalizado;
- Alimentos de a_w intermediária: apresentam atividade de água entre 0,60 e 0,85 e são sujeitos a processos de deterioração provocados principalmente por bolores e leveduras.

- Alimentos de alta a_w: apresentam atividade de água maior que 0,85 e são alimentos considerados altamente perecíveis por permitirem o crescimento de uma ampla variedade de microrganismos.

As isotermas de sorção são curvas que exibem o conteúdo de umidade de equilíbrio do alimento *versus* a umidade relativa ou a atividade de água do ambiente. As curvas vinculam o conteúdo de água no alimento com a temperatura e umidade relativa do ar circundante (PEDRO, 2005). Para materiais alimentícios as isotermas podem ser de dessorção (eliminação de água) e de adsorção (adsorção de água). A diferença entre as curvas de dessorção e adsorção é conhecida como histerese (GONELI et al., 2010; PEDRO, 2005). A Figura 1 apresenta as isotermas de dessorção e adsorção da celulose microcristalina a 25 °C.

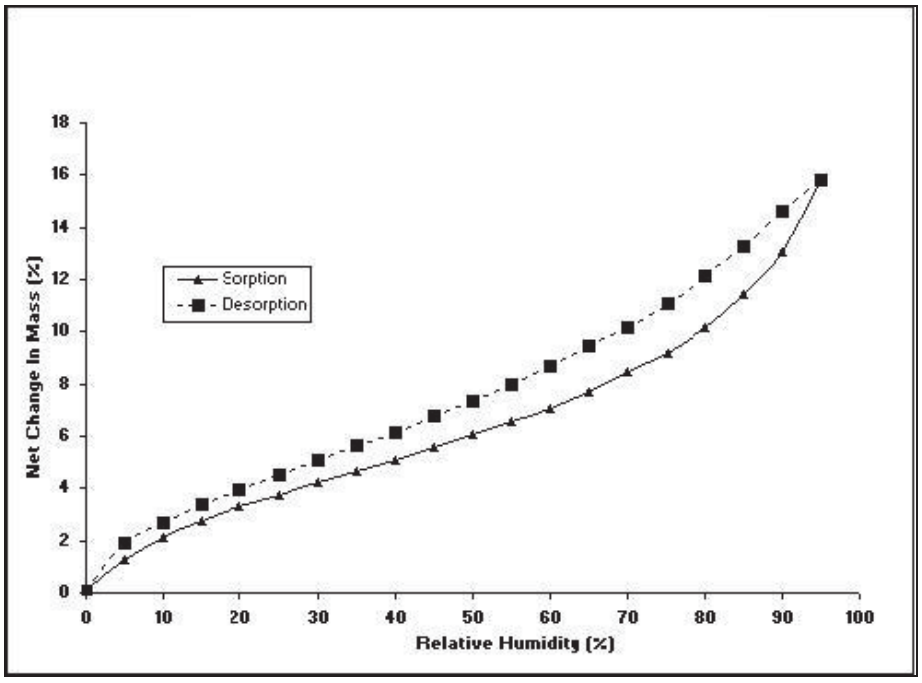

Figura 1. Isotermas de dessorção e adsorção da celulose microcristalina a 25 °C.

O conteúdo de água de equilíbrio pode ser determinado pelo método gravimétrico, assumindo que apenas a água deixa a amostra, e a porcentagem de umidade pode ser determinada a partir da secagem da amostra pré-pesada até ela atingir um peso constante. Qualquer que seja o material alimentício que entra em equilíbrio com a mistura gás-vapor ambiente, a atividade de água será determinada com base na atividade do vapor de água medido no ambiente. Caso a umidade relativa do ambiente seja conhecida e mantida

constante, a uma temperatura constante, para cada umidade relativa o alimento apresentará, no equilíbrio, um conteúdo de água distinto.

O método dos dessecadores, embora seja muito trabalhoso e demorado, precisa de um instrumental simples e, principalmente, apresenta uma boa reprodutibilidade quando adequadamente utilizado. Na dessorção, os cuidados devem se concentrar na deterioração das amostras por estarem em condições de alta umidade e porque a deterioração microbiológica altera as condições das determinações (TEIXEIRA NETO, 1987).

A termodinâmica aplicada a alimentos tem sido útil para a compreensão do comportamento e de aspectos estruturais da água na superfície e no interior de produtos desidratados (RIZVI & BENADO, 1984). A termodinâmica da sorção de água em alimentos desidratados é importante segmento na engenharia de alimentos, pois proporciona maior nível de interpretação sobre as isotermas, estima o calor necessário para certos processos, além de permitir predizer as condições ótimas de armazenamento que asseguram a máxima estabilidade dos produtos alimentícios (BERISTAIN *et al*., 1994). Parâmetros termodinâmicos como a entalpia e a entropia de sorção são necessários em conjunto para o desenvolvimento de projetos e para o entendimento qualitativo do estado da água na superfície do alimento (RIZVI & BENADO, 1984).

O estudo de processos de secagem e desenhos de secadores envolve o conhecimento dos gradientes de pressão de vapor de água na fase gasosa (VAN DEN BERG; BRUIN, 1981). O conhecimento dos dados de sorção de umidade, como uma função de duas ou mais temperaturas, é importante para a análise termodinâmica e modelagem da cinética de secagem (VEGA-GÁVEZ et al., 2007). O conhecimento das propriedades termodinâmicas também pode proporcionar informações quanto à microestrutura associada a um alimento, bem como permitir interpretações teóricas para o fenômeno físico que ocorre nas interfaces água-alimento (AL-MUHTASEB et al., 2002; RIZVI, 1995).

López-Malo et al. (1997) constataram que isotermas de adsorção e dessorção de mamão osmoticamente tratado apresentaram pouco efeito de histerese e que para o mamão branqueado não ocorreu histerese. Essa diferença de comportamento do material branqueado e do mamão tratado osmoticamente ocorreu devido à composição de sólidos, que é menor para as frutas branqueadas.

Existem muitos modelos matemáticos para descrever as isotermas de sorção de alimentos, porém nenhuma equação apresenta resultados precisos para todo o intervalo de atividade de água e tipos de alimentos, uma vez que a ligação da água com o alimento pode

ocorrer por diferentes mecanismos em diferentes regiões de atividade de água. Alguns modelos mais comumente usados são:

- BET: fornece uma estimativa do valor da monocamada de água na superfície dos alimentos. É útil na definição de um conteúdo de umidade ótimo para a secagem, da estabilidade de armazenamento e na estimativa da área de superfície de um alimento (AL-MUHTASEB et al., 2002).

$$X = \frac{X_m.C.a_w}{(1-a_w)(1+(C-1)a_w)} \tag{4}$$

em que X é o conteúdo de umidade de equilíbrio, em base seca; a_w representa a atividade de água; X_m é o conteúdo de umidade da monocamada, em base seca; C é uma constante relacionada ao calor isostérico de sorção.

- GAB: é um modelo de sorção multimolecular semi teórico. É um modelo versátil que se adapta bem a amplas faixas de atividade de água (AL-MUHTASEB et al., 2002), definido como:

$$X = \frac{X_m.C.K.a_w}{(1-K.a_w)(1-K.a_w+C.K.a_w)} \tag{5}$$

em que X é o conteúdo de umidade de equilíbrio, em base seca; a_w representa a atividade de água; X_m é o conteúdo de umidade da monocamada, em base seca; C e K são constantes relacionadas a energia de ligação entre a primeira e as próximas camadas de água em um local de sorção.

- Halsey: a equação de Halsey fornece uma expressão para a condensação das multi-camadas a uma distância relativa da superfície (AL-MUHTASEB et al., 2002):

$$X = \left[\frac{-A}{\ln(a_w)}\right]^{\frac{1}{B}} \tag{6}$$

em que X é o conteúdo de umidade de equilíbrio, em base seca; a_w representa a atividade de água; A e B são constantes.

- Henderson: é um dos modelos mais utilizados relacionando a atividade de água à quantidade de água adsorvida, porém não fornece informação precisa sobre o estado físico da água (AL-MUHTASEB et al., 2002).

$$X = \left(\frac{\ln\left(1 - a_w\right)}{-A} \right)^{\frac{1}{B}} \quad (7)$$

em que X é o conteúdo de umidade de equilíbrio, em base seca; a_w representa a atividade de água; A e B são constantes.

- Oswin: é um modelo empírico de uma série de expansão para curvas com formato sigmoidal (AL-MUHTASEB et al., 2002).

$$X = A \left[\frac{a_w}{(1 - a_w)} \right]^B \quad (8)$$

em que X é o conteúdo de umidade de equilíbrio, em base seca; a_w representa a atividade de água; A e B são constantes.

- Peleg: é também um modelo empírico, que geralmente apresenta um bom ajuste aos dados experimentais de sorção.

$$X = A\left(a_w\right)^B + C\left(a_w\right)^D \quad (9)$$

em que X é o conteúdo de umidade de equilíbrio, em base seca; a_w representa a atividade de água; A, B, C e D são constantes.

3.7. Cor

A cor é um fator importante para a aceitação dos produtos pelos consumidores, pois é diretamente relacionada a frescor e sabor. Ela é definida como a impressão variável que a luz refletida pelos corpos produz no órgão da vista.

No âmbito da física ótica, a cor é um feixe de radiações luminosas com uma determinada distribuição espectral. Os materiais transferem a luz que recebem de forma que a luz transmitida tem diferente distribuição espectral. A capacidade de um material de alterar a distribuição espectral da luz depende da sua composição química e da sua estrutura (CALVO; DURAN, 1997 *apud* SANGINEZ-ARGADOÑA, 2005).

Há três requisitos básicos para a percepção da cor: a fonte de luz, o objeto (que refletirá a luz) e o observador (que processará a luz refletida enxergando um objeto colorido). Como a luz é uma onda eletromagnética, ela possui a propriedade de se propagar no vácuo com velocidade constante (c) igual a 299792458 $m \cdot s^{-1}$. Em decorrência disto, sabendo-se a

freqüência da onda eletromagnética (*f*), é possível determinar seu comprimento de onda (λ): $\lambda = c \cdot f^{-1}$.

A Figura 2 apresenta o espectro das ondas eletromagnéticas.

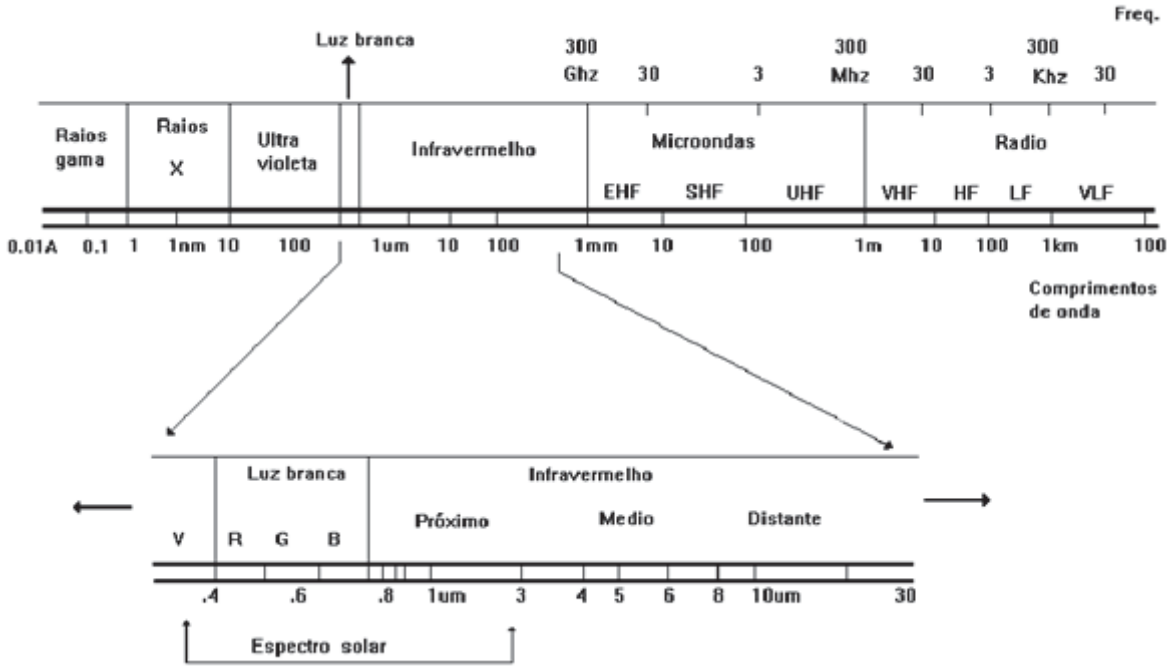

Figura 2. Espectro de ondas eletromagnéticas.

O espectro de luz visível ou branca pode, portanto, assumir diversas cores, desde o violeta até o vermelho. Sabe-se que a luz branca (luz solar) ao passar através de um prisma decompõe-se nos feixes de luzes coloridas. Logo, a luz branca representa a “soma” de todas as cores; em contrapartida, o preto representa a ausência de luz.

A percepção da cor envolve impulsos nervosos captados por sensores presentes na retina dos olhos humanos, que são levados através do nervo ótico até o cérebro, onde ocorrerá a percepção da imagem. Assim, o fenômeno envolve física, fisiologia e psicologia e, portanto, sua medida está relacionada à percepção humana (AURAND et al., 1987 *apud* OLIVEIRA, 2007).

Os sensores presentes na retina são chamados cones e bastonetes. Existem cerca de 100 milhões de bastonetes na retina, responsáveis pela percepção da luminosidade. Os seis milhões de cones são os responsáveis pela “visualização” da cor. Existem três tipos de cones: os vermelhos, os verdes e os azuis, formando o chamado sistema tricromático de medição das cores. Logo, apenas três cores da informação espectral são recebidas, as demais são perdidas. O olho humano não é capaz de diferenciar cada componente, mas sim a cor resultante, de

maneira que os intervalos de sobreposição das cores recebidas pelos cones são principalmente os enxergados.

Técnicas instrumentais utilizando espectrofotômetros ou colorímetros têm sido aplicadas para obter uma avaliação objetiva da cor através dos sistemas de cores (Munsell, Hunter, CIE, CIELAB). Os sistemas se baseiam numa mistura de cores a partir dos três estímulos fundamentais (vermelho, verde e azul), com relação à percepção humana dos atributos sensoriais de tonalidade, luminosidade e saturação analisados sob o espaço cromático em coordenadas retangulares (CALVO; DURAN, 1997 *apud* SANGINEZ-ARGADOÑA, 2005). Entre estes sistemas, o sistema CIELAB, é atualmente o mais aplicado porque além de definir o espaço cromático em coordenadas retangulares (L*, a*, b*), o define também em coordenadas cilíndricas (L*, h*, C*). A luminosidade é representada por L* e varia de 0 (claro) a 100 (escuro); a intensidade da cor é representada pelos parâmetros de croma a* e b*, onde a* varia do vermelho (valor positivo) ao verde (valor negativo) e b*, do amarelo (valor positivo) ao azul (valor negativo) (CALVO; DURAN, 1997 *apud* SANGINEZ-ARGADOÑA, 2005).

Não existe uma recomendação geral quanto ao procedimento de mensuração da cor, pois os instrumentos de medida (colorímetros e espectrofotômetros) podem ter características distintas quanto ao diâmetro de abertura (10 – 22 mm), tipo de iluminante (fonte C, que simula a luz média do dia, ou D65, que simula a luz do entardecer) e ângulo de observação (2° e 10°, por exemplo), produzindo resultados semelhantes, mas não iguais. A Comissão Internacional de Iluminação em documento datado de 1986 recomenda o uso da fonte D65 e angulo de 10°.

Durante a DO de frutas e vegetais, geralmente, verifica-se aumento nos parâmetros de croma das amostras, como resultado da concentração dos pigmentos coloridos. Rodrigues et al. (2003) desidrataram osmoticamente mamões Formosa em soluções de sacarose adicionadas de diferentes ácidos e sais. Os pesquisadores verificaram aumento na claridade e nos parâmetros de croma das frutas desidratadas em solução contendo cálcio. Falade et al. (2007) também verificaram aumento de L*, a* e b* de melões submetidos a tratamentos osmóticos em solução de sacarose.

O branqueamento, por outro lado, ocasiona escurecimento das amostras, como verificado em purê de tomate por Patras et al. (2009) e para couve de Bruxelas por Viña et al. (2007) e Oliveira et al. (2008).

3.8. Microscopia

O conhecimento da microestrutura de alimentos é reconhecido atualmente como um pré-requisito para entender suas propriedades. O processamento de um alimento pode ocasionar mudanças em sua estrutura, afetando suas propriedades. Assim, as técnicas de análise de microestrutura são necessárias para a compreensão das relações entre estrutura celular e propriedades do alimento (ANTONIO, 2002).

Segundo Galleti (2011) cada tipo de microscópio e técnica de preparação de material oferece vantagens específicas na demonstração dos elementos morfológicos.

Os microscópios pertencem, basicamente, a duas categorias: luminoso e eletrônico. Na microscopia de luz utiliza-se radiação de ondas luminosas, que é refratada através de lentes de vidro. A área observada aparece brilhantemente iluminada e os objetos estudados, mais escuros (GALLETI, 2011).

Na microscopia eletrônica emprega-se a radiação de feixe de elétrons, o qual é refratado por meio de lentes eletrônicas. O aumento produzido é de 200 a 400 vezes maior que o do microscópio de luz. Basicamente, os microscópios eletrônicos são de dois tipos: de transmissão e de varredura (GALLETI, 2011; ANTONIO, 2002).

O Microscópio Eletrônico de Transmissão (MET) é bastante difundido no estudo de materiais biológicos, pois permite a definição de imagens intracelulares, possibilitando estudos de morfologia celular, aspectos gerais das organelas e também da interação de parasitas com as células (GALLETI, 2011).

O Microscópio Eletrônico de Varredura (MEV) destina-se basicamente ao exame da superfície de amostras, sendo que as superfícies internas podem ser visualizadas desde que sejam fraturadas e expostas, utilizando, por exemplo, seu congelamento em nitrogênio líquido (ANTONIO, 2002). O MEV é indispensável em muitos tipos de pesquisa biológica, contribuindo para a classificação e taxonomia de insetos e fungos, estudo da morfologia de pólens e em pesquisas de superfícies de diversas estruturas de plantas e animais (GALLETI, 2011).

Estão disponíveis na literatura diversos trabalhos relacionando as alterações causadas ao tecido celular de frutas e hortaliças aos processamentos aos quais essas amostras foram submetidas (CASTRO-GIRÁLDEZ et al., 2011; GÓMEZ et al., 2011; FERNANDES et al., 2009; PEREIRA et al., 2009; FERNANDES et al., 2008; GÓNZALEZ-FÉSLER et al., 2008;

NUNES et al., 2008; KHIN et al., 2007; DELGADO; RUBIOLO, 2005; NIETO et al., 2004; MORENO et al., 2000; NIETO et al., 1998).

Castro-Giráldez et al. (2011) correlacionaram as variações no volume de fatias de kiwi durante a DO com as imagens obtidas através de Crio-MEV. Os autores verificaram a existência de duas zonas distintas na estrutura da fruta, as quais, dependendo do tempo de desidratação osmótica, sofriam contração e expansão em momentos distintos.

Utilizando Microscopia de Luz, Gónzalez-Fésler et al. (2008) avaliaram o efeito do branqueamento em vapor e da DO em soluções aquosas de glicose adicionadas de sais de cálcio na estrutura celular de fatias de maçã. Os autores observaram que o branqueamento ocasionou a ruptura das membranas celulares e danificou a parede celular das células da fruta. As células de maçã impregnadas com cálcio apresentaram arranjo celular semelhante ao das frutas frescas, uma vez que o íon liga-se a compostos pécticos encontrados na lamela média e parede celular da fruta, reforçando sua estrutura.

3.9. Estrutura celular

Grandes porções dos vegetais são constituídas por células de parênquima, as quais são encontradas nas partes comestíveis dos frutos, tipos especiais de caules que armazenam material de reserva, como bulbos e tubérculos, além da porção do córtex da maioria das raízes. O termo parênquima refere-se a tecidos com pouca especialização e que podem estar relacionados a diversas funções fisiológicas das plantas. Células parenquimáticas conservam a habilidade de se dividirem ainda quando adultas (FAHN, 1987 *apud* MAURO, 1998).

Muitas células parenquimáticas são poliédricas e seus diâmetros tendem a ser mais ou menos iguais, embora outras formas também sejam comuns (alongadas, lobulares). Quando maduro, o tecido parenquimático pode estar compactado e sem espaços intercelulares, ou pode ter uma arquitetura que abriga espaços intercelulares bem definidos, como ocorre nas folhas (FAHN, 1987 *apud* MAURO, 1998).

A Figura 3 apresenta uma representação sumária das células vegetais parenquimáticas de plantas superiores, destacando a parede celular, o plasmalema, o citoplasma, o tonoplasto e o vacúolo.

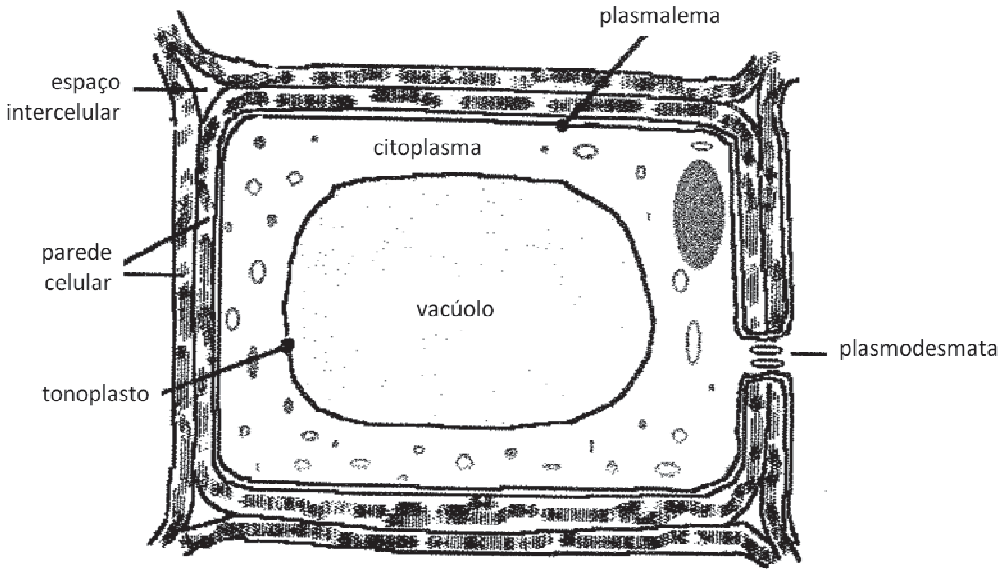

Figura 3. Representação esquemática de uma célula vegetal de uma planta superior.

A parede celular contém numerosos interstícios relativamente grandes, sendo permeável a água e a pequenos solutos (NOBEL, 1991 *apud* MAURO, 1998). A espessura das paredes celulares varia entre 0,1-10 μm, sendo que a celulose compõe de 25-50% de seu material orgânico. A celulose está organizada estruturalmente com outros polissacarídeos não celulósicos, como a pectina e a lignina, além de quantidade pequena de proteínas e considerável de cálcio e outros cátions e, algumas vezes, de silicatos (NOBEL, 1991 apud MAURO, 1998).

Na pectina, os grupos carboxil encontram-se geralmente dissociados, restringindo a mobilidade de cátions (BAKER; HALL, 1988 *apud* MAURO, 1998). Os íons cálcio são capazes de se ligar à pectina presente na parede celular e lamela média, melhorando sua integridade estrutural (PEREIRA et al., 2009; PANI et al., 2008; MARTÍN-DIANA et al., 2007; ANINO et al., 2006; MASTRANTONIO et al., 2005).

No caso particular dos vegetais, plasmalema é a denominação dada à membrana plasmática que envolve o protoplasto, ou seja, todo o conteúdo da célula, excetuando-se a parede celular (KOTYK et al., 1988 *apud* MAURO, 1998).

A etapa que limita a velocidade de movimentação de muitas moléculas para dentro e para fora das células vegetais é a difusão através do plasmalema, devido ao forte empacotamento e às interações entre as moléculas que compõem a membrana (NOBEL, 1991 *apud* MAURO, 1998). Segundo Bidwell (1979 *apud* MAURO, 1998), as membranas celulares geralmente apresentam permeabilidade diferencial, ou seja, permitem que solventes atrevessem-nas mais facilmente restringindo a passagem dos solutos, ainda que estes penetrem muito lentamente.

O citoplasma é delimitado pelo plasmalema e pelo tonoplasto (membrana citoplasmática que retém o conteúdo vacuolar) (Figura 3), sendo constituído por uma solução, denominada citosol, e contendo organelas como cloroplasto, mitocôndrias, peroxissomos, ribossomos, proteínas e outras macromoléculas, além de estruturas que influenciam as propriedades termodinâmicas da água (NOBEL, 1991 *apud* MAURO, 1998).

Existem canais de ligação ligando as células entre si, de citoplasma a citoplasma, denominados plasmodesmata. Esses canais contêm um desmoduto, que é uma estrutura membranosa contíguas com os retículos endoplasmáticos de duas células (NOBEL, 1991 *apud* MAURO, 1998).

Nas células maduras de plantas superiores e de muitas plantas menos avançadas existe uma grande fase aquosa central, que pode atingir até 90% do volume da célula, chamado vacúolo, o qual é delimitado pelo tonoplasto (NOBEL, 1991 *apud* MAURO, 1998). A solução aquosa contém principalmente íons inorgânicos ou ácidos orgânicos como solutos, porém quantidades consideráveis de açúcares e aminoácidos podem estar presentes em algumas espécies.

Nobel (1991 *apud* MAURO, 1998) afirma que, da mesma forma que o plasmalema, o tonoplasto deve apresentar permeabilidade diferencial, restringindo a passagem de solutos, especialmente os de alto peso molecular. No vacúolo, o abaixamento da atividade de água ocorreria praticamente pela presença de solutos, já que o mesmo contém poucos colóides ou outras interfaces. A atividade de água do citosol, por sua vez, é abaixada tanto pelos solutos quanto pela presença de colóides ou outras interfaces. Assim, concluí-se que o vacúolo tem maior concentração de solutos osmoticamente ativos do que o citosol.

Toupin et al. (1989 *apud* MAURO, 1998) afirmam que a deformação de uma célula vegetal durante o processo de desidratação osmótica pode ser descrita de acordo com três etapas diferentes. Inicialmente, a célula encontra-se em condição de turgor total, apresentando o máximo volume que a mesma pode atingir, o que é alcançado quando a célula entra em equilíbrio com a água pura. Neste estado o protoplasto exerce pressão sobre a parede celular,

que por sua vez oferece resistência ao mesmo. Consequentemente o plasmalema encontra-se firmemente colado à parede. Na primeira etapa de DO, a célula passa do turgor total à plasmólise incipiente. Ocorre fluxo de água apenas de dentro para fora e devido à redução do volume celular, o protoplasto deixa de exercer pressão sobre a parede. A célula já não apresenta pressão interna, mas o plasmalema ainda permanece próximo à parede celular. Na segunda etapa, a célula vai da plasmólise incipiente até o volume crítico, ponto onde a estrutura celular começa a entrar em colapso. Neste estágio permanece o fluxo de água de dentro para fora da célula e inicia um fluxo de soluto no sentido contrário, que, dependendo do seu peso molecular, não atravessa o plasmalema. O volume da célula não sofre variação, mas o protoplasto se reduz devido à perda de água. Portanto, o plasmalema começa a se distanciar da parede celular, gerando um espaço livre a ser preenchido pelo soluto. Na terceira etapa, a célula vai do volume crítico ao volume de equilíbrio. O volume celular sofre significativa diminuição, causando o colapso de toda a estrutura. A célula continua perdendo água e ganhando soluto, até entrar em equilíbrio com a solução osmótica. Mauro et al. (2003) demonstraram que a utilização de microscopia ótica com corantes vitais permite a visualização parcial destas etapas, pois causa o mínimo de impacto nas células constituintes do tecido vegetal.

CAPÍTULO I

COMBINED METHODS TO OBTAIN DRIED PAPAYA OF FORMOSA CULTIVAR (*Carica papaya* L.)

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Abstract

The aim of this work was to evaluate the influence of blanching in hot water, osmotic dehydration in sucrose solutions and pectin coating on color and water activity of papaya slices after convective drying. The study was divided into two stages. In the first experiments, the influence of concentration and time of dehydration on color changes and water activity of fresh and blanched papaya slices was evaluated based on a factorial design. It was not verified significant color changes at the studied ranges. An expression for the water activity of osmotically dehydrated blanched samples was found. Then, the influences of blanching, osmotic dehydration, edible coating and combinations among these pre-treatments on color and water activity were evaluated after convective drying of papaya slices. Blanching was not an advantageous pre-treatment to maintain color and to reduce water activity of dried fruits. Osmotically dehydrated fresh samples with or without coating presented the best results.

Keywords: Osmotic dehydration, blanching, edible films, drying, papaya, color, water activity

1. Introduction

Papaya is a fruit from the south of Mexico, typical of tropical regions. When ripe, papaya pulp presents color ranging from yellow to red-orange and a sweet and soft taste. The fruit is nutritive, presenting high A, C and B-complex vitamin contents, besides mineral salts such as iron, calcium and phosphor. The major carotenoids of papaya pulp are: lycopen, β-cryptoxanthin and β-carotene, typically red-orange pigments (KIMURA et al., 1991).

Due to perishability, the processing of papaya is an alternative to reduce the high losses during fruit harvest and post-harvest, adding value to the final product.

Osmotic dehydration (OD) is a technique that involves the immersion of the product in hypertonic aqueous solutions resulting in decrease of water content with simultaneous increase of solids, apart from a change in the chemical composition of the dehydrated food (EREN; KAYMAK-ERTEKIN, 2007; FERNANDES et al., 2006; SERENO et al., 2001). During OD it is possible to modify the food formulation by incorporating solutes in the porous structure of fruits and vegetables (TORREGIANI; BERTOLO, 2001; FITO et al., 2001). Calcium addition in osmotic solution has received great attention due to the desirable effects maintaining the tissue firmness (HEREDIA et al., 2007; RODRIGUES et al. 2003).

Blanching is widely used at minimally processed food preparation aiming enzymatic inactivation, microorganisms destruction/injury to reduce initial microbial of the food or awareness of the survivor microorganisms to posterior procedures (ALZAMORA et al., 1997). The great inconvenient of thermal treatment procedure is the tissue damage of the vegetal resulting in its softening (DOYMAZ, 2008; KOWALSKA et al., 2008; ESCOBAR et al., 2007; ALZAMORA et al., 1997). When applied prior to drying or another treatment, as OD, several authors verified higher dehydration rates and/or shorter time of process for the same final moisture (DOYMAZ, 2008; KOWALSKA et al., 2008; ESCOBAR et al., 2007).

Edible coatings are thin layers of edible material applied to the surface of the food to create a selective barrier to the gases transport (VARGAS et al., 2008). Polysaccharides coatings generally present effective properties to oxygen barrier, particularly under low moisture content conditions (CUQ et al., 1995), being therefore suitable as basic compounds for the formulation of edible coatings applied before drying.

Drying is a complex thermal process in which unsteady heat and moisture transfer occur simultaneously. The aim of drying is water removal from the food until levels that minimize or inhibit microbial growth and deterioration reactions (DOYMAZ, 2008; DOYMAZ, 2007). Drying effects at sensorial and nutritional characteristics of the final product and the use of pre-treatments as an alternative to improve these qualities has been studied by many authors (Leeratanarak et al., 2006; ZHAO & CHANG, 1995).

The purpose of the present work was to evaluate the influence of pre-treatments on color and water activity of papaya slices after convective drying. The experiments were splited into two sets: in the first one, the influence of the solution concentration and osmotic dehydration time on color and water activity of fresh and blanched papaya slices of Formosa cultivar immersed in sucrose solutions added with calcium lactate and lactic acid were determined. In the second set, the influence of pre-treatments (blanching, edible coating,

osmotic dehydration and combinations among them) on color changes and water activity reduction after convective drying was studied.

2. Materials and methods

2.1. *Samples preparing*

Papaya of the Formosa cultivar from São Paulo State (Brazil) were purchased at São José do Rio Preto Supplying Center (CEAGESP – São José do Rio Preto, São Paulo, Brazil). Ripe fruits with orange peels and approximately 8.89% ± 0.73 of reducing sugars were used in the experiments.

Papayas were longitudinally cut in four pieces, the seeds were removed and each portion was sectioned with a manual cutter in cylinders with diameter of 3.6 cm. These pieces were cut in slices with 1.0 cm of thickness. The samples were kept and mixed in a plastic bag from where the slices to be used in the experiments were randomly removed.

2.2. *Blanching*

Papaya slices were put in a stainless basket and dipped in boiling water (98.3 °C at São José do Rio Preto-SP-Brazil) for 2 min. After this time, the samples were cooled in tap water for 1 min to be used in the further experiments.

2.3. *Osmotic dehydration experiments*

Osmotic solutions were prepared with commercial sucrose (refined sugar) purchased in a local market; food grade pentahydrate calcium lactate in powder from PURAC Synthesis – Brazil; food grade L (+)-lactic acid in 85% solution from PURAC Synthesis – Brazil and distilled water.

Fresh and blanched papaya slices were dehydrated in sucrose solutions at 40, 50 and 60% w/w added with 1% w/w of calcium lactate and 0.1 M of lactic acid. The times of dehydration were 60, 120 and 180 min for the fresh samples and 30, 105 and 180 min for the blanched ones. The time range of blanched experiments was extended to find significant differences among the answers.

OD experiments were conducted into a stainless steel vat with an external jacket for water circulation at 27 °C. Osmotic solutions were continuously stirred using a 1.6 kW mechanical stirrer (Marconi, model MA-261 - Brazil) with a 10 cm diameter propeller and rotation at 1000 rpm. The ratio between sample and solution masses were at least 1:10. After

processing time, samples were removed from the solutions, washed in distilled water for 10 s and dried on absorbed paper.

Fresh and blanched samples were pre-dehydrated before drying using the conditions set out in the first set of experiments. So, fresh samples were osmotically dehydrated in 50% w/w sucrose solution added with 1% w/w of calcium lactate and 0.1 M of lactic acid for 120 min and blanched slices were dehydrated in 50% w/w sucrose solution added with 1% w/w of calcium lactate and 0.1 M of lactic acid for 105 min.

2.4. *Edible coating application*

Low methoxylated amidated pectin GRINDSTED® LA 210 (degree of methoxilation of 0.34; degree of amidation of 0.17) from Danisco – Brazil was used to coat fresh papaya slices. A 2% w/w pectin solution was prepared at 70 °C and applied on samples surface at 40 °C by immersion during 1 min. Gelling was activated with subsequent placement of samples in a 2.8% w/w aqueous solution of food grade pentahydrate calcium lactate in powder from PURAC Synthesis – Brazil for 30 s. To remove the excess of coating, samples were washed in distilled water for 30 s.

2.5. *Drying*

Fresh and blanched, osmotically dehydrated or not, coated or not, were dried in a convective drier with air velocity of 1.0 $m.s^{-1}$ at 70 °C for 240 min. The rotation of the trays inside the drier was conducted at each 30 min.

Two batches of samples were processed: in the first one, fresh and blanched fruits, osmotically dehydrated or not (F, B, F-OD and B-OD), in duplicate, were dried in parallel; in the second batch, the samples were coated with pectin before drying (F-CO, B-CO, F-OD-CO and B-OD-CO), in duplicate.

2.6. *Analytical methodologies*

Water activity and color of the samples were evaluated before and after each process.

Water activity was taken in triplicate at 25 °C in a hygrometer AW SPRINT (NOVASINA, Switzerland). The first set of experiments was conducted in the first semester of 2007. At that time, Colorflex did not have been purchased, so color was measured in six replicates at 25 °C using an images captor with digital camera installed in an appropriated light compartment and academic software (V-01 Lens Eye) that analyses color (LUZURIAGA et al., 1997). The used standards were L* = 71.941, a* = 36.067 and b* =

67.857. The second set of assays was performed in the first semester of 2008, then color was evaluated in four replicates using a spectrophotometer Colorflex (HunterLab, USA) and version 4.10 of the Universal software with the settings: illuminant D65, observer at 10° and reading of the absolute values of L*, a * e b*.

2.7. *Experimental design and modeling*

A 2^2 factorial design with 3 center points was used to design OD assays and to estimate the main effects of process variables (osmotic solution concentration and process time) on water activity and color of fresh and blanched papaya slices (dependent variables or responses). Each realized experiment was performed with a different fresh sample, so the presented responses represent a ratio between the value for the processed sample and their respective fresh samples.

The regression coefficients were used to derive a mathematical model, allowing the evaluation of a linear model (Equation 1). The analysis of variance enabled to identify the coded variables that had significant effects on the responses of interest at the 95% confidence level ($p < 0.05$) (BARROS NETO et al., 2002).

$$Y=\beta_{k0}+\sum_{i=1}^{2}\beta_{ki}x_i+\sum_{i=1}^{1}\sum_{j=i+1}^{2}\beta_{kij}x_i x_j \quad (1)$$

where β_{k0}, β_{ki} e β_{kij} are the coefficients (constants), x are the factors or independent variables and Y are the responses or dependent variables.

The coded values for using in the above equation can be obtained from the uncoded values using the following expression:

$$x_1=(C-50)/10 \quad (2)$$

$$x_2^F=(t-120)/60 \quad (3)$$

$$x_2^B=(t-105)/75 \quad (4)$$

where C is the osmotic solution concentration (% w/w) and t is the process time (min). Superscript F indicates experiments with fresh fruits and B with blanched ones.

All statistical analyses were performed using Statistica 7.0 (StatSoft Inc. South America, Tulsa, OK, USA) and the experiments were performed randomly.

3. Results and discussions

3.1. *Evaluation of the osmotic dehydration conditions of the fresh samples*

Table 1 presents the factorial design, the coded variables and obtained responses for the fresh samples.

Table 1. Experimental design and obtained responses for the OD optimization of fresh papaya.

	Uncoded variables		Coded variables		Responses			
Exp.	Sucrose conc. (% w/w)	Time of OD (min)	x_1	x_2^B	$\frac{aw_{F-OD}}{aw_F}$	$\frac{L^*_{F-OD}}{L^*_F}$	$\frac{a^*_{F-OD}}{a^*_F}$	$\frac{b^*_{F-OD}}{b^*_F}$
1	40	60	-1	-1	0.987	0.90	1.01	0.91
2	60	60	+1	-1	0.988	1.00	0.99	0.98
3	40	180	-1	+1	0.981	1.04	1.01	1.05
4	60	180	+1	+1	0.977	1.00	1.01	1.00
5	50	120	0	0	0.989	0.86	1.01	0.84
6	50	120	0	0	0.990	1.00	0.98	0.88
7	50	120	0	0	0.983	1.03	0.96	1.02

The average water activity, L*, a* and b* parameters of the fresh samples were 0.990 ± 0.000, 36.57 ± 4.98, 38.15 ± 0.72 and 31.80 ± 3.65, respectively.

Fresh samples osmotically dehydrated in 60% w/w osmotic solution for 3 hours presented the minor water activity: 0.968 and the minor ratio between processed and fresh fruits (Table 1).

The analysis of variance of aw_{F-OD}/aw_F, L^*_{F-OD}/L^*_F, a^*_{F-OD}/a^*_F and b^*_{F-OD}/b^*_F ratio showed that any term of the linear model was statistically significant in a confidence level of 95%. The coded models that would represent the behavior of these responses in the osmotic dehydration treatment did not show a good fit, with determination coefficients of 0.5557, 0.3906, 0.1368 and 0.3007, respectively.

It was observed that L*, a* and b* values of the most of the dehydrated fresh fruits were close to the nature samples since the ratios (Table 1) had values of approximately 1.00.

Parameters a* and b* indicate the color intensity, where a* values vary from red (positive value) to blue (negative value) and b* values from yellow (positive value) to green

(negative value). Since carotenoids content is related to samples color, the results suggested having considerable carotenoids retention in papaya during OD, probably because samples were submerged in osmotic solutions and processes temperature were suitable (27 °C) (TONON et al., 2007; SHI et al., 1999; HENG et al., 1990).

3.2. *Evaluation of the osmotic dehydration conditions of the blanched samples*

Table 2 presents the factorial design, the coded variables and obtained responses for the blanched papayas.

Table 2. Experimental design and obtained responses for the OD optimization of blanched papaya.

	Uncoded variables		Coded variables		Responses			
Exp.	Sucrose conc. (% w/w)	Time of OD (min)	x_1	x_2^B	$\frac{aw_{B-OD}}{aw_F}$	$\frac{L^*_{B-OD}}{L^*_F}$	$\frac{a^*_{B-OD}}{a^*_F}$	$\frac{b^*_{B-OD}}{b^*_F}$
1	40	30	-1	-1	0.995	0.95	1.22	1.04
2	60	30	+1	-1	0.994	1.03	1.18	1.07
3	40	180	-1	+1	0.986	1.03	1.21	1.11
4	60	180	+1	+1	0.971	0.96	1.32	1.01
5	50	105	0	0	0.988	1.09	1.28	1.2
6	50	105	0	0	0.988	0.94	1.31	1.00
7	50	105	0	0	0.987	0.99	1.00	1.08

In these tests, the average water activity, L*, a* and b* parameters of the fresh papayas were 0.989 ± 0.001, 41.61 ± 1.56, 36.52 ± 2.02 and 39.61 ± 1.86, respectively. The values of water activity and yellowness (b* values) were similar to that found in the fresh fruits experiments. The average water activity of the blanched samples (0.990 ± 0.000) did not significantly differ from the values of the fresh slices (0.989 ± 0.001) in a 95% confidence level (Table 2).

The water activities of the dehydrated blanched and the osmotically dehydrated fresh samples processed in the same conditions resulted very closer (Tables 1 and 2).

All the terms of the linear model of the $aw_{B\text{-}OD}/aw_F$ ratio were significant in a 95% confidence level. Equation 5 represents the model.

$$Y = 0.987 - 0.004x_1 - 0.008x_2^B - 0.003x_1x_2^B \quad (5)$$

where Y is the $aw_{B\text{-}OD}/aw_F$ ratio; x_1 is the concentration of the osmotic solution (% w/w); x_2^B, is the process time (min); and $x_1x_2^B$ is the term of interaction between the two independent variables.

Equation 5 showed a good fit to the experimental data, with a determination coefficient of 0.9945. Both variables had a negative effect on the response, as expected, which meant that the smaller water activity was correlated with the higher solution concentration and osmotic dehydration time. Time of the process affected more negatively the $aw_{B\text{-}OD}/aw_F$ ratio than the concentration.

In papayas of Solo cultivar Chan and Kwok (1975) verified that the invertase activity was 0.821 U.mg^{-1} of protein. After manipulation, which involved removing of skin and seeds, cutting and crushing, half of the sucrose presented in the fruits was converted in reducing sugars in 2.66 min if the enzyme was not inactivated (CHAN; KWOK, 1975). In this work, it is possible that invertase activity created fluxes that became unstable the system. Thereby sucrose transferred from the solution to papaya had been enzymatically inverted to reducing sugars which subsequently left the fruit. On the other hand, blanched papayas had a more stable sugar composition during OD contributing to fit an equation to water activity experimental data.

The coded model that would describe the behavior of L^*_{B-OD}/L^*_F, a^*_{B-OD}/a^*_F and b^*_{B-OD}/b^*_F ratios in the osmotic dehydration treatments did not show a good fit, with a determination coefficient of 0.3141, 0.1541 and 0.2195, respectively.

Blanched fruits submitted to OD presented more pronounced color than fresh papayas, being the a* vales the most affected by osmotic treatment (Table 2). Processed samples showed red intensification probably as a result of the pigments concentration due to fruits dehydration (OSORIO et al., 2007; MASTRANTONIO et al., 2005; MORENO et al., 2000).

Generally OD results in higher L* values for the processed fruits when compared to the fresh samples which can be attributed to sugar uptakes and concentration effects (OSORIO et al., 2007; MASTRANTONIO, 2005; ARGADOÑA et. al, 2002; MORENO et al., 2000).

As observed for the osmo-dehydrated fresh fruits, the results suggested that there was carotenoids retention in osmo-treated blanched papaya.

3.3. *Effects of pre-treatments on convective drying of papayas*

Drying experiments were conducted in two batches. In the first batch, fresh (F) and blanched (B) samples, osmotically dehydrated (F-OD and B-OD) or not were dried. In the second batch, these samples were coated with low methoxylated amidated pectin before drying (F-CO, B-CO, F-OD-CO and B-OD-CO). The conditions of the central points were selected to the further OD experiments to avoid excessive solute impregnation during pre-treatment and to achieve reasonable decrease of water activity.

Tables 3 and 4 present the moisture content and water activity of the samples dried in the first and second batches of experiments, respectively.

Table 3. Moisture content and water activity of fresh and blanched samples, osmotically dehydrated or not (F, B, F-OD and B-OD) before and after drying at 70 °C for 4 h.

	Before drying	**After drying**	**Before drying**	**After drying**
Sample	**Moisture (% bu)**		**Water activity**	
F	91.30 ± 0.04	29.73 ± 0.003	0.990 ± 0.001	0.792 ± 0.004
B	93.02 ± 0.02	7.75 ± 0.78	0.991 ± 0.001	0.546 ± 0.004
F-OD	82.46 ± 0.09	10.30 ± 0.71	0.976 ± 0.001	0.481 ± 0.018
B-OD	72.51 ± 0.05	11.40 ± 1.13	0.975 ± 0.000	0.708 ± 0.004

Table 4. Moisture content and water activity of coated fresh and blanched samples, osmotically dehydrated or not (F-CO, B-CO, F-OD-CO and B-OD-CO) before and after drying at 70 °C for 4 h.

	Before drying	**After drying**	**Before drying**	**After drying**
Sample	**Moisture (% bu)**		**Water activity**	
F-CO	91.30 ± 0.01	30.90 ± 2.40	0.992 ± 0.001	0.795 ± 0.025
B-CO	91.92 ± 0.01	7.35 ± 0.21	0.991 ± 0.001	0.497 ± 0.010
F-OD-CO	85.64 ± 0.04	10.50 ± 0.01	0.985 ± 0.001	0.473 ± 0.010
B-OD-CO	76.74 ± 0.08	10.85 ± 0.64	0.981 ± 0.001	0.690 ± 0.011
F	90.71 ± 0.02			

It was observed that moisture content of the fresh fruits was slightly different between the two sets of experiments (91.30 and 90.71), being the samples of the second batch the ones with the lowest moisture (Tables 3 and 4). Blanching and edible coating application caused an

increase in samples moisture due to their immersion in water and/or aqueous solution. However, the water activity of these samples were similar to the values found for the fresh slices (Tables 3 and 4).

As expected, osmotic treatment caused a decrease at moisture content and water activity (F-OD, B-OD, F-OD-CO and B-OD-CO) compared to the non-dehydrated samples (F, B, F-CO and B-CO). The blanched slices had the lowest moisture content after osmotic treatment (B-OD and B-OD-CO) (Tables 3 and 4) probably because the thermal treatment caused damages to the cellular tissue, increasing solutes uptake. Del Valle et al. (1998), Moreno et al. (2000) and Escobar et al. (2007) obtained similar results working with apple, strawberry and carrot, respectively.

After drying fresh slices coated or not (F and F-CO) presented high values of moisture and water activity (Tables 3 and 4). The drying of these samples (F and F-CO) under the established conditions (70 ^{o}C, 1 m.s^{-1}, 4 h) was not enough to reduce water activity to values that could ensure its microbiological safety (<0.6, YAN et al., 2008).

Osmotically dehydrated fresh samples coated or not (F-OD and F-OD-CO) presented low values of moisture and the lowest water activity after drying, which was related to solute impregnation (Tables 3 and 4). In fact, these were the dried fruits with the lowest values of water activity among all the samples ensuring that no microbial growth in the products would take place.

After drying, blanched fruits with or without coating (B and B-CO) presented the lowest moisture content among the studied pre-treatments. Water activity of these samples were lower than 0.6, however were not the lowest values among pre-treated and dried samples (Tables 3 and 4).

Osmo-dehydrated blanched papayas coated or not (B-OD and B-OD-CO), presented low moisture after drying but their water activity resulted higher than 0.6. This behavior is probably related to the sorption properties of products with high sugar content (Tables 3 and 4) because usually sorption isotherms of food present sigmoidal shape. However, when food composition had high contents of low weight soluble compounds, as sugars, the inflexion point is related to low moisture and high water activity. Among the studied pre-treatments to drying, B-OD and B-OD-CO were the least effective to decrease water activity.

Tables 5 and 6 present color parameters L*, a* and b* of the samples dried in the first and second batch of experiments, respectively.

Blanching caused darkening of the fresh slices (Tables 5 and 6), which is usually a result of thermal treatment of fruits and vegetables (DUTTA et al., 2006; OSORIO et al., 2007).

Regarding F and F-CO samples, OD of these fruits (F-OD and F-OD-CO) increased a* and b* values of the slices (Tables 5 and 6), probably due to the concentration effect. OD of blanched fruits (B-OD and B-OD-CO) decreased L* and b* values of the samples in comparison to the blanched slices (B and B-CO). B-OD and B-OD-CO samples were darker than the fresh fruits (F and F-CO) (Tables 5 and 6).

Working with pumpkins puree, Dutta et al. (2006) verified that blanching caused darkening of the samples and Osorio et al. (2007) observed color loss in tamarillo after osmotic treatment.

After drying uncoated and coated samples became darker during drying (Tables 5 and 6), except for the coated osmo-treated blanched slices (B-OD-CO) (Table 6).

After drying, a* and b* parameters of non-coated samples (F, F-OD, B, B-OD) decreased. Yellowness was more affected by drying than redness (Table 5). On the other hand, redness and yellowness of coated papaya slices increased after drying, probably because coat applying on the fruits surface avoided oxidative degradation of the pigments during drying.

Visual color changes have been related to carotenes degradation in papaya puree (AHMED et al., 2002). In this work, coated fruits (F-CO, F-OD-CO, B-CO, B-OD-CO) presented higher a* and b* parameters after drying (Table 6), suggesting that pectin coat avoided pigments oxidation of papaya during drying.

Table 5. L*, a* and b* values of fresh and blanched samples, osmotic dehydrated or not (F, B, F-OD and B-OD) before and after drying at 70 °C for 4 h.

	Before drying	After drying	Before drying	After drying	Before drying	After drying
Sample	**L***		**a***		**b***	
F	55.93 ± 0.41	45.91 ± 2.11	31.26 ± 2.37	29.67 ± 1.70	43.69 ± 1.62	28.06 ± 1.09
B	44.78 ± 0.30	43.82 ± 0.42	29.22 ± 0.32	27.77 ± 1.52	44.78 ± 0.14	37.12 ± 2.07
F-OD	55.48 ± 1.05	57.59 ± 2.68	34.34 ± 1.11	29.64 ± 1.40	48.25 ± 0.29	45.25 ± 1.64
B-OD	43.08 ± 0.74	40.24 ± 0.38	31.95 ± 0.72	29.26 ± 0.79	44.23 ± 0.87	24.17 ± 1.04

Table 6. L*, a* and b* values of coated fresh and blanched samples, osmotic dehydrated or not (F-CO, B-CO, F-OD-CO and B-OD-CO) before and after drying at 70 °C for 4 h.

	Before drying	After drying	Before drying	After drying	Before drying	After drying
Sample	**L***		**a***		**b***	
F-CO	47.09 ± 1.08	39.03 ± 0.94	24.77 ± 2.02	31.55 ± 0.6	37.99 ± 3.43	40.43 ± 2.55
B-CO	44.18 ± 0.70	42.04 ± 2.04	26.68 ± 0.76	35.50 ± 0.46	31.25 ± 0.88	47.14 ± 1.16
F-OD-CO	57.40 ± 2.55	51.61 ± 1.00	28.68 ± 2.40	33.09 ± 0.53	43.04 ± 0.43	45.77 ± 0.86
B-OD-CO	43.29± 0.42	54.87 ± 0.75	26.96 ± 0.15	35.16 ± 0.71	26.62 ± 1.28	45.41 ± 0.87

4. Conclusions

OD experiments using different solution concentrations and dehydration times presented slight and non-significant papaya color changes in the studied ranges, showing that papaya pigments were preserved during this process. An expression for the water activity of osmotically dehydrated blanched samples was found.

Drying experiments showed that blanching was not an appropriated pre-treatment to preserve sample color and B-OD was not effective to reduce water activity.

Osmotic dehydration of fresh fruits coated or not resulted in the lowest water activities after drying showing the potential of this process to obtain stable products and to save heat energy.

Coated fruits presented higher a* and b* parameters than non-coated samples after drying, suggesting that coatings avoided oxidation of color pigments during drying.

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CAPÍTULO II

EFFECT OF BLANCHING ON OSMOTIC DEHYDRATION OF PAPAYA OF FORMOSA CULTIVAR (*Carica papaya* L.)

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Abstract

The aim of this work was to evaluate the influence of blanching on osmotic dehydration in sucrose solutions of papaya of Formosa cultivar. The influence of both treatments on texture was also evaluated. Blanching caused damages to the cellular structure resulting in higher water loss and sugar gain as well as greater effective diffusion coefficients than those determined in fresh papayas. Invertase influenced sugar diffusivities during osmotic dehydration and was characterized. Softening of blanched slices was verified. Probably as a result of calcium impregnation, osmotically dehydrated fresh and blanched fruits tend to be harder. Considering the low cost of production of papaya and the invertase characteristics verified in the experiments, the fruit presented as a potential source for the invertase extraction.

Keywords: osmotic dehydration, blanching, papaya, diffusion coefficients, invertase

1. Introduction

Papaya is a typical fruit of tropical regions presenting high amounts of vitamin C and mineral salts. The fruit does not present high pro-vitamin A activity, however, because it has long periods of crop and low cost of production, it becomes an important source of carotenoids (Kimura et al., 1991; Rodriguez-Amaya, 1996). Processing of the fruit could represent an alternative to minimize the great harvest and post-harvest losses, adding value to the final product.

Osmotic dehydration (OD) is a process based on water removal from a product through its immersion in a hypertonic solution. During the process, solutes gain from the solution to the food and loss of some intracellular constituents with low molecular weight

from the food to the solution are verified. The result of these transfers is a decrease on moisture content with a simultaneous increase on solids, besides changes on chemical composition of the dehydrated food (EREN; KAYMAK-ERTEKIN, 2007; FERNANDES et al., 2006; LENART, 1996; RAOULT-WACK, 1994).

An advantageous of this minimal process is the possibility of modify food formulation by incorporating different solutes in the porous structure of fruits and vegetables. Desirable effects to maintain tissue firmness were verified adding calcium to osmotic solutions (HEREDIA et al., 2007; RODRIGUES et al. 2003), since calcium ions can bind to the pectin compounds present in the cellular wall and middle lamella reinforcing their structural integrity (PEREIRA et al., 2009; PANI et al., 2008; MARTÍN-DIANA et al., 2007; ANINO et al., 2006; ALZAMORA et al., 1997).

Blanching is widely used at minimally processed food preparation aiming enzymatic inactivation, microorganisms destruction or injury to reduce initial microbial of the food or awareness of the survivor microorganisms to posterior procedures (ALZAMORA et al., 1997). The great inconvenient of thermal treatment are the structural damages to the food resulting in its softening (SILVA et al., 2011; KOWALSKA et al., 2008; PAREDES ESCOBAR et al., 2007; ALZAMORA et al., 1997). When applied prior to drying or another treatment, researchers verified higher dehydration rates and/or reduced time of process (KOWALSKA et al., 2008; ESCOBAR et al., 2007).

Invertase (β-fructofuranosidase) is the enzyme that catalysis enzymatic hydrolysis of sucrose in equal proportions of glucose and fructose (Ensymm, 2007; Guimarães et al., 2007). Chan and Kwok (1975) noticed that invertase activity in papayas of Solo cultivar was 0.821 $U.mg^{-1}$ of protein. Authors verified that the enzyme must be inactivated, because half of the sucrose content is hydrolysis by invertase in 2.66 min after manipulation.

The objectives of this work were as follow: a) to evaluate the effect of blanching on OD kinetics of papaya of Formosa cultivar, b) verify the effect of blanching and osmotic dehydration on the fruits texture and c) characterize the invertase present in papaya regarding its stability, optimums operating pH and temperature and determination of its kinetics parameters.

2. Materials and methods

2.1. Samples preparing

Papaya of Formosa cultivar from São Paulo State (Brazil) were purchased at São José do Rio Preto Supplying Center (CEAGESP – São José do Rio Preto, São Paulo, Brazil). Ripe fruits with orange peels and approximately 8.00% ± 0.00 of reducing sugars were used in the experiments.

Papayas were longitudinally cut in four pieces, the seeds were removed and each portion was sectioned using a manual cutter in cylinders with diameter of 3.6 cm. These pieces were cut in slices with 1.0 cm of thickness. The samples were kept and mixed in a plastic bag from where the slices to be used in the experiments were randomly removed.

For the enzymatic assays two ripe fruits with orange peels weighting about 0.950 g were used. The fruits were cut longitudinally in four pieces and two opposites sections of each one were taken to be used in the experiments. These sections were crushed and 100 g of pulp were used to extract the enzyme. Soluble solids content and total solids of the pulp were measured.

2.2. Blanching

Papaya slices were put in a stainless basket and dipped in boiling water (98.3 °C at São José do Rio Preto – SP – Brazil) for 2 min. After this time, the samples were cooled in tap water for 1 min.

2.3. Osmotic dehydration

Osmotic solutions were prepared with commercial sucrose (refined sugar) purchased in a local market, food grade pentahydrate calcium lactate in powder from PURAC Synthesis – Brazil (1% w/w concentration), food grade L (+)-lactic acid in 85% solution from PURAC Synthesis – Brazil (concentration of 0.1 M) and distilled water.

The OD experiments of fresh and blanched fruits were conducted in 40%, 50% and 60% w/w sucrose aqueous solutions with 1% w/w of calcium lactate and 0.1 M of lactic acid. Processing times were 30, 60, 120, 240 and 360 min.

OD experiments were conducted into a stainless steel vat with an external jacket for water circulation at 27 °C. Osmotic solutions were continuously stirred using a 1.6 kW

mechanical stirrer (Marconi, model MA-261 - Brazil) with a 10 cm diameter propeller and rotation at 1000 rpm. The ratio between sample and solution masses were at least 1:10.

Equilibrium experiments were conducted with fresh and blanched cylindrical slices of 4 mm thickness. The experiments were conducted in a BOD incubator at 27 °C with orbital agitation of 117.5 rpm. To achieve the equilibrium, samples dehydrated in 50 and 60% solutions were kept into the solutions for 48 h and samples dehydrated in 40% solutions were treated for 72 h. The ratio between sample and solution masses was 1:10.

After osmotic treatments samples were removed from the solutions, washed with distilled water for 10 s and dried on absorbed paper.

Total solids, reducing and total sugar contents and texture of the samples were determined before and after process.

Texture of fresh and blanched papaya slices submitted to OD was measured in an independent assay. Sucrose solutions at 40%, 50% and 60% w/w concentration added with 1% w/w calcium lactate and 0.1 M of lactic acid were prepared in glass recipients and dehydrations were conducted in a BOD incubator at 27 °C with orbital agitation of 160 rpm for 30, 60, 120, 240 and 360 min.

2.4. Control of OD process

Through experimental data, total mass variation in relation to initial mass during papaya OD was calculated according to Equation 1.

$$\Delta M = \frac{M - M^0}{M^0} \quad (1)$$

where ΔM represents total mass variation in relation to initial mass; M is the mass at time t, in kg; M^0 is the initial mass at time t = 0, in kg.

Water loss WL and sugar gain SG in relation to initial mass were calculated for the OD, through the mass balances shown in Equations 2 and 3.

$$WL = \frac{(w_w M) - (w_w^0 M^0)}{M^0} \quad (2)$$

$$SG = \frac{(w_s M) - (w_s^0 M^0)}{M^0} \quad (3)$$

where w_w and w_s represent, respectively, water and sugar contents after a time t of OD. w_w^0 and w_s^0 are these contents before OD (t = 0). For the fresh samples, as sucrose was not

detected, due to invertase activity, calculations considered the reducing sugar contents. For the blanched fruits, sucrose contents were used in the calculations.

Solutes gain was calculated according to Equation 4.

$$Sol = \Delta M - WL \tag{4}$$

where *Sol* represents sucrose, calcium lactate and lactic acid gains.

2.5. Effective diffusion coefficients

To calculate the effective diffusion coefficients (D_k) of water and sugar (sucrose/reducing sugar) of papaya slices submitted to OD, Fick´s second law applied to an infinite slab (Equation 5) and the superposition of the solution for an infinite cylinder and an infinite slab (Equation 6), ie a finite cylinder, were used. The integrated solutions in terms of the mean water or sugar contents in the slice at time *t* of OD $\overline{w}_k(t)$, are (CRANK, 1975):

$$w_k = \left(\frac{\overline{w}_k(t) - w_k^{eq}}{w_k^0 - w_k^{eq}} \right)_{\substack{\text{inf.} \\ slab}} = \frac{8}{\pi^2} \left[e^{\left(\frac{-\pi^2}{4} \frac{D_k t}{z^2} \right)} + \frac{1}{9} e^{\left(\frac{-9\pi^2}{4} \frac{D_k t}{z^2} \right)} + \frac{1}{25} e^{\left(\frac{-25\pi^2}{4} \frac{D_k t}{z^2} \right)} + \ldots \right] \tag{5}$$

$$w_k = \left(\frac{\overline{w}_k(t) - w_k^{eq}}{w_k^0 - w_k^{eq}} \right)_{\substack{\text{inf.} \\ slab}} \cdot \left(\frac{\overline{w}_k(t) - w_k^{eq}}{w_k^0 - w_k^{eq}} \right)_{\substack{\text{inf.} \\ cyl.}} =$$

$$= \left\{ \frac{8}{\pi^2} \left[e^{\left(\frac{-\pi^2}{4} \frac{D_k t}{z^2} \right)} + \frac{1}{9} e^{\left(\frac{-9\pi^2}{4} \frac{D_k t}{z^2} \right)} + \frac{1}{25} e^{\left(\frac{-25\pi^2}{4} \frac{D_k t}{z^2} \right)} + \frac{1}{49} e^{\left(\frac{-49\pi^2}{4} \frac{D_k t}{z^2} \right)} + \ldots \right] \right\} \cdot$$

$$\cdot \left\{ 4 \left[\frac{1}{5.783} e^{\left(-5.783 \frac{D_k t}{a^2} \right)} + \frac{1}{30.472} e^{\left(-30.472 \frac{D_k t}{a^2} \right)} + \frac{1}{74.887} e^{\left(-74.887 \frac{D_k t}{a^2} \right)} + \frac{1}{139.039} e^{\left(-139.039 \frac{D_k t}{z^2} \right)} + \ldots \right] \right\} \tag{6}$$

$$k = w, sc, rs$$

where *k* represents the species: *w* is the water, *sc* is sucrose and *rs* is reducing sugar; *eq* indicates equilibrium; *0* indicates initial (at t = 0); w_k represents mass fraction of the specie; $\overline{w}$ is the average content of the specie at time *t*; D_k is the effective diffusion coefficient of the specie, in $m^2.s^{-1}$; *z* is the initial thickness of the samples; *a* is the initial radius of the samples.

2.6. Extraction of invertase from papaya

Based on the methodology used by Chan and Kwok (1975), 100 g of papaya pulp were homogenated and submitted to an extraction process using ketone at 4 °C. It was obtained 4.35 g of dried extract (DE). Enzymatic extract was obtained shaking a solution of 0.5 g of

DE in 100 mL of acetate buffer pH 5.0 (0.01 M) during 20 min. After this time the suspension was centrifuged at 800 rpm for 15 min at 13 °C and then filtered in glass wool.

2.7. Enzymatic assay

Enzymatic assays were conducted with 150 μL of acetate/phosphate buffer 0.2 M, 100 μL of substrate (sucrose 1 M), enzyme and distilled water completing a final volume of 500 μL. The reducing sugars were determined using Somogy-Nelson method. To determine optimum pH the range of 3.0-8.0 was analyzed. To find optimum operating temperature the range of 30-65 °C was studied. Michaelis-Menten kinetic parameters, K_m and V_m, were estimated from Lineweaver-Burk plots in the range of 0.03-0.4 μM of sucrose concentration.

Lineweaver-Burk plot provides a useful graphical method for determine Michaelis–Menten parameters, taking the reciprocal of Michaelis–Menten equation. Thus:

$$\frac{1}{V} = \frac{K_m + [S]}{V_m [S]} = \frac{K_m}{V_m}\frac{1}{[S]} + \frac{1}{V_m} \quad (7)$$

where: V is the reaction rate; K_m is Michaelis-Menten constant; V_m is the maximum reaction rate and $[S]$ is the substrate concentration.

The linear coefficient of plotting $1/[S]$ *versus* $1/V$ is the reciprocal of V_m and the angular coefficient is K_m/V_m.

2.8. Analytical methodologies

Total solid contents of fresh, blanched and osmotically dehydrated fruits were determined in triplicate in a vacuum oven at 60 °C, 10 kPa, until constant weight.

Total and reducing sugars of fresh, blanched and osmotically dehydrated samples were determined in replicate by redox titration (WILLIAM, 1970).

Textures of these samples were determined in replicate (7-9 samples) using TPA (Texture Profile Analysis) method in an Universal texturometer (TA-XT2i Texture Analyser, Stable Micro System, Surrey, UK.). Cylindrical samples (10 mm of height and 260.155 mm^2 of cross sectional area) were individually compressed until 30% of total strain for 5 s in a rate of 1 $mm.s^{-1}$ using an acrylic probe with 35 mm of diameter. Results were expressed as hardness.

2.9. Statistical analysis and fitting

Results were statistically analyzed by an Analysis of Variance (ANOVA) and Tukey test in a 5% significance level, using Excel 2007 (Microsoft – USA).

Effective diffusion coefficients were calculated according to Equations 5 and 6 using experimental data by minimizing the squares of the deviations between predicted and observed values, with the aid of Statistica 7.0 software. The series converged quickly so six terms were enough. The fitting efficiency was evaluated by the determination coefficient (R^2) and the mean relative error root square (*RRMS*); the latter according to Equation 8.

$$RRMS\ (\%) = 100\left\{\frac{1}{N}\sum_{n=1}^{N}\left[\frac{\left(x^{calc} - x^{\exp}\right)}{x^{\exp}}\right]\right\}^{1/2} \quad (8)$$

where x^{calc} represents water or sugar contents calculated according to Equations 5 and 6; x^{exp} is the observed value; N represents the number of observations.

3. Results and discussions

3.1. Osmotic dehydration

Tables 1 to 6 present total solids (w_{ts}), sucrose (w_{sc}) and reducing sugars (w_{rs}) average contents, total mass variation (ΔM), water loss (*WL*) and solutes gain (*Sol*) in relation to initial mass (M^0) during OD of fresh and blanched papaya slices of Formosa cultivar in 40%, 50% and 60% w/w sucrose solutions added with 1% w/w of calcium lactate and 0.1 M of lactic acid.

Blanching resulted in decrease of total solids of papaya samples comparing to the respective fresh fruits, due to losses of soluble solids during thermal treatment. This lost was reflected in the reducing sugars content which was higher in fresh than in blanched fruits (Tables 1 to 6).

As noticed by Kowalska et al. (2008) and Silva et al. (2011) for pumpkins, thermal treatment possibly damaged the cellular tissue of papayas, since, for the same process time, higher solute gain was observed for the blanched fruits in relation to the fresh samples independently of solution concentration (Tables 1 to 6). Sugar gain of fresh samples (reducing sugar) increased when increasing dewatering time and low dependence with solution concentration was observed (Tables 1, 3 and 5). Tables 2, 4 and 6 showed that sucrose gain of blanched fruits presented low dependence with solution concentration for short OD times.

Increasing solution concentration increased sucrose gain of these samples for dewatering times up to 120 min (Tables 2, 4 and 6).

Water loss increased with operation time and solution concentration independently of the performance of a pretreatment. Meanwhile, for equal process time and solution concentration, blanched samples presented higher water loss than fresh fruits, corroborating the possibility of tissue damages (Tables 1 to 6).

Blanched fruits presented higher total mass variation than fresh samples at the beginning of the dehydration processes. Considering long time of processes, total mass variations of fresh samples were higher than the blanched slices since their solutes gain were very low (Tables 1 to 6.). Similar results were found for pumpkin and carrots (KOWALSKA et al., 2008; ESCOBAR et al., 2007).

After the same process time, the higher the osmotic concentration, the higher the total solid and reducing sugar contents measured in the fresh samples dehydrated in sucrose solutions at 40% and 50%. However, minor changes were observed for fresh papayas dehydrated in 60% osmotic solution (Tables 1, 3 and 5). For the blanched fruits, the higher the osmotic concentration, the higher were the total solids in the dehydrated samples after the same time of OD. Considering sucrose content, few differences between blanched samples dehydrated in 50% and 60% solutions were observed (Tables 2, 4 and 6).

Tables 1, 3 and 5 showed that no sucrose content was detected in the fresh fruits as a result of the fast sugar inversion that occurred during samples manipulation. In fact, during OD of fresh samples, independently of process time, sucrose was not detected (Tables 1, 3 and 5). On the other hand, reducing sugars average contents of osmo-treated fresh slices increased with process time in relation to the initial value (Tables 1, 3 and 5), corroborating the hypothesis of the fast inversion of these sugars.

Regarding OD of blanched papayas it was verified the increasing on sucrose content with time due to solids incorporation (Tables 2, 4 and 6). Reducing sugars content of blanched samples remained practically constant during the first hour of OD. For longer dehydration times, slight decrease of reducing sugars was noticed, which were probably transferred to the dehydrating solution (Tables 2, 4 and 6).

Table 1. Total solids (w_{ts}), sucrose (w_{sc}) and reducing sugars (w_{rs}) average contents, total mass variation (ΔM), water loss (WL) and solutes gain (Sol) in relation to initial mass during OD of fresh papaya slices in 40% w/w sucrose solution added with 1% w/w of calcium lactate and 0.1 M of lactic acid.

Sample	**w_{ts} (g/100 g)**	**w_{sc} (g/100 g)**	**w_{rs} (g/100 g)**	**ΔM (g/100 g)**	**WL (g/100 g)**	**Sol (g/100 g)**
Fresh	12.12 ± 0.34	ND	8.24 ± 0.68	-	-	-
			Osmotic dehydration			
Time (min)						
30	16.49 ± 0.90	ND	11.64 ± 0.72	-8.07	-11.11	3.04
60	18.11 ± 0.87	ND	13.01 ± 1.27	-11.85	-15.70	3.86
120	20.10 ± 0.28	ND	15.84 ± 0.26	-17.89	-22.27	4.39
240	23.25 ± 3.45	ND	19.23 ± 0.06	-26.26	-31.27	5.02
360	28.25 ± 0.12	ND	21.90 ± 0.38	-31.76	-38.92	7.16

ND: Non-detected.

Table 2. Total solids (w_{ts}), sucrose (w_{sc}) and reducing sugars (w_{rs}) average contents, total mass variation (ΔM), water loss (WL) and solutes gain (Sol) in relation to initial mass during OD of blanched papaya slices in 40% w/w sucrose solution added with 1% w/w of calcium lactate and 0.1 M of lactic acid.

Sample	**w_{ts} (g/100 g)**	**w_{sc} (g/100 g)**	**w_{rs} (g/100 g)**	**ΔM (g/100 g)**	**WL (g/100 g)**	**Sol (g/100 g)**
Fresh	10.88 ± 0.02*	ND	7.56 ± 1.02*	-	-	-
Blanched	9.33 ± 0.01*	2.61 ± 0.02*	4.14 ± 0.05*	-	-	-
Osmotic dehydration						
Time (min)						
30	16.30 ± 0.03	9.47 ± 0.01	3.53 ± 0.02	-16.27	-20.58	4.31
60	19.66 ± 0.12	12.61 ± 0.03	3.35 ± 0.02	-16.94	-23.93	6.99
120	23.95 ± 0.06	17.46 ± 0.07	2.88 ± 0.01	-19.13	-29.16	10.04
240	29.59 ± 0.05	24.33 ± 0.07	1.54 ± 0.00	-26.68	-39.04	12.37
360	34.14 ± 0.05	28.43 ± 0.02	2.02 ± 0.02	-22.09	-39.36	17.27

*Average values among all the fresh and blanched samples.

ND: Non-detected.

Table 3. Total solids (w_{ts}), sucrose (w_{sc}) and reducing sugars (w_{rs}) average contents, total mass variation (ΔM), water loss (WL) and solutes gain (Sol) in relation to initial mass during OD of fresh papaya slices in 50% w/w sucrose solution added with 1% w/w of calcium lactate and 0.1 M of lactic acid.

Sample	**w_{ts} (g/100 g)**	**w_{sc} (g/100 g)**	**w_{rs} (g/100 g)**	**ΔM (g/100 g)**	**WL (g/100 g)**	**Sol (g/100 g)**
Fresh	9.82 ± 1.43	ND	7.06 ± 1.47	-	-	-
Osmotic dehydration						
Time (min)						
30	14.78 ± 0.55	ND	11.72 ± 0.39	-13.08	-16.11	3.03
60	16.21 ± 0.21	ND	12.49 ± 0.67	-17.85	-21.34	3.49
120	19.51 ± 0.94	ND	15.78 ± 0.16	-25.43	-29.28	4.72
240	25.13 ± 0.35	ND	20.12 ± 1.21	-37.29	-41.26	5.93
360	30.00 ± 0.15	ND	24.71 ± 2.18	-46.99	-53.07	6.08

ND: Non-detected.

Table 4. Total solids (w_{ts}), sucrose (w_{sc}) and reducing sugars (w_{rs}) average contents, total mass variation (ΔM), water loss (WL) and solutes gain (Sol) in relation to initial mass during OD of blanched papaya slices in 50% w/w sucrose solution added with 1% w/w of calcium lactate and 0.1 M of lactic acid.

Sample	w_{ts} **(g/100 g)**	w_{sc} **(g/100 g)**	w_{rs} **(g/100 g)**	ΔM **(g/100 g)**	WL **(g/100 g)**	Sol **(g/100 g)**
Fresh	10.88 ± 0.02*	ND	7.56 ± 1.02*	-	-	-
Blanched	9.33 ± 0.01*	2.60 ± 0.02*	4.15 ± 0.04*	-	-	-
			Osmotic dehydration			
Time (min)						
30	18.61 ± 0.02	11.64 ± 0.05	3.50 ± 0.00	-13.00	-19.86	6.85
60	21.60 ± 0.01	14.58 ± 0.01	3.16 ± 0.02	-16.24	-24.99	8.76
120	28.48 ± 0.01	21.80 ± 0.00	3.18 ± 0.00	-26.20	-37.88	11.68
240	34.08 ± 0.01	27.17 ± 0.74	2.57 ± 0.00	-37.91	-49.74	11.83
360	39.04 ± 0.03	31.10 ± 0.74	2.21 ± 0.10	-47.08	-58.40	11.32

*Average values among all the fresh and blanched samples.

ND: Non-detected.

Table 5. Total solids (w_{ts}), sucrose (w_{sc}) and reducing sugars (w_{rs}) average contents, total mass variation (ΔM), water loss (WL) and solutes gain (Sol) in relation to initial mass during OD of fresh papaya slices in 60% w/w sucrose solution added with 1% w/w of calcium lactate and 0.1 M of lactic acid.

Sample	**w_{ts} (g/100 g)**	**w_{sc} (g/100 g)**	**w_{rs} (g/100 g)**	**ΔM (g/100 g)**	**WL (g/100 g)**	**Sol (g/100 g)**
Fresh	10.01 ± 0.04	ND	7.18 ± 0.04	-	-	-
Osmotic dehydration						
Time (min)						
30	14.17 ± 0.06	ND	11.23 ± 0.04	-12.44	-14.83	2.39
60	16.88 ± 0.02	ND	13.56 ± 0.06	-17.95	-21.79	3.84
120	19.48 ± 0.02	ND	15.38 ± 0.02	-27.30	-31.45	4.15
240	25.39 ± 0.01	ND	20.99 ± 0.63	-42.79	-47.30	4.51
360	31.23 ± 0.01	ND	26.86 ± 0.13	-53.27	-57.85	4.58

ND: Non-detected.

Table 6. Total solids (w_{ts}), sucrose (w_{sc}) and reducing sugars (w_{rs}) average contents, total mass variation (ΔM), water loss (WL) and solutes gain (Sol) in relation to initial mass during OD of blanched papaya slices in 60% w/w sucrose solution added with 1% w/w of calcium lactate and 0.1 M of lactic acid.

Sample	**w_{ts} (g/100 g)**	**w_{sc} (g/100 g)**	**w_{rs} (g/100 g)**	**ΔM (g/100 g)**	**WL (g/100 g)**	**Sol (g/100 g)**
Fresh	10.88 ± 0.02*	ND	7.56 ± 1.02*	-	-	-
Blanched	9.33 ± 0.01*	2.53 ± 0.12*	4.14 ± 0.05*	-	-	-
			Osmotic dehydration			
Time (min)						
30	17.70 ± 0.12	10.51 ± 0.14	3.66 ± 0.00	-14.87	-20.60	5.73
60	22.04 ± 0.05	13.70 ± 0.26	3.78 ± 0.01	-22.05	-29.89	7.84
120	29.48 ± 0.04	20.09 ± 0.25	3.57 ± 0.04	-39.21	-47.80	8.59
240	41.47 ± 0.01	30.44 ± 0.01	3.35 ± 0.04	-42.27	-56.88	14.61
360	46.60 ± 0.00	36.12 ± 0.00	2.93 ± 0.01	-48.25	-63.03	14.78

*Average values among all the fresh and blanched samples.

ND: Non-detected.

Comparing OD of fresh papaya and blanched samples (Tables 1 to 6), it could be concluded that papaya of Formosa cultivar presented invertase activity which is responsible by sucrose hydrolysis in glucose and fructose. This fact explained the failure to measure the sucrose contents of osmo-dehydrated fresh fruits and the increases in their reducing sugar contents over dehydration (Tables 1, 3 and 5). Blanching inactivated the enzyme so sucrose contents of blanched fruits increased during OD (Tables 2, 4 and 6).

The effective diffusion coefficients of fresh and blanched papayas submitted to OD in 40%, 50% and 60% w/w sucrose aqueous solutions with calcium lactate (1% w/w) and lactic acid (0.1 M) are presented in Tables 7 and 8. It was calculated the effective diffusion coefficients of water and reducing sugars for the fresh fruits and water and sucrose for the blanched papaya slices. Table 7 presents the coefficients determined from Equation 5 that considered the slices as an infinite slab while Table 8 shows the coefficients determined from Equation (6) that considered the slices as a finite cylinder.

Even though the samples were better represented by a cylinder (0.9 cm thickness and 3.6 cm diameter), the fitting was higher for Equation 5 which considered an infinite slab representation. A good fitting was obtained from Equation 5 and the effective diffusion coefficients determination presented values of R^2 higher than 0.95 and *RRMS* values lower than 10% (Table 7).

Fitting of Equation 6, for a finite cylinder, presented R^2 values higher than 0.86 and *RRMS* lower than 10% (Table 8). The addition of one or more terms after the fourth term of the series in Equation 6 practically did not change the diffusion coefficient (less than 1%). Several terms would be necessary to reduce the determination coefficient as well as *RRMS*, specially the residual corresponding to the first experimental point (1800 s). Using Statistica version 7.0 only six terms were entered in the modeling and this mathematical difficulty resulted in the best fitting obtained using infinite slab representation.

As for pure sucrose solutions (HENRION, 1964), for each treatment time, the diffusion coefficients decreased when the concentration of the osmotic solution increased, except for the water diffusivities of blanched samples osmo-treated in 60% solutions which were higher than that treated in 50% solutions, even though these diffusivities were close. It was expected that samples dehydrated in 60% sucrose solution presented lower water diffusivities, but, probably, at high osmotic concentrations, the tissue damages were high, enhancing diffusion processes In addition, at 50% the water diffusion coefficient resulted lower than the sucrose ones (Tables 7 and 8).

Table 7. Effective diffusion coefficients of water ($D_w \times 10^{10}$, in $m^2 \cdot s^{-1}$) and sugar ($D_s \times 10^{10}$, in $m^2 \cdot s^{-1}$) during OD of fresh and blanched papayas of Formosa cultivar, calculated considering the slices as infinite slabs (Equation 5).

Sample	Osmotic solution, (%w/w)	Water			Sugar		
		D_w	*RRMS* (%)	R^2	D_s	*RRMS* (%)	R^2
Fresh	**40**	2.10	5.90	0.96	2.92	3.72	0.99
Blanched		5.32	5.67	0.99	4.35	3.49	0.99
Fresh	**50**	1.92	4.67	0.97	1.99	5.53	0.96
Blanched		3.94	3.22	0.99	4.27	4.91	0.99
Fresh	**60**	1.45	4.79	0.96	1.94	7.25	0.95
Blanched		4.51	8.82	0.98	3.15	5.17	0.98

s is the reducing sugars for the fresh samples (D_{rs}) and sucrose for the blanched fruits (D_{sc}).

Table 8. Effective diffusion coefficients of water ($D_w \times 10^{10}$, in $m^2 \cdot s^{-1}$) and sugar ($D_s \times 10^{10}$, in $m^2 \cdot s^{-1}$) during OD of fresh and blanched papayas of Formosa cultivar, calculated considering the slices as a finite cylinder (Equation 6).

Sample	Osmotic solution, (%w/w)	Water			Sugar		
		D_w	*RRMS* (%)	R^2	D_s	*RRMS* (%)	R^2
Fresh	**40**	1.25	3.88	0.92	1.69	3.72	0.93
Blanched		3.35	5.67	0.96	2.68	3.49	0.95
Fresh	**50**	1.14	4.33	0.91	1.48	6.76	0.91
Blanched		2.40	3.22	0.96	2.63	4.91	0.96
Fresh	**60**	0.75	4.79	0.86	1.05	7.25	0.87
Blanched		2.77	8.82	0.93	1.84	5.17	0.93

s is the reducing sugars for the fresh samples (D_{rs}) and sucrose for the blanched fruits (D_{sc}).

Diffusion coefficients of blanched fruits were considerable higher than the fresh ones. These results are in agreement with the hypothesis that thermal treatment damaged the cellular tissue, leading to loss of selective permeability of the plasmalemma and thus facilitating diffusive process (SILVA et al., 2011). Effective diffusivities of reducing sugar in non-treated tissues were higher than effective diffusion coefficients of water. This unusual behavior was related to invertase enzyme in papaya. This enzyme changes the papaya composition during osmotic dehydration thus influencing the kinetic of this process in different ways. Probably a new sugar flux of reducing sugar arose from fruit to solution. Moreover, it is possible that equilibrium values did not correspond to surface conditions during osmotic process due to the reaction rate.

The invertase in fresh papaya makes the osmotic dehydration difficult to control. Viberg and Sjömohol (1998) recommended that when an osmotic medium is selected for processing fruits which contain an active invertase it should be noted that the composition can change in the course of processing. They showed that the invertase activity in strawberries caused significant changes in the composition of the surrounding sucrose osmotic solution.
Blanched samples dehydrated in 50% solution presented higher sucrose diffusivity than effective diffusion coefficient of water which could be related to the use of different raw materials in the experiments (Tables 7 and 8).

The effective diffusion coefficients calculated for papaya of Formosa cultivar were lower than the ones calculated for pumpkins by Silva et al. (2011) and for carrots by Escobar et al. (2007), but within the range found for pumpkins by Garcia et al. (2007) and Kowalska et al. (2008).

3.2. Texture

Independent assays were conducted to determine the hardness of fresh, blanched and osmo-dehydrated papaya slices. For each OD time, a fresh and a blanched sample were used. Fresh and blanched samples were osmotically dehydrated in parallel.
Hardness, in N, of fresh, blanched and of these samples osmotically dehydrated is shown in Figures1 to 3.

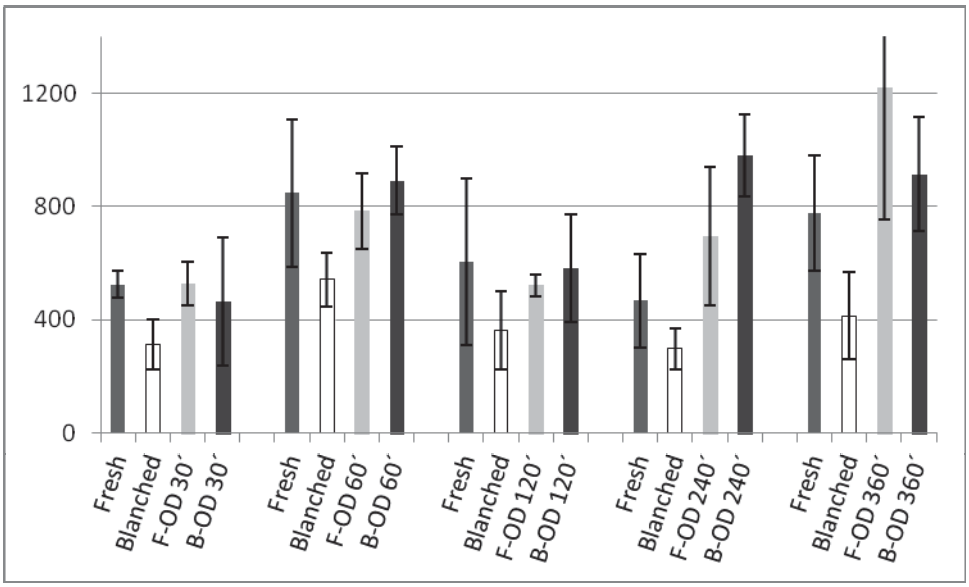

Figure 1. Hardness of fresh, blanched and these samples osmotically dehydrated in sucrose solution at 40% w/w with 1% w/w of calcium lactate and 0.1 M of lactic acid. F-OD: Osmo-dehydrated fresh fruits. B-OD: Osmo-dehydrated blanched fruits.

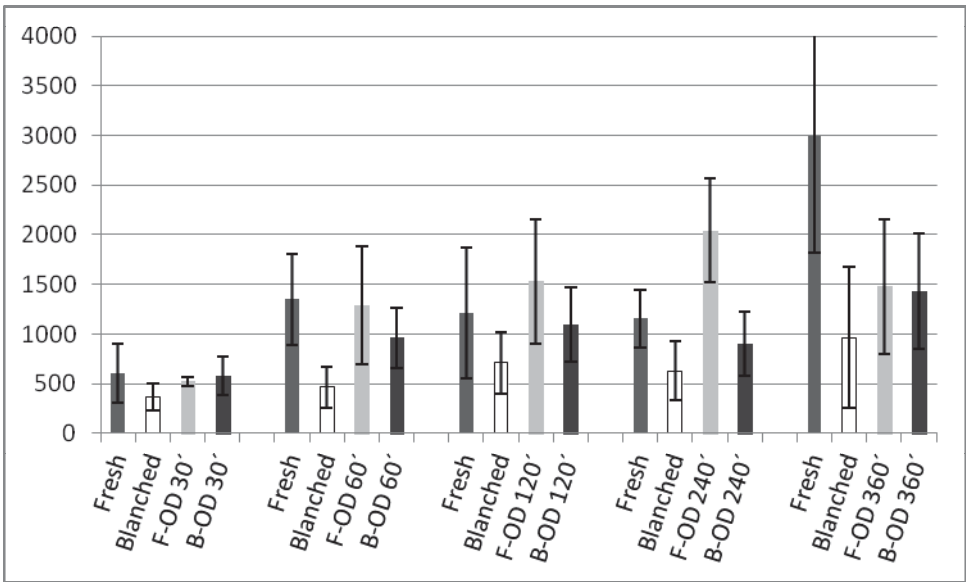

Figure 2. Hardness of fresh, blanched and these samples osmotically dehydrated in sucrose solution at 50% w/w with 1% w/w of calcium lactate and 0.1 M of lactic acid. F-OD: Osmo-dehydrated fresh fruits. B-OD: Osmo-dehydrated blanched fruits.

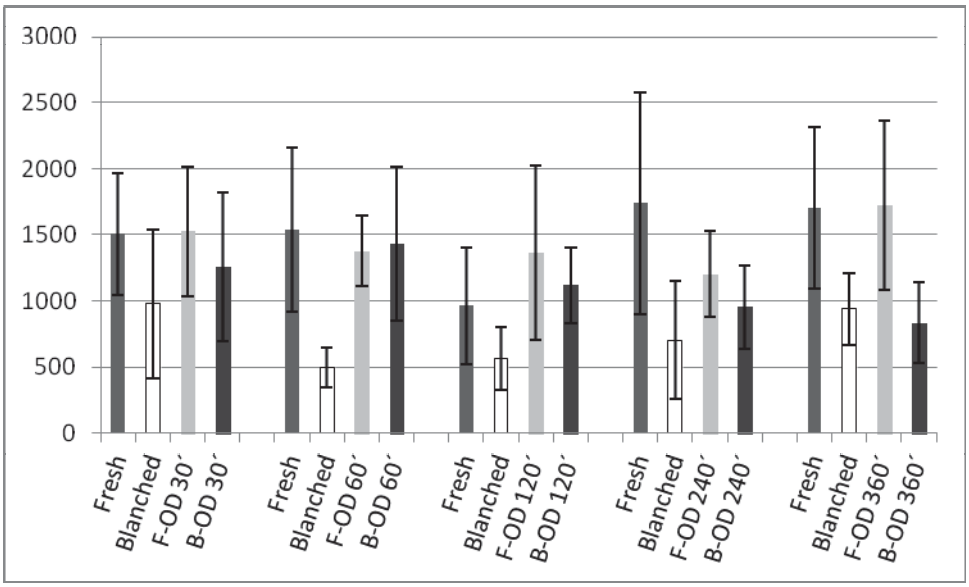

Figure 3. Hardness of fresh, blanched and these samples osmotically dehydrated in sucrose solution at 60% w/w with 1% w/w of calcium lactate and 0.1 M of lactic acid. F-OD: Osmo-dehydrated fresh fruits. B-OD: Osmo-dehydrated blanched fruits.

Heterogeneity of the data resulted in high standard deviations which are difficult to be avoided since mechanical properties of a cellular material are determined by structure and cell wall constituents. Small variations on ripeness, cultivar, harvest time and possible experimental errors influence the results and so increase the samples standard deviations (ARGADOÑA et al., 2002).

Besides the great standard deviations of the experiments some interesting tendencies were verified. Blanched samples were softer than the fresh slices denoting that blanching damaged the cellular structure, resulting in softning, as expected (Figures 1 to 3). Similar results were found by Kowalska et al. (2008), Moreno et al. (2000) and del Valle et al. (1998). In Figures 1 to 3 it was verified that osmo-dehydrated fresh fruits (F-OD) presented a trend to had similar hardness than the raw papaya which was related to solute impregnation, especially calcium ions. Adding calcium ions to the osmotic solution resulted in linkages between these ions and the pectic compounds of the cell walls and middle lamella, maintaining or even improving the hardness of osmo-treated fruits in relation to the fresh papayas.

It is possible that the damages caused to cellular membranes as a result of thermal treatment improved calcium uptake once osmotically dehydrated blanched fruits (B-OD) presented higher hardness than the blanched samples, denoting that the addition of calcium ions to the osmotic solution had a beneficial effect over papaya texture (Figures 1 to 3). In fact, the uptake of calcium into the matrix of the cell would help to maintain cell wall and

middle lamella integrity, since it would promote crosslinking of pectic polymers. Extensive cross-linking would facilitate formation of a cell wall network that would increase mechanical strength, as was observed for the B-OD samples.

The improvement on texture of calcium impregnated samples was observed by several authors. Higher rupture forces of apple cylinders impregnated with calcium comparing with non-treated samples were obtained by Anino et. al (2006). OD of guavas and papayas in sugar osmotic solutions with calcium resulted in higher rupture forces or higher hardness of the treated samples due to the ion incorporation (PEREIRA et al., 2009; MASTRANTONIO et al., 2005; RODRIGUES et al., 2003).

3.3. Characterization of papaya invertase

The papaya pulp used in the characterization of invertase presented total solids content of 14.78 ± 0.005 g/100 g and soluble solids content of 14.1%.

Figures 4 and 5 present the optimum temperature and pH of the enzyme which were 45 °C and 4.8, respectively. These values were close to the ones found for strawberries and Japanese pear (Viberg; Sjöholm, 1998; Hashizume et al., 2003). The fungal enzymes usually used in inverted sugar production present optimum pH around 4.5 and optimum temperature ranging from 55-60 °C (Romero-Gómez et al., 2000; Ensymm, 2007; Guimarães et al., 2007).

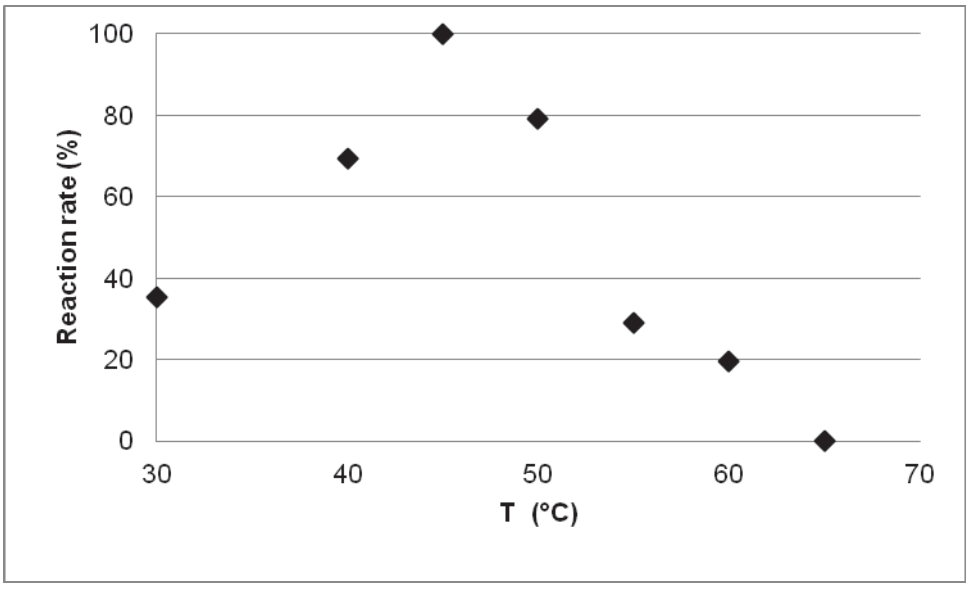

Figure 4. Optimum temperature of invertase extracted from papaya of Formosa cultivar.

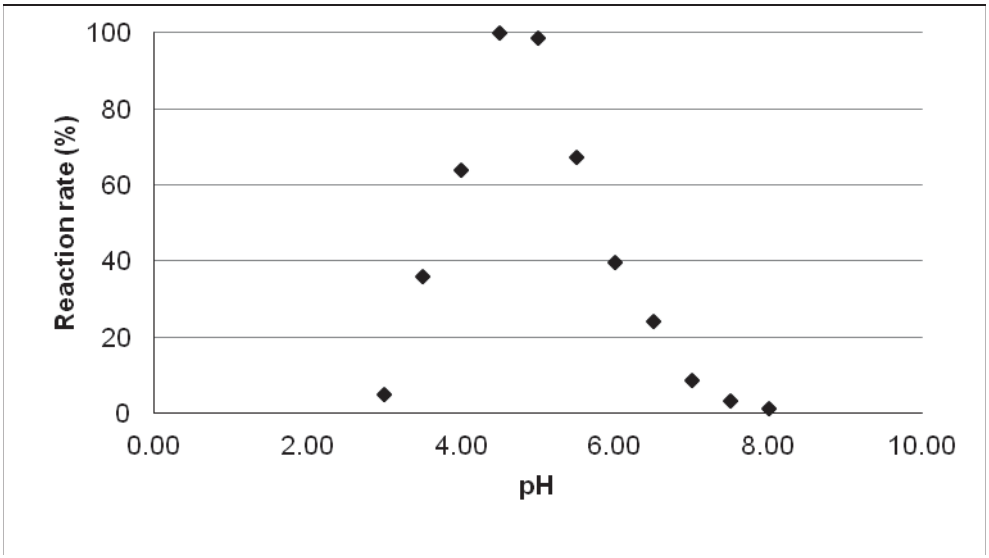

Figure 5. Optimum pH of invertase extracted from papaya of Formosa cultivar.

Increasing pH values up to 6.5, enzymatic activity decreased 75%; pH values below 3.5 decreased invertase activity from 35% of its original value.

Invertase contained in papayas of Formosa cultivar presented activity up to 30% when kept at 35 or 40 °C for until 4 h. At 55 °C its activity decreased 75% after 15 min.

Michaellis-Menten constant (K_m) and maximum velocity (V_m) of invertase extracted from papaya were 66.31 mM and 0.896 $\mu M \cdot min^{-1}$, respectively.

K_m of the invertase extacted from papaya of Formosa cultivar was higher than the found for bananas (2.7 mM), mango (5.0 to 0.9 mM) and Japanese pear (3.33 to 4.58 mM). The fungal invertases present K_m ranging from 2 to 7 mM (Chhatpar; Modi, 1974; Romero-Gómez et al., 2000; Hashizume et al., 2003; Ensymm, 2007; Guimarães et al., 2007).

Simplifying, Michaellis-Menten constant indicates the affinity of the enzyme by the substrate; higher its value, lower affinity. Therefore it was verified that the invertase of papaya presented lower affinity to sucrose than the enzymes extracted from fungi and another fruits (Ensymm, 2007; Guimarães et al., 2007; Hashizume et al., 2003; Romero-Gómez et al., 2000; Chhatpar; Modi, 1974).

The velocity of hydrolysis is proportional to the concentration of the sucrose-invertase compound, and to the fraction of the invertase which is combined with sucrose. Therefore, when the sucrose concentration is such that the velocity is at its maximum, then this proportion is equal 1. Papaya of Formosa cultivar presented high invertase concentration since the V_m value was almost 1.

4. Conclusions

Blanching of papaya damaged the cellular tissue in agreement with the greater diffusion coefficients found for the blanched fruits compared to the fresh papaya slices. In fresh tissue the effective diffusivities of sugar, measured as reducing sugar, were higher than the water ones, what was attributed to the invertase activity that changed the sugar composition during osmotic dehydration thus affecting the mass transfer of this process.

Softening of thermal treated slices was verified. Calcium impregnation during OD tend to improve hardness of fresh (F-OD) and blanched (B-OD) fruits.

Invertase was detected in papaya of Formosa cultivar and presented high concentration in the fruit.

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CAPÍTULO III

MOISTURE SORPTION ISOTHERMS AND THERMODYNAMIC PROPERTIES OF PAPAYA (*Carica papaya* L.)

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Abstract

Sorption isotherms of fresh, blanched and osmotically dehydrated papaya slices of Formosa cultivar were determined by static gravimetric method at 30, 50 and 70 °C. Equilibrium moisture content data were correlated by six mathematical models. The best fit of the experimental data was dependent of the sample treatment and the temperature. However, GAB model presented a good fit for all the treatments at all the studied temperatures. Blanched fruits presented an unusual behavior because, at the lowest and the highest levels of water activities, the isotherms of 70 °C overlapped the curves of 50 °C. The isosteric heats of sorption indicated that the water removal from the osmo-treated fruits required the largest amount of heat, followed by the fresh samples. Finally, water removal from the blanched papaya slices required the lowest quantities of heat. Differential enthalpy presented a linear relation to the differential entropy, so the enthalpy-entropy compensation theory was satisfied. The sorption processes were characterized as enthalpy driven.

Keywords: net isosteric heat of sorption, enthalpy-entropy compensation theory, osmotic dehydration, blanching, papaya

1. Introduction

Papaya is a tropical fruit that presents biological active compounds, such as vitamin C and carotenes. Besides its nutritive content, low cost and availability during all over the year, papaya becomes an important source of such compounds (RODRIGUEZ-AMAYA, 1996). Brazil is placed as the second major producer of papaya in the world (FAOSTAT, 2011).

Drying is one of the most widely used methods for food preservation. The aim of this process is the water removal from the food until levels that minimize or inhibit microbial

growth and deterioration reactions (DOYMAZ, 2008; DOYMAZ, 2007). Drying also contributes to a reduction in production losses and to an extension in the storage life. During drying occur physical, chemical and biochemical transformations which can lead to product quality depreciation (MUJUMDAR, 1997). The use of pre-treatments as an alternative to improve food qualities has been studied by many authors (SILVA et al., 2011; GONZÁLEZ-FÉSLER et al., 2008; LEERATANARAK et al., 2006). Blanching is commonly used prior to drying to avoid enzymatic reactions that could affect food quality during processing and storage (MOLINA FILHO et al., 2011; SILVA et al., 2011). Osmotic dehydration as a pre-treatment for food drying can prevent losses of nutritional compounds and natural color, also becoming possible to incorporate solutes of interest in the porous structure of fruits and vegetables (SANTOS; SILVA, 2008; PANI et al., 2008; HEREDIA et al., 2007).

Water sorption isotherms represent the equilibrium relationship between the water activity and the moisture content of the food sample at constant temperature. As appointed by Moreira et al. (2010a), sorption isotherms models constitute an essential component of the overall drying theory. Knowledge of the relationship between water content and vapor pressure equilibrium permits to design drying process and equipments and calculate the energy consumption since the vapor pressure gradients are the essential driving forces during these processes (VAN DEN BERG; BRUIN, 1981). Also the isotherms are useful to select appropriate packing systems and calculate moisture changes that usually occur during storage (MIRANDA et al., 2011; AL-MUHTASEB et al., 2002).

The objectives of the present work were: a) to provide experimental data for the sorption characteristics of fresh, blanched and osmotically dehydrated papaya slices of Formosa cultivar; b) model the sorption isotherm using six selected equations; c) determine differential thermodynamic properties and d) evaluate the application of the enthalpy–entropy compensation theory to the sorption phenomena.

2. Materials and methods

2.1. Samples preparing

Papaya of Formosa cultivar from São Paulo State (Brazil) were purchased at São José do Rio Preto Supplying Center (CEAGESP – São José do Rio Preto, São Paulo, Brazil). Ripe fruits with orange peels and reducing sugars content of approximately 8.89% ± 0.73 were used in the experiments.

Papayas were longitudinally cut in four pieces, the seeds were removed and each portion was sectioned with a manual cutter in cylinders with diameter of 3.6 cm. These pieces were cut in slices with 1.0 cm of thickness. The samples were kept and mixed in a plastic bag and randomly removed to be used in blanching and osmotic dehydration experiments.

2.2. Blanching

Papaya slices were put in a stainless basket and dipped in boiling water (98.3 °C at São José do Rio Preto-SP-Brazil) for 2 min. After this time, the samples were cooled in tap water for 1 min to be used in the experiments.

2.3. Osmotic dehydration experiments

Osmotic solutions were prepared with commercial sucrose (refined sugar) purchased in a local market; food grade pentahydrate calcium lactate in powder from PURAC Synthesis – Brazil; food grade L (+)-lactic acid in 85% solution from PURAC Synthesis – Brazil and distilled water.

Papaya slices were dehydrated in sucrose aqueous solution at 50% w/w with 1% w/w of calcium lactate and lactic acid 0.1 M for 120 min.

OD experiments were conducted into a stainless steel vat with an external jacket for water circulation at 27 °C. Osmotic solutions were continuously stirred using a 1.6 KW mechanical stirrer (Marconi, model MA-261 - Brazil) with a 10 cm diameter propeller and rotation at 1000 rpm. The ratio between sample and solution masses were at least 1:10. After processing time samples were removed from the solutions, washed in distilled water for 10 s and dried on absorbed paper.

2.4. Sorption isotherms

The gravimetric static method was employed to determine the equilibrium moisture of fresh, blanched and osmotically dehydrated papaya at 30, 50 and 70 ºC. Saturated salt solutions were prepared in glass flasks previously cleaned with formaldehyde (40%) to avoid microbial growth. The water activity ranged from 0.02 to 0.90, obtained using ten salts: NaOH, LiCl, CH_3COOK, $MgCl_2$, K_2CO_3, NaBr, $NaNO_2$, NaCl, KCl and $BaCl_2$. Fresh, osmo-dehydrated and blanched slices were cut in approximately 1 mm thickness pieces and two to five grams (in triplicate) were weighed in plastic baskets and placed on acrylic tripods arranged in the glass flasks to maintain the samples above the salt solutions. Each flask was hermetically closed and placed in a temperature-controlled incubator. A BOD (Biological Oxygen Demanding) incubator was used at 30 and 50 ^oC (sensor PT100, precision of ±¨0.1 and homogeneity of ± 1 ^oC). An air circulation oven was used at 70 ^oC (type J sensor, precision of ± 2 ^oC and homogeneity of ± 4 ^oC). The samples were weighed periodically until the weight (expressed on a dry basis) did not exceed 0.1% (0.001 g.g dry solids^{-1}). The total solids contents were then determined and the average equilibrium moisture contents related to the corresponding water activity.

Fresh, blanched and osmotically dehydrated papaya were characterized with respect to total solids and sugars content.

2.5. Analytical methods

The solids contents of the fresh, blanched and osmotically dehydrated samples were determined gravimetrically by drying to constant weight in a vacuum oven at 60 ^oC and 10 kPa. The reducing and total sugar contents were determined by oxy-reduction titration (WILLIAM, 1970).

2.6. Mathematical modeling

2.6.1. Sorption isotherms

BET, GAB, Halsey, Henderson, Oswin and Peleg models (Table 1) were fitted to the food system moisture sorption.

Table 1. Water sorption isotherm models.

Model	Equation	
BET	$X = \frac{X_m.C.a_w}{(1-a_w)(1+(C-1)a_w)}$	(1)
GAB	$X = \frac{X_m.C.K.a_w}{(1-K.a_w)(1-K.a_w+C.K.a_w)}$	(2)
Halsey	$X = \left[\frac{-A}{\ln(a_w)}\right]^{\frac{1}{B}}$	(3)
Henderson	$X = \left(\frac{\ln(1-a_w)}{-A}\right)^{\frac{1}{B}}$	(4)
Oswin	$X = A\left[\frac{a_w}{(1-a_w)}\right]^{B}$	(5)
Peleg	$X = A(a_w)^B + C(a_w)^D$	(6)

where *K*, *A*, *B*, *C* and *D* are constants; aw is the water activity; *X* represents the moisture content on a dry weight basis (g water·g dry matter^{-1}) and X_m is the monolayer of water (g water·g dry matter^{-1}).

2.6.2. Net isosteric heat of sorption

The net isosteric heat of water sorption (q_{st}) can be determined from Equation 7, which is derived from the Clausius – Clapeyron equation, applied to food and pure water (McMINN et al., 2004).

$$\left.\frac{\partial \ln(a_w)}{\partial(1/T)}\right|_X = -\frac{Q_{st}-\lambda}{R} = -\frac{q_{st}}{R} \quad (7)$$

where Q_{st} is the differential isosteric heat of sorption (kJ·mol^{-1}), λ is the molar enthalpy of vaporization of pure water (kJ·mol^{-1}), *R* is the gases constant (8.314 J·mol^{-1}·K^{-1}) and *T* the temperature (K). The net isosteric enthalpy is obtained by subtracting the value obtained for the corresponding equation for pure water.

The net isosteric heat of sorption is the slope (q_{st}/R) of the curve obtained by plotting the sorption isostere as $ln(a_w)$ *versus 1/T* for a specific moisture content of the material (TSAMI et al., 1990). This procedure is repeated for different moisture contents in order to

determine the dependence of q_{st} on the moisture content (X_{eq}). The method presents an overall error due to plotting graphical differentiation (KAYMAK-ETERKIN; GEDIK, 2004).

2.6.3. Enthalpy-Entropy Compensation Theory

The relation between the isosteric heat ($q_{st,}$ kJ·mol^{-1}) and the differential entropy (ΔS_d, kJ·mol^{-1}·K^{-1}) of sorption was given by:

$$\left(-\ln a_w\right)_X = \Delta H_{dif} / RT - \Delta S_d / R \qquad (8)$$

where ΔH_{dif} correspond to the net isosteric heat of sorption (q_{st}), T is temperature (in K) and R is the gases constant (8.314 J·mol^{-1}·K^{-1}).

The differential entropy can be found from the interception with Y-axis by plotting *ln(aw) versus 1/T* for a specific moisture content of the material (McMINN et al., 2004).

The compensation theory proposed a linear relationship between ΔH_{dif} and ΔS_d which is valid only if ΔH_{dif} *versus* ΔS_d curves show a linear proportion. Thus:

$$\Delta H_{dif} = T_\beta . \Delta S_d + \Delta G_B \qquad (9)$$

where ΔG_B is the Gibbs´ isokinetic energy, in kJ·mol^{-1} and T_β is the isokinectic temperature, in K, which represents the temperature where all the reactions occur at the same rate.

According to McMinn et al. (2004) to corroborate the compensation theory a statistical analysis test was proposed by Krug et al. (1976a, b). This involves a comparison of the isokinetic temperature (T_β) with the harmonic mean temperature (T_{hm}):

$$T_{hm} = \frac{n}{\sum_{i=1}^{n}\left(1/T\right)} \qquad (10)$$

The compensation theory only applies if $T_\beta \neq T_{hm}$.

2.7. Statistical methods

The efficiency of fit was evaluated based on the adjusted coefficient of determination (R^2) and on the mean relative modulus, P, defined by Equation 11 (LOMAURO et al., 1985).

$$P\left(\%\right) = \frac{100}{N}\sum_{1}^{n}\frac{\left|x^{\exp} - x^{calc}\right|}{x^{calc}} \qquad (11)$$

where x^{exp} represents the experimental values, x^{calc} the calculated values and N is the number of observations.

The statistical software Statistica 7.0 (StatSoft Inc., USA), which adjusts non-linear functions, was used to determine the adjustment constants of the isotherms.

3. Results and discussions

Table 2 presents sugar and moisture contents of fresh, blanched and osmotically dehydrated samples used in sorption experiments.

Table 2. Moisture content, in g·g wet matter^{-1}, and reducing and non-reducing sugars contents, in g·g of wet matter^{-1}, of fresh, osmotically dehydrated and blanched fruits.

Sample	Isotherm temperature	Reducing sugar	Non-reducing	Moisture content
Fresh	30 and 50 °C	0.0817 ± 0.0047	ND	0.8716 ± 0.0002
	70 °C	0.0962 ± 0.0100	ND	0.8554 ± 0.0006
Blanched	30 and 50 °C	0.0415 ± 0.0021	0.0262 ± 0.0045	0.8919 ± 0.0003
	70 °C	0.0353 ± 0.0004	0.0400 ± 0.0120	0.8857 ± 0.0006
OD	30, 50 and 70 °C	0.2423 ± 0.0102	ND	0.7970 ± 0.0010

ND: Non-detected.

Concerning the fresh fruits, it was observed that blanching slightly increased moisture content of the slices while osmotically dehydrated papayas presented a decrease of the initial moisture content due to the water loss and the sugar gain. It was not possible to detect the sucrose at fresh and osmotically dehydrated samples because of invertase activity in the papayas (CHAN JR.; KNOK, 1975). On the other hand, non-reducing sugar contents were found in blanched samples once the enzyme was inactivated. During blanching the losses of total sugar were approximately 20% of the fresh fruit sugar content.

Table 3 shows the best model fitting constants for fresh, blanched and osmotically dehydrated papaya. All the tested models presented R^2 values higher than 0.97 and, with minor exceptions, the mean relative modulus (P) was lower than 10%, which is an indicative of good fit for practical purposes (LOMAURO et al., 1985).

Peleg, GAB and Henderson models were the ones that better represented the hygroscopicity of fresh samples at 30, 50 e 70 °C, respectively, presenting R^2 higher than 0.99 e P lower than 5% for a wide range of water activity (0.02–0.9). In general GAB was the best model to represent the experimental data of water sorption of blanched samples (R^2 higher than 0.99 e P lower than 5% for all the temperatures), even Halsey, Henderson and Peleg equations had been better to fit the experimental data at 30, 50 and 70°C, respectively. For the osmotically

dehydrated papayas, GAB model showed the best fitting to the experimental data at all the studied temperatures (R^2 higher than 0.99 e P lower than 5.5% for all the temperatures).

The monolayer moisture content is the minimum moisture covering hydrophilic sites on the material surface, and it is a necessary data for achieving storage with minimum quality loss for long time. Furthermore, at this condition, the rates of spoilage reactions, except for oxidation of unsaturated fats, are minimal. Therefore it is a useful value defining an optimum moisture content for drying and storage stability of foods, and in the estimation of the surface area of a food. This value is particularly important in storage of fresh and processed papaya since below this level the water does not act as a solvent, being biologically inert (MOREIRA et al., 2008; ARÉVALO-PINEDO et al., 2006; AL-MUHTASEB et al., 2002).

As can be seen in Table 3, the values of X_m of GAB model were in the range of 0.05 to 0.13 g of water·g of dry solids^{-1}, which are within the reported values for fruits (MOREIRA et al., 2008; ARÉVALO-PINEDO et al., 2006; Lahsasni et al., 2004; MORAGA et al., 2004; HOSSAIM et al., 2001).

Figures 1, 2 and 3 present the equilibrium water contents experimentally determined at 30 °C, 50 °C and 70 °C compared with the values calculated according to the GAB equation (Equation 2).

Table 3. Model fitting constants and corresponding R^2 and P for fresh, blanched and osmotically dehydrated papaya, determined at 30, 50 and 70 °C.

Sample	**Model**	**T (°C)**	**R^2**	P **(%)**	**Parameters**					
					X_m	K	C	A	B	D
		30*	-	-	-			-		
	BET	50	0.9937	3.83	0.0795			1.7141		
		70	0.9905	2.91	0.0759			1.0187		
		30	0.9984	5.36	0.0518	1.1736	1.0851			
	GAB	50	0.9987	2.75	0.1069	0.9866	0.9685			
		70	0.9999	2.72	0.1011**	0.9736	0.5838			
		30	0.9798	39.21				0.1493	0.4476	
Fresh	Halsey	50	0.9904	7.16				0.0810	0.9390	
		70	0.9841	5.67				0.0895	0.7861	
		30	0.9695	59.22				1.9934	0.1832	
	Henderson	50	0.9903	7.74				2.8297	0.5746	
		70	0.9925	1.74				3.4868	0.6320	
		30	0.9764	81.68				0.0202	2.1307	
	Oswin	50	0.9941	3.43				0.0996	0.9111	
		70	0.9906	2.93				0.0768	0.9945	
		30	0.9998	4.71			39.9053	0.3977	1.6057	29.0317
	Peleg	50	0.9958	14.24			0.604	181.457	74.706	2.607

		70	0.9912	7.75			-0.5308	1.0502	2.7375	2.7369
		30	0.9969	4.77	0.1005			1.0426		
	BET	50	0.9936	5.72	0.0831			1.5136		
		70	0.9878	5.79	0.1143			0.4443		
		30	0.9970	4.90	0.1119	0.9859	0.8826			
	GAB	50	0.9963	4.02	0.1379	0.9491	0.5840			
		70	0.9905	4.28	0.0565	1.0557	1.8940			
		30	0.9927	4.69				0.1056	0.8218	
Blanched	Halsey	50	0.9876	8.49				0.0835	0.9401	
		70	0.9895	5.81				0.1026	0.7384	
		30	0.9932	10.61				2.7249	0.5896	
	Henderson	50	0.9983	2.56				2.7607	0.5565	
		70	0.9820	13.71				2.4651	0.4470	
		30	0.9969	4.73				0.1028	0.9895	
	Oswin	50	0.9946	5.61				0.1008	0.9201	
		70	0.9884	5.38				0.0719	1.1652	
		30	0.9769	38.68			-1.8319	2.7071	3.3695	3.3698
	Peleg	50	0.9976	5.01			0.6525	2.1754	19.7316	2.9219
		70	0.9909	3.59			0.3483	3.6540	15.9123	1.9857
		30	0.9822	30.04	0.3455			0.0936		
	BET	50	0.9972	4.19	0.0962			1.6392		

		70	0.9928	5.50	0.0910			1.1487		
		30	0.9981	5.46	0.0688	1.0641	4.3867			
	GAB	50	0.9979	3.06	0.1158	0.9799	1.1009			
		70	0.9932	4.93	0.1085	0.9804	0.8538			
		30	0.9880	14.47				0.1403	0.6291	
OD	Halsey	50	0.9932	8.01				0.0967	0.9351	
		70	0.987	8.24				0.0933	0.8594	
		30	0.9685	261.04				2.0094	0.3074	
	Henderson	50	0.995	7.79				2.5533	0.5772	
		70	0.9909	7.93				2.7298	0.5696	
		30	0.9832	36.25				0.0644	1.4535	
	Oswin	50	0.9977	3.82				0.1192	0.9136	
		70	0.9956	5.01				0.0941	1.0161	‘
		30	0.9983	36.27			19.3237	0.6744	2.4184	27.2874
	Peleg	50	0.9736	88.08			1.5274	-0.3730	3.9002	3.9001
		70	0.9718	100.91			-0.5312	1.4695	3.6083	3.6089

*Non-convergence

**Fitted with a fixed parameter

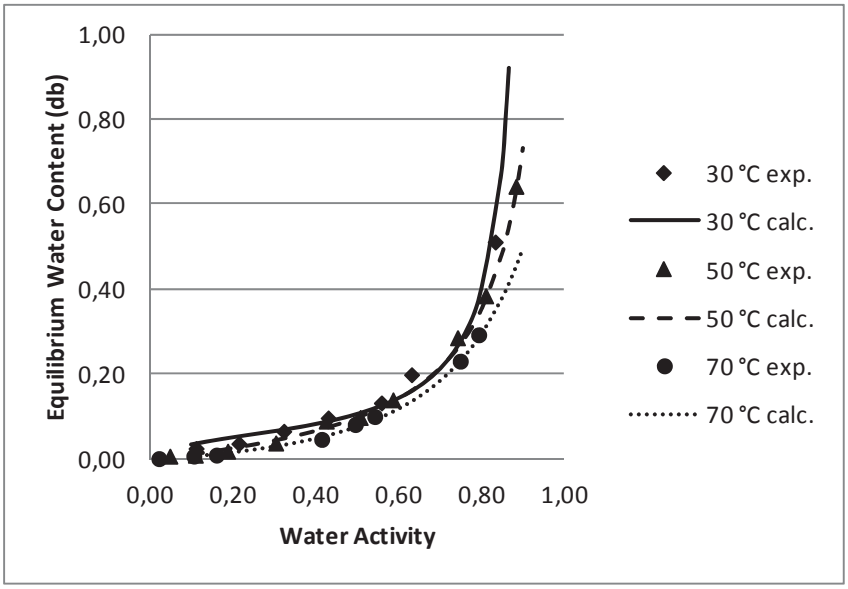

Figure 1. Experimental and calculated, according to GAB model, equilibrium water contents determined for the fresh papaya.

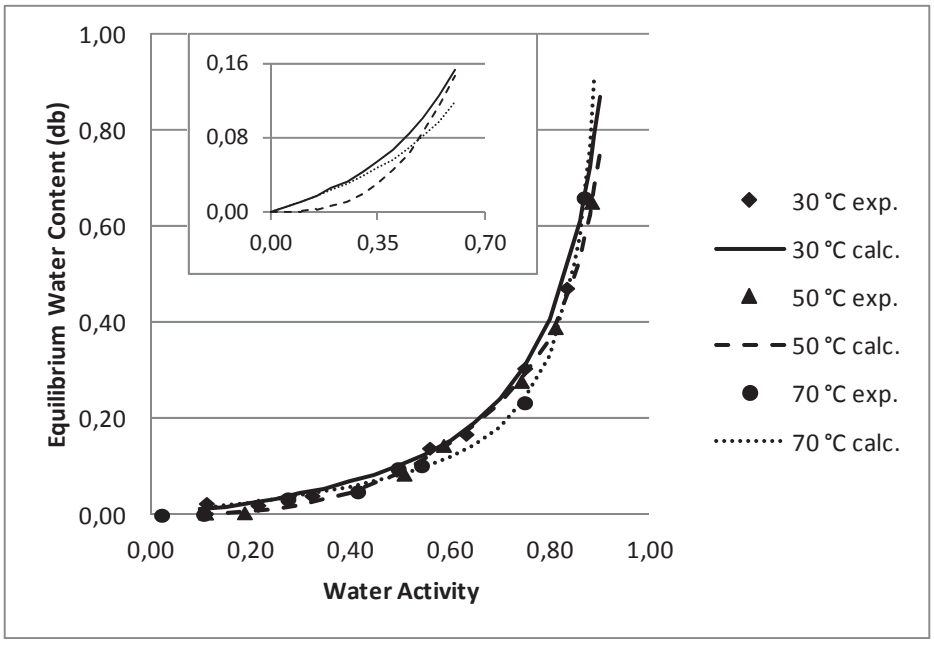

Figure 2. Experimental and calculated, according to GAB model, equilibrium water contents determined for the blanched papaya.

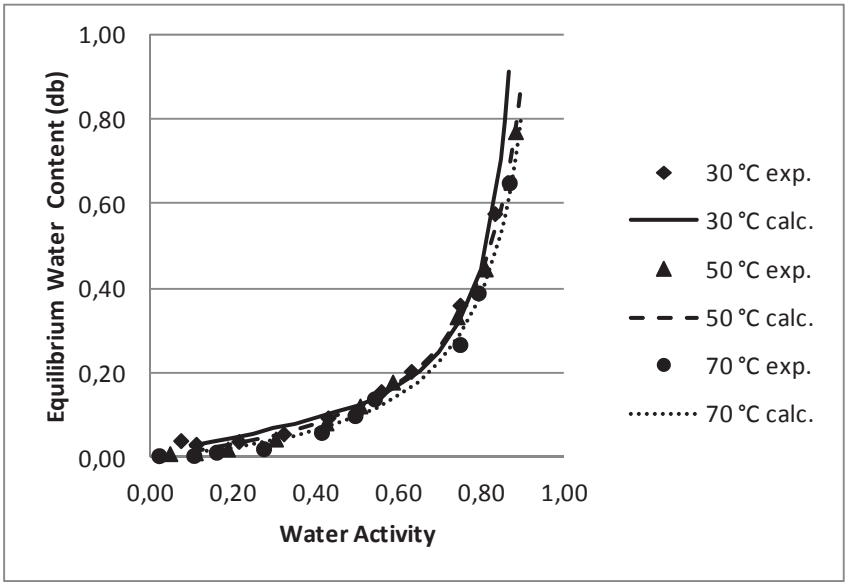

Figure 3. Experimental and calculated, according to GAB model, equilibrium water contents determined for the osmotically dehydrated papaya.

It was verified in Figures 1, 2 and 3 that the moisture sorption isotherms presented behavior characteristic of foods rich in soluble components (KAYMAK-ERTEKIN; GEDIK, 2004; AL-MUHTASEB et al., 2002). The higher the amount of sugars the less water available for physical-chemical reactions. Consequently, in agreement with the sugar content of the samples (Table 2), blanched papaya must be dried to the lowest levels of moisture content to be stable during storage, followed by fresh and osmo-dehydrated fruits (Figures 1, 2 and 3). The effect of temperature on the sorption isotherm is important since foods are exposed to a range of temperatures during storage and processing, and water activity changes with temperature for the same moisture content (GOULA et al., 2008).

The desorption isotherms of fresh and osmo-treated fruits (Figures 1 and 3) showed that at constant water activities the equilibrium moisture content values decreased with rise in temperature, following the same tendency as the majority of agricultural products (MOLINA FILHO et al., 2011; GONELI et al., 2010; YAN et al., 2008; AL-MUHTASEB et al., 2002; HOSSAIM et al., 2001). This fact could be explained by the activated state of water molecules at higher temperature, causing them to break away from the sorption sites, thus decreasing moisture content (GONELI et al., 2010; YAN et al., 2008; AL-MUHTASEB et al., 2002; HOSSAIM et al., 2001).

Isotherm curves of blanched samples showed an unusual behavior of the equilibrium moisture with temperature. At the lowest and the highest water activity ranges, the equilibrium moisture value at 70 °C isotherm was higher than the ones at 50 °C isotherm

(Figure 2). Blanching damaged the cellular structure, probably resulting in loss of selective permeability of plasmalema. Thus, blanched fruits behaved as a food with high sugar constituents and crossing over of desorption isotherms at greater moisture ranges is usually observed (ARÉVALO-PINEDO et al., 2006; AL-MUHTASEB et al., 2002).

Figure 4 shows the variation in the net isosteric heat of sorption (q_{st}) of fresh, blanched and osmo-dehydrated papaya slices as a function of moisture content.

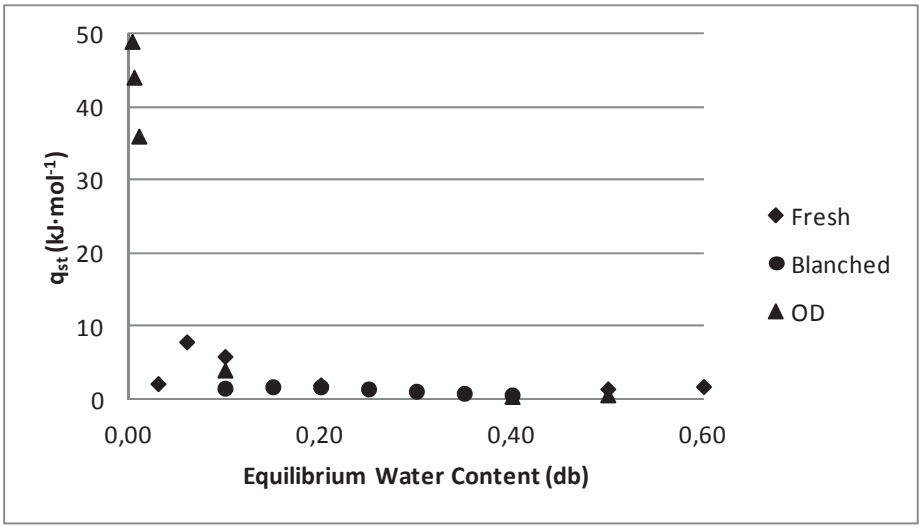

Figure 4. q_{st} variations of fresh, blanched and osmo-treated papaya slices as a function of moisture.

Figure 4 illustrates a progressive increase in the net isosteric heat of sorption (differential enthalpy) with decreasing moistures of the fresh, blanched and osmotically dehydrated papaya. At high water contents the heat of sorption approaches zero, meaning that q_{st} tends to the heat of vaporization of pure water, reflecting the fact that at higher moisture levels the strength of the water binding decreases (MOREIRA et al., 2010b; GOULA et al., 2008; McMINN et al., 2004; KAYMAK-ERTEKIN; GEDIK, 2004; AL-MUHTASEB et al., 2002).

Some authors asserted that the increase in the heat of sorption at low moisture was due to the existence of highly active polar sites on the surface of the food material which are covered with water molecules forming a monomolecular layer (MOREIRA et al., 2008; LAHSASNI et al., 2004 HOSSAIM et al., 2001; JOHNSON; BRENNAN, 2000). Moreover, the net isosteric heat of sorption of some fruits was found to be slightly negative at high moisture, due to the contribution of the endothermic dissolution of sugars in the sorbed water (KAYMAK-ERTEKIN; GEDIK, 2004), a phenomenon that was not observed in this study.

Sorption entropy is proportional to the number of available sorption sites at a specific energy level and can be calculated according to Equation 8. The changes in the differential entropy of fresh, blanched and osmotically dehydrated samples at different moisture are shown in Figure 5.

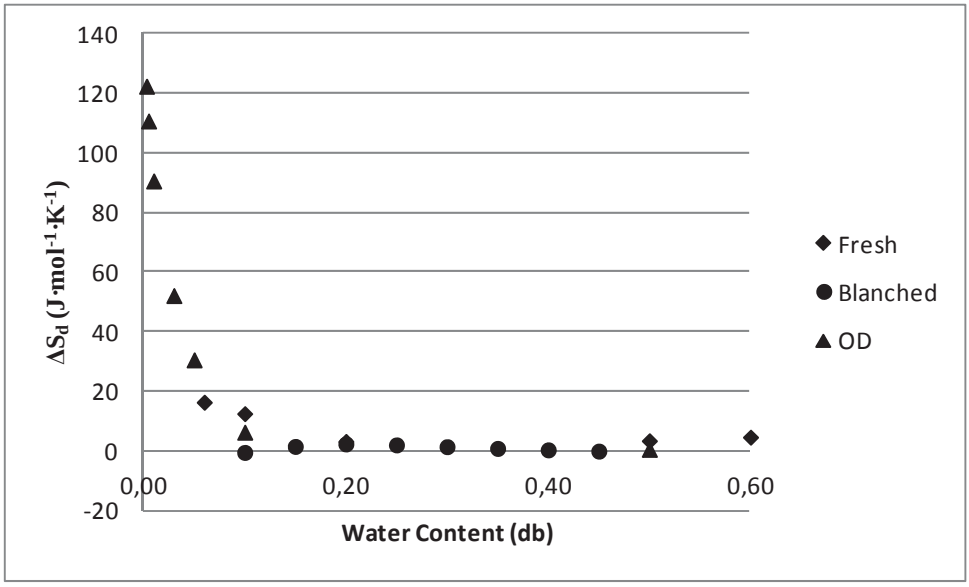

Figure 5. Variation of differential entropy with moisture of fresh, blanched and osmo-dehydrated papaya slices.

In Figure 5 it can be observed the strong dependence of differential entropy on moisture content with similar trend to the behavior exhibited by the differential enthalpy. These results are in agreement with the literature data determined for foods (GONELI et al., 2010; GOULA et al., 2008; MOREIRA et al., 2008).

A plot of differential enthalpy *versus* differential entropy of the samples showed a linear correlation and then the isokinetic temperatures were determined according to the Equation (9). Table 4 presents the isokinetic temperatures T_β, in K, and the respective determination coefficients (R^2).

The calculated harmonic mean temperature (T_{hm}) was 322.32 K. Since $T_\beta \neq T_{hm}$, the compensation theory could be applied to the data. Additionally, as $T_\beta > T_{hm}$ is satisfied, the processes can be characterized as enthalpy driven. In addition, the enthalpy-entropy compensation presented only one mechanism in the moisture range studied, which led to conclude that the tissue structure of the papaya did not change during moisture sorption (MOREIRA et al., 2008, DOMÍNGUEZ et al., 2007).

Table 4. Isokinetic temperatures T_β, in K, and determination coefficients (R^2) of plotting ΔH_{dif} *versus* ΔS_d of fresh, blanched and osmotically dehydrated papaya slices.

Samples	T_β	R^2
Fresh	500.36	0.94
Blanched	505.37	0.99
Osmotically dehydrated	393.19	0.99

3. Conclusions

Peleg, GAB and Henderson models were the ones that better represented the hygroscopicity of fresh samples at 30, 50 e 70 °C, in the range of water activity studied. The desorption of blanched samples were better represented by Halsey, Henderson and Peleg models at 30, 50 and 70 °C, respectively, but GAB model was also efficient. For the osmotically dehydrated papayas, GAB model also showed the best fitting of the experimental data at 30, 50 and 70 °C.

Blanched samples presented an unusual behavior related to the equilibrium moisture content with temperature: crossing over of 70 °C and 50 °C sorption isotherms was verified at low and high water activities.

The net isosteric heat of sorption of the samples decreased as moisture content increased. Differential entropy of the samples showed a strong dependence on moisture content.

The plot of differential enthalpy *versus* differential entropy data satisfied the enthalpy–entropy compensation theory and the sorption processes of the samples were characterized as enthalpy driven.

Acknowledgments

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CAPÍTULO IV

EFFECT OF BLANCHING AND COATING ON CONVECTIVE DRYING AND QUALITY OF PAPAYA (*Carica papaya*) SLICES

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Abstract

The aim of this work was to investigate drying characteristics of fresh, blanched and coated papaya slices of the Formosa cultivar and the effects of these processes on sample quality. Modeling the moisture effective diffusion coefficients presented a better fit when shrinkage was incorporated into the calculations. Light and transmission electron microscopy images revealed that blanching damaged cellular structures, thereby resulting in higher moisture diffusivity as compared with fresh fruits. Blanching negatively influenced color and vitamin C retention of dried papaya, which resulted in lower color parameters and vitamin C retention as compared with dried fresh papaya. The cellular arrangement of coated papaya slices was similar to that of fresh papaya slices. The drying of coated papaya slices resulted in higher moisture diffusivities than the drying of fresh fruit samples, due to the hydrophilic nature of the coating. Dried coated papaya slices retained approximately 95% vitamin C.

Keywords: Papaya, drying, blanching, coating, vitamin C, microscopic features

1. Introduction

Papaya is a popular, low cost fruit characteristic of tropical and subtropical regions, and it has a long harvest period, allowing it to be available throughout the year. As a result, papaya is an important source of provitamin A and vitamin C in addition to mineral salts (RODRIGUEZ-AMAYA, 1996; TACO, 2011). Harvest and postharvest losses of papaya are large with approximate losses of 30-40% in Brazil (DURIGAN et al., 2005; LUENGO et al., 2001), which has increased interest in preventing these losses, processing parts of the fruit, adding value to the final products.

Drying is one of the most common preservation methods. Drying aims to remove water from the food materials to prevent the growth of spoilage microorganisms, prevent the reproduction of spoilage microorganisms, slow down the action of enzymes and minimize many of the water-mediated deteriorative reactions, physical changes and chemical changes during food storage (DOYMAZ; GÖL, 2011; DOYMAZ, 2010; LEERATANARAK et al., 2006). As a result of physical, chemical and biochemical changes, quality degradation of food submitted to drying processes is a major concern. Therefore, to improve the quality of the products and, in some cases, to reduce energy consumption, most food products are usually submitted to a form of pretreatment prior to drying (GARCIA et al., 2007; LEERATANARAK et al., 2006; NIETO et al., 1998; MUJUMDAR, 1997).

Blanching is a pretreatment method that is used to arrest some physiological processes before the drying of vegetables and fruits and blanching is usually performed to prevent off-flavors and color changes resulting from enzymatic reactions and to decrease the initial microorganism load. Blanching has also been included as an additional step to enhance mass transport in the tissue (JAISWAL et al., 2012; DOYMAZ, 2010; GONZÁLEZ-FÉSLER et al., 2008). Despite its preserving advantage, blanching causes nutrient degradation, particularly vitamins, and loss of color (NEGY; ROY, 2001).

Traditionally, edible coatings have been used in the fresh-cut industry as a strategy to reduce the deleterious effects that minimal processing imposes on intact vegetable tissues, thereby contributing to the extension of the shelf-life of fresh-cut fruits by reducing moisture, solute migration, gas exchange, respiration reaction rates, oxidative reaction rates and physiological disorders (DUAN et al., 2011; ROJAS-GRAÜ et al., 2009, WONG et al., 1994). Recently, edible coatings have been extensively used prior to osmotic dehydration to minimize the uptake of osmotic solids (MITRAKAS et al., 2008; KHIN et al., 2006; MATUSKA et al., 2006).

As a pretreatment for the drying process, coating can protect against the oxidation of biologically active compounds. The barrier properties of coatings mostly depend on their composition and the method used for their fabrication. Coatings formed from polysaccharide materials, such as low methoxylated pectin, present a great oxygen barrier, especially under low moisture conditions, which is useful as a pretreatment for drying because it prevents the oxidation of nutritional compounds, thereby improving the quality of the dried product (ZHAO; CHANG, 1995; MCHUGH; KROCHTA, 1994).

Important changes in physicochemical and structural properties occur in vegetable and fruit tissues after processing. Thus, microscopic analysis can be used to address the structure

and ultrastructure changes produced at the cellular level to better understand the dehydration kinetics and effects of pretreatments (GONZÁLEZ-FÉSLER et al., 2008; NIETO et al., 2001).

One of these changes is vitamin C degradation. Vitamin C is an essential substance that is found mainly in fruits and vegetables and that can be easily degraded depending on many variables, such as pH, temperature and light, as well as the presence of enzymes, oxygen and metallic catalyzers (SANTOS; SILVA, 2008). The preservation of vitamin C after processing is indicative of improved food quality.

The objectives of this work were as follows: a) to determine the convective drying characteristics of fresh, blanched and pectin-coated papaya slices of the Formosa cultivar; b) to measure effective moisture diffusivities of fresh and pretreated fruits during drying; c) to evaluate the effect of pretreatments and drying processes on the color and retention of vitamin C of papaya slices and d) to clarify the effects of the processes on cell structure modifications.

2. Materials and Methods

2.1. Sample preparation

Papayas of the Formosa cultivar from São Paulo State (Brazil) were purchased at São José do Rio Preto Supplying Center (CEAGESP; São José do Rio Preto, SP, Brazil). Ripe fruits with orange peels and average reducing sugar contents of 4.87% ± 0.08 were used in the experiments.

Papayas were longitudinally cut into four pieces, and the seeds were removed. Each portion was sectioned into cylinders (diameter of 3.6 cm) with a manual cutter. These pieces were then cut into slices with a thickness of 0.9 cm. Coated slices had an average thickness of approximately 0.96 cm. The samples were kept and mixed in a plastic bag until the slices were randomly removed to be used in the experiments.

2.2. Blanching

Papaya slices were put in a stainless steel basket and dipped in boiling water at a temperature of 98.3 °C (São José do Rio Preto, SP, Brazil) for 2 min. After this time, the samples were cooled in tap water for 1 min.

2.3. Edible coating application

Low methoxylated amidated pectin (GRINDSTED® LA 210; degree of methoxylation of 0.34; degree of amidation of 0.17; Danisco, Brazil) was used to coat the fresh papaya slices. A 2% (w/w) pectin solution was prepared at 70 °C and applied to the surface of the samples at 40 °C by immersion for 1 min. Gelling was activated with subsequent placement of samples in a 2.8% (w/w) aqueous solution of food grade calcium lactate pentahydrate (PURAC Synthesis, Brazil) for 30 s. To remove excess coating, samples were washed in distilled water for 30 s.

The average mass of the pectin coating was determined (0.62 g) and used to calculate the initial thickness of the coat by considering the solution density to be approximately 1000 $kg \cdot m^{-3}$.

2.4. Convective drying

Fresh, blanched and coated papaya slices were dried in convective driers at 60 and 70 °C with an air velocity of 1.0 $m.s^{-1}$ until a water activity minor than 0.6 was achieved. The following experiments were performed at each temperature: raw papaya (control) and blanched or coated fruits were placed into driers and dehydrated in parallel (fresh *versus* blanched; fresh *versus* coated) to allow a control for each batch. The air flowed parallel to the bed, which consisted of three wire nets. Approximately 330 g of fresh and treated samples were dried. Samples were weighted every 20 min during the first hour of drying and every 30 min during the remaining time. At the weighting time, the trails inside the driers were rotated. After drying time, three slices of each sample were kept into the driers for more 2-3 h to determine the equilibrium water content. Total solid contents, reducing sugar contents, total sugar contents, water activity, color and vitamin C contents were determined in fresh and dried (treated or untreated) fruits. The shrinkage of the dried papayas was calculated.

2.5. Microscopic observations

Conventional microscopy techniques were used to reveal structural changes in the papaya slices. Thin parallelepipeds from the internal zone of the sample were fixed in a 4% (w/w) glutaraldehyde solution overnight at room temperature, postfixed in a 1% (w/w) OsO_4 solution for 2 h and dehydrated in a graded acetone series prior to being embedded in Araldite 502 resin. For light microscopy (LM), the sections were stained with toluidine blue in a borax solution, and they were examined with an Olympus BX60 light microscope (Olympus, Hamburg, Germany) equipped with an Olympus DP71 camera. For transmission electron

microscopy (TEM), ultrathin sections were stained with uranyl acetate and lead citrate and the sections were examined under a Zeiss EM906 transmission electron microscope (Zeiss, Cambridge, England) at 80 kW.

The images were digitized using Image-Pro Plus software (version 6.0; Media Cybernetics Incorporation, Singapore) and the internal diameters of the cells were measured from light micrographs.

2.6. Analytical methodologies

The solid contents of fresh, blanched and coated slices were gravimetrically determined in triplicate by drying the samples in a vacuum oven at 60 °C and 10 kPa until a constant weight was achieved. The reducing and total sugar contents of fresh and blanched fruits were determined in triplicate by an oxidation-reduction titration (WILLIAM, 1970). Water activity of the samples was measured in triplicate at 25 °C in a hygrometer (AW SPRINT; NOVASINA, Switzerland). The color of fresh, blanched and coated fruits either dried or not dried was evaluated in six replicates using a Colorflex spectrophotometer (HunterLab, USA) and version 4.10 of the Universal software with the following settings: illuminant D65, observer at 10° and reading of the absolute values of L*, a* and b*.

The vitamin C content of the samples was determined in duplicate using the modified method described by Benassi and Antunes (1988). Samples (25 g) were homogenized in 50 mL of extractor solution (oxalic acid at 2%; w/w) using Turratec equipment (TECNAL, TE-102 model) for 1 min. An aliquot (20 g) was volumetrically diluted with the extractor solution to 50 mL. Ten milliliters of the diluted solution was titrated with 2.6-dichlorophenolindophenol. The calculations of vitamin C contents of dried papaya slices were based on the total solids contents of the samples. The calculations and the retention of vitamin C related to the fresh samples are presented at Appendix II.

The density of fresh and dried papayas was determined in four replicates by a volume dislocation technique using a Gehaka kit (GEHAKA, BK-300 model). Methanol was used as the displacement fluid.

2.7. Calculations

2.7.1. Shrinkage

To calculate the shrinkage of fresh and dried samples, the volume variation was expressed by Equation 1 as follows:

$$\frac{V^a - V^0}{V^0} = \left(\frac{m^a}{\rho^a} - \frac{m^0}{\rho^0}\right) \cdot \left(\frac{m^0}{\rho^0}\right)^{-1} \quad (1)$$

where V is the volume; *0* indicates control and *a* indicates the sample condition as either, fresh dried (FD), blanched dried (BD) or coated dried (CD).

2.7.2. Effective diffusion coefficients

The effective diffusion coefficients of moisture (D_m) were determined according to Fick's Law applied to an infinite slab. The diffusion model has been applied to the drying of biological materials by changing the fractional contents to express the moisture on a dry basis (db) (GARCIA et al., 2007). The analytical solution of the following diffusion equation (Equation 2) has been previously described by Crank (1975):

$$\boldsymbol{X} = \left(\frac{\overline{X}(t) - X^{eq}}{X^0 - X^{eq}}\right) = \frac{8}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{(2n-1)^2} \exp\left[-(2n-1)^2 \frac{\pi^2 D_m t}{z^2}\right] \quad (2)$$

where *eq* indicates equilibrium; *0* indicates initial moisture (at t = 0); $\boldsymbol{X}$ is the fractional or residual moisture, dry basis (dimensionless); X represents moisture (db); $\overline{X}$ is the average moisture (db) at time t; D_m is the effective diffusion coefficient of moisture ($m^2.s^{-1}$) and z is the thickness of the samples.

The coefficients in Equation 2 were determined by considering z as the following parameters: a) initial thickness of the slices (0.9 cm), b) an average thickness calculated using the initial and final measurements and c) a variable thickness assuming z as a linear function of $\boldsymbol{X}$ (Table 1) to incorporate shrinkage in the analytical solution. Table 1 presents the linear functions used in the calculations.

Table 1. Linear functions used in the calculations to incorporate shrinkage in the solutions.

Sample after drying	60 °C	70 °C
Fresh	$z = 0.463\boldsymbol{X} + 0.436$	$z = 0.408\boldsymbol{X} + 0.491$
Blanched	$z = 0.519\boldsymbol{X} + 0.380$	$z = 0.487\boldsymbol{X} + 0.412$
Fresh	$z = 0.407\boldsymbol{X} + 0.492$	$z = 0.461\boldsymbol{X} + 0.438$
Coated	$z = 0.421\boldsymbol{X} + 0.538$	$z = 0.453\boldsymbol{X} + 0.506$

For long drying times, the terms in Equation 2 were expected to converge rapidly, so only a few terms were required to perform the calculations (DOYMAZ; GÖL, 2011).

2.8. Statistical analysis and fitting

The data were statistically analyzed by an analysis of variance (ANOVA) and Tukey's test at a 5% significance level using Excel 2007 (Microsoft, USA).

Effective diffusion coefficients were calculated from the experimental data according to Equation 2 using Statistica 7.0 software. The fitting efficiency was evaluated by the determination coefficient (R^2) and the mean relative error root square (*RRMS*). RRMS was calculated with Equation 3 as follows:

$$RRMS\ (\%) = 100\left\{\frac{1}{N}\sum_{n=1}^{N}\left[\frac{\left(x^{calc} - x^{\exp}\right)}{x^{calc}}\right]\right\}^{1/2} \quad (3)$$

where x^{calc} represents moisture content on a dry basis and was calculated according to Equation 2; x^{exp} is the experimental value and N represents the number of observations.

3. Results and Discussion

3.1. Effect of blanching and coating on convective drying of papaya slices

Tables 2 and 3 show the moisture content (wet basis) and water activity of fresh, blanched and coated fruits before and after convective drying at 60 and 70 °C. The shrinkage after convective drying at both temperatures is also shown in Tables 2 and 3.

Table 2. Moisture content (w, %, wet basis) and water activity (a_w) of fresh and blanched papaya slices before and after convective drying at 60 and 70 °C; and shrinkage (%) after drying of fresh and blanched fruits.

60 °C					
	Before drying		**After drying**		
Sample	***w***	***a_w***	***w***	***a_w***	**Shrinkage**
Fresh	86.85 ± 0.04	0.987 ± 0.001	17.39 ± 0.58	0.575 ± 0.000	88.62
Blanched	89.17 ± 0.11	0.989 ± 0.000	14.99 ± 0.91	0.614 ± 0.000	92.46
70 °C					
	Before drying		**After drying**		
Sample	***w***	***a_w***	***w***	***a_w***	**Shrinkage**
Fresh	88.18 ± 0.06	0.987 ± 0.001	22.51 ± 3.20	0.635 ± 0.002	83.70
Blanched	89.83 ± 0.07	0.989 ± 0.000	23.43 ± 2.73	0.793 ± 0.002	90.37

The first experiment compared the drying of fresh and blanched samples at 70 °C (Table 2). The moisture of these samples was high and mold growth on the surface of the blanched samples was observed after 15 days. Thus, lower moisture (approximately 15%; wet basis), which is related to lower a_w, were intentionally achieved in subsequent experiments (Tables 2 and 3). Raw papaya presented similar characteristics in experiments with each treatment. The fresh samples used in the preblanched experiments had lower moisture contents and water activities before drying than fresh fruits used in the precoated assays (Tables 2 and 3).

Table 3. Moisture content (*w*, %, wet basis) and water activity (a_w) of fresh and coated papaya slices before and after convective drying at 60 and 70 °C; and shrinkage (%) after drying of fresh and coated fruits.

60 °C					
	Before drying		**After drying**		
Sample	***w***	***a_w***	***w***	***a_w***	**Shrinkage**
Fresh	90.35 ± 0.03	0.992 ± 0.001	13.24 ± 1.05	0.510 ± 0.009	83.58
Coated	91.89 ± 0.01	ND	12.52 ± 0.17	0.502 ± 0.004	82.32
70 °C					
	Before drying		**After drying**		
Sample	***w***	***a_w***	***w***	***a_w***	**Shrinkage**
Fresh	91.23 ± 0.04	0.991 ± 0.000	13.24 ± 0.86	0.565 ± 0.006	88.45
Coated	92.37 ± 0.01	ND	15.24 ± 0.82	0.575 ± 0.001	85.28

ND: non-determined.

Blanched samples showed an increase in initial moisture content (Table 2 and Figure 1). As verified by other researchers (GONZÁLEZ-FÉSLER et al., 2008; NIETO et al., 1998), steam-blanched samples showed an increase in initial moisture content due to a partial disruption of cells and consequent absorption of water from the cooling medium used after blanching. Coated papaya slices also had higher moisture contents than the fresh fruit slices (Table 3 and Figure 2). After drying, blanched samples presented high water activities even though low values of moisture were present, which was related to the loss of solids during pretreatment (Table 2).

The drying curves of moisture content *versus* drying time are presented in Figures 1 and 2 where the effects of blanching and coating on the drying time of papaya slices are shown.

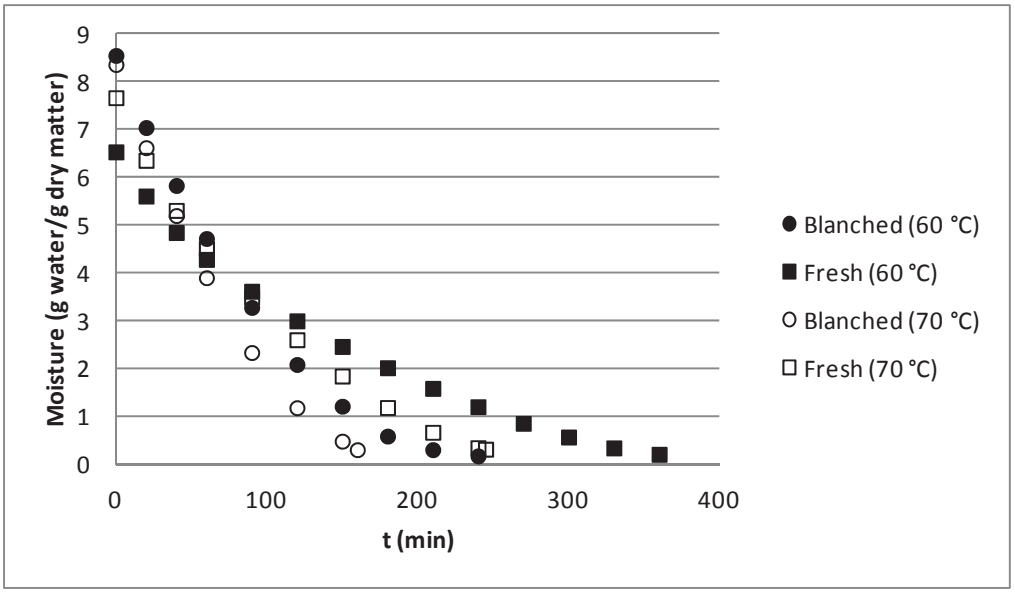

Figure 1. Variations of moisture with drying time of fresh and blanched papaya slices at 60 and 70 °C.

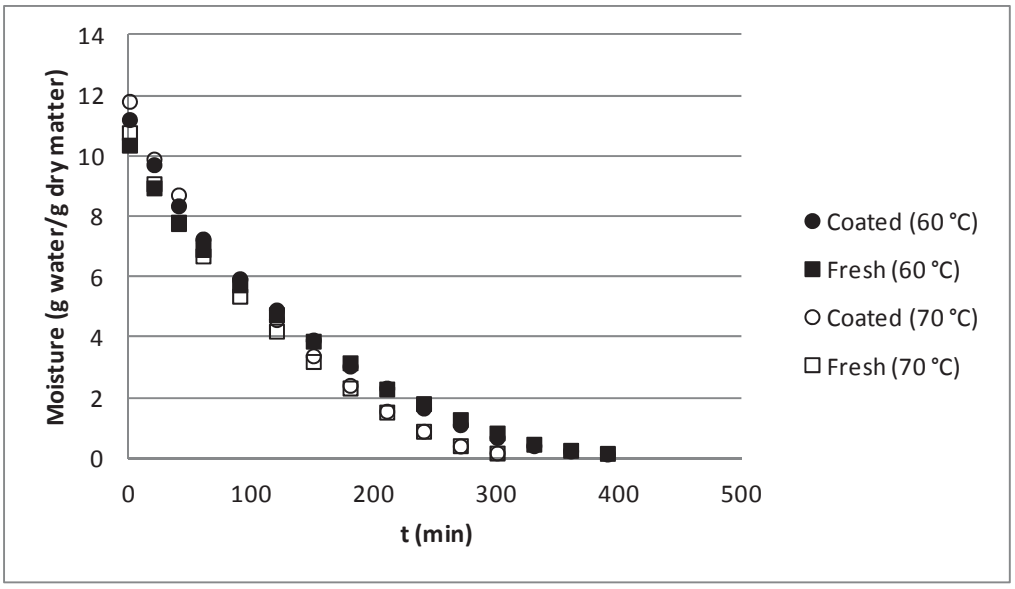

Figure 2. Variations of moisture with drying time of fresh and coated papaya slices at 60 and 70 °C.

Moisture content decreased continuously with drying time. The drying process of fresh and pretreated papayas occurred in the range of the falling-rate period, which indicated that internal diffusion was the dominant physical mechanism governing the moisture movement in the samples during drying. Similar results were obtained by Doymaz (2010) and El-Aouar et al. (2003).

According to the results shown in Figures 1 and 2, the pretreatments and drying air temperature had a significant effect on the moisture content of the papaya slices. The blanched samples dried faster than the unblanched samples as indicated by the moisture content curves of the blanched samples being below the curves of the unblanched samples. Blanching reduces the resistance to moisture transport, thereby increasing the drying rate, which has been demonstrated in studies using eggplants (DOYMAZ; GÖL, 2011), apples (DOYMAZ, 2010) and potatoes (LEERATANARAK et al., 2006).

Figure 2 shows that the application of edible coating did not interfere in the drying behavior of the fruits because the moisture content curves of fresh and coated papaya slices were overlapped.

Tables 4 and 5 show the effective diffusion coefficients of water calculated according to Equation 2 and considering *z* as the following parameters: a) initial thickness, b) average thickness (average of initial and final state) and c) variable thickness (a linear function of water content, Table 1). In both the second and third (b and c) diffusivity determinations, the thickness (*z*) was estimated considering similar shrinkage in all dimensions. Fick´s Law better predicted the water content when variable thickness was considered. Good fitting was obtained with this assumption because the values of R^2 were higher than 0.99 and the *RRMS* values were lower than 16% (Tables 4 and 5). Incorporating shrinkage in the calculations enhanced the fitting and resulted in lower coefficient values than the other calculation methods.

Drying time decreased with increasing drying temperatures (Figures 1 and 2). Additionally, the moisture diffusivities of fresh, blanched and coated papaya slices were higher at the 70 °C drying temperature (Tables 4 and 5). Blanched fruits presented higher diffusivities than fresh samples (Table 4) because of softening caused by blanching, which facilitates water removal (DOYMAZ; GÖL, 2011; DOYMAZ, 2010; GONZÁLEZ-FÉSLER et al., 2008). The effective moisture diffusion coefficients of coated papayas were higher than the coefficients of fresh papayas (Table 5), due to the strong hydrophilic nature of the pectin (MCHUGH; KROCHTA, 1994).

Table 4. Effective diffusion coefficients of moisture ($D_m \times 10^{10}$, in $m^2 \cdot s^{-1}$), calculated according Equation 2, considering initial, average and variable thickness (Table 1) and equilibrium moisture contents (X^{eq}, in %, dry basis) of fresh and blanched papaya slices after convective drying at 60 and 70 °C.

60 °C								
	Fresh				**Blanched**			
	Initial thickness	**Average thickness**	**Variable thickness**	X^{eq}	**Initial thickness**	**Average thickness**	**Variable thickness**	X^{eq}
D_m	7.85	4.35	3.15		12.81	6.48	4.43	
R^2	0.96	0.96	0.99	9.27 ± 0.35	0.94	0.94	0.99	5.31 ± 0.17
RRMS (%)	33.62	33.61	11.94		42.44	42.44	13.57	
70 °C								
D_m	10.76	6.33	4.91		15.78	8.49	7.65	
R^2	0.95	0.95	0.99	5.50 ± 0.15	0.94	0.94	0.99	3.57 ± 0.07
RRMS (%)	35.16	35.16	15.31		38.09	38.09	11.28	

Table 5. Effective diffusion coefficients of moisture ($D_m \times 10^{10}$, in $m^2 \cdot s^{-1}$), calculated according Equation 2, considering initial, average and variable thickness (Table 1) and equilibrium moisture contents (X^{eq}, in %, dry basis) of fresh and coated papaya slices after convective drying at 60 and 70 °C.

60 °C								
	Fresh				**Coated**			
	Initial thickness	**Average thickness**	**Variable thickness**	X^{eq}	**Initial thickness**	**Average thickness**	**Variable thickness**	X^{eq}
D_m	7.95	4.81	3.60		9.69	5.91	4.49	
R^2	0.96	0.96	0.99	7.17 ± 0.06	0.96	0.96	0.99	9.77 ± 0.24
RRMS (%)	39.77	39.77	22.77		43.03	43.03	27.97	
70 °C								
D_m	9.51	5.27	3.83		10.94	6.33	4.80	
R^2	0.95	0.95	0.99	7.76 ± 0.13	0.95	0.95	0.99	6.61 ± 0.03
RRMS (%)	20.87	20.87	19.77		39.70	39.70	22.79	

3.2. Effect of processing on quality of papaya slices

3.2.1. Color

The applied blanching, coating and drying processes promoted color changes in the fruit surface. Tables 6 and 7 show the average and standard deviation of the lightness (L*), redness (a*) and yellowness (b*) color parameters obtained for fresh, blanched, coated and dried fruits.

Blanching had a severe effect on the color of fresh fruits resulting in significantly decreased L*, a* and b* values (Table 6). Other researchers have found similar results for cabbage and Brussel sprouts after blanching (JAISWAL et al., 2012; VIÑA et al., 2007). The highest retention of color relative to fresh papayas was achieved with the coating pretreatment (Table 7).

Non-enzymatic browning, isomerization and/or oxidation of color pigments, such as carotenoids, during drying were considered to be the causes of the color changes in the papaya slices. After drying, blanched fruits became significantly darker as shown by significant decreases in the a* and b* parameters independent of the air drying temperature (Table 6).

Considering the fresh-coated experiments, fresh fruits became slightly lighter after drying at 60 °C. On the other hand, coated papaya slices had significant increases in the a* and b* parameters after drying at 60 °C, which indicated that the color of the slices changed to a more vivid orange (Table 6). After drying at 70 °C, the color parameters of fresh and coated papaya slices did not change, with an exception for the b* values of the coated samples, which were significantly higher. These non-significant changes may be a result of decreased oxygen exposure of the fruits as a result of lower drying times at 70 °C (Table 6).

As expected, pectin coat presented a protective effect on color pigments of papaya during drying, due to the selective permeability of the coat which avoided pigments oxidation. Ahmed et al. (2002) verified that color changes are related to carotenoids degradation during thermal treatment of papaya puree. Thus, the results suggested that pectin coat protected the major carotenoids in papaya during drying, which are typically red-orange pigments (KIMURA et al., 1991).

Table 6. L*, a* and b* parameters of fresh and blanched papaya slices before and after convective drying at 60 and 70 °C.

	60 °C					
	Before drying			After drying		
Sample	**L***	**a***	**b***	**L***	**a***	**b***
Fresh	50.63 ± 2.53[a]	39.04 ± 1.82[a]	47.81 ± 2.93[a]	53.24 ± 2.45[b]	33.61 ± 0.60[b]	40.17 ± 0.70[b]
Blanched	36.05 ± 0.64[a]	34.53 ± 1.75[a]	37.63 ± 2.90[a]	31.48 ± 2.14[b]	30.74 ± 0.46[b]	20.70 ± 2.10[b]
	70 °C					
	Before drying			After drying		
Sample	**L***	**a***	**b***	**L***	**a***	**b***
Fresh	52.03± 2.92[a]	37.29 ± 3.81[a]	44.00 ± 4.03[a]	51.55 ± 0.73[a]	35.20 ± 2.30[a]	43.24 ± 0.84[a]
Blanched	36.04 ± 0.59[a]	33.29 ± 0.10[a]	39.37 ± 0.32[a]	32.32 ± 1.46[b]	29.93 ± 0.87[b]	20.67 ± 1.88[b]

Different letters for the same parameter in the same line indicate statistically significant differences at $p < 0.05$.

Table 7. L*, a* and b* parameters of fresh and coated papaya slices before and after convective drying at 60 and 70 °C.

	60 °C					
	Before drying			After drying		
Sample	**L***	**a***	**b***	**L***	**a***	**b***
Fresh	54.88 ± 2.11[a]	26.54 ± 2.16[a]	40.94 ± 2.22[a]	60.62 ± 2.65[a]	28.72 ± 1.50[a]	44.98 ± 3.95[a]
Coated	58.36 ± 1.80[a]	21.03 ± 1.09[a]	39.23 ± 3.51[a]	56.01 ± 2.71[a]	30.74 ± 0.46[b]	47.23 ± 4.47[b]
	70 °C					
	Before drying			After drying		
Sample	**L***	**a***	**b***	**L***	**a***	**b***
Fresh	53.28 ± 1.59[a]	28.55 ± 1.07[a]	37.20 ± 1.38[a]	55.32 ± 1.68[a]	30.03 ± 1.12[a]	40.60 ± 1.14[a]
Coated	53.54 ± 0.72[a]	26.45 ± 1.09[a]	33.21 ± 2.24[a]	49.98 ± 1.63[a]	24.59 ± 0.24[a]	39.92 ± 0.74[b]

Different letters for the same parameter in the same line indicate statistically significant differences at $p < 0.05$.

3.2.2. Vitamin C

The effects of the pretreatments and drying temperatures on the degradation of vitamin C are shown in Tables 8 and 9.

Blanching caused losses of vitamin C less than 10% (Table 8), which may be related to the vitamin C heat sensibility and to the vitamin leaching during immersion of the samples in water. However, the mass variation was not determined before and after blanching. Consequently, only water gain was considered during the blanching process as the calculation was based on the moisture content. If the solid losses had been considered, retention during blanching would be approximately 80%. Considering the water loss, vitamin C losses after drying may reach 30% of the vitamin C content found in fresh papayas. Negi and Roy (2001) obtained 20% losses of ascorbic acid during blanching of carrots.

The influence of temperature on the degradation of vitamin C during hot air drying was clear. Increasing temperatures increased the loss of vitamin C, which was similar to previously reported findings (SANTOS; SILVA, 2008; NEGY; ROY, 2001).

Coating fresh papaya slices with pectin avoided ascorbic acid degradation during air drying (Table 9). The retention of vitamin C in coated samples reached 95% as compared with 83-87% in fresh fruits. Because of the permeability properties of the pectin coat, the coat was effective as a barrier because it minimized the contact between oxygen and papaya slices during drying.

Table 8. Vitamin C contents, in mg/100 g of sample, of fresh and blanched papaya slices before and after drying at 60 and 70 °C and retention of the referred vitamin related to the fresh sample after drying.

	Before drying		After drying		Before drying		After drying	
	60 °C				70 °C			
	Vitamin C	Retention (%)	Vitamin C	Retention (%)	Vitamin C	Retention (%)	Vitamin C	Retention (%)
Fresh	64.97 ± 1.05	-	378.64 ± 1.56	92.74	58.23 ± 1.01	-	352.27 ± 1.03	92.29
Blanched	51.69 ± 0.03	96.60	350.19 ± 0.36	83.35	45.10 ± 4.33	90.01	301.81 ± 1.90	80.02

Table 9. Vitamin C contents, in mg/100 g of sample, of fresh and coated papaya slices before and after drying at 60 and 70 °C and retention of the referred vitamin related to the fresh sample after drying.

	Before drying	After drying		Before drying	After drying	
	60 °C			70 °C		
	Vitamin C	Vitamin C	Retention (%)	Vitamin C	Vitamin C	Retention (%)
Fresh	44.29 ± 1.69	330.86 ± 8.19	83.03	55.61 ± 1.00	480.52 ± 27.97	87.30
Coated	ND	382.4 ± 3.71	95.17	ND	510.79 ± 5.82	95.00

ND: Non-determined.

3.2.3. Microscopic features

Changes produced at the cell structure level in the fresh fruit tissues subjected to blanching, coating and drying were examined by microscopy techniques as shown in Figures 3 and 4.

Figures 3A and 3B show LM images of fresh tissue cells (control) and intercellular spaces arranged in a net-like pattern. The cells had a rounded and turgid shape (Table 10) with consistent cell wall structures. The large amount of cell volume was occupied by the central vacuole. The cytoplasm, which was bound by the plasmalemma and tonoplast, was present as a thin layer lining the cell surface where the nucleus of the fresh cell was observed. Various shapes and sizes were verified for the intercellular spaces. A well-defined middle lamella cemented adjacent cells.

Figure 3C shows TEM images of stained cell walls with a clear longitudinal fibrillar pattern and with a greater intensity toward the margin and in the central zone of the middle lamella, which was intact. The arrangement of cytoplasm was marginal, and the tonoplast and plasmalemma appeared intact. Some cytoplasm content with lipid inclusions are shown in the detail of Figure 3C.

Blanching of the tissues resulted in cells with regular shape but with folded cell walls (Figure 3D and Table 10). Breakage of the tonoplast and plasmalemma resulted from the heat treatment (Figures 3D and 3E). As compared with fresh tissue, myofibrillar disorganization of the cell wall was present in the blanched tissue, which had an optical density slightly lower than that of raw papaya (Figure 3E). Many points of solubilization of the chelator-soluble pectin were observed in the middle lamella (Figures 3D and 3E), which is the substance that contributes most to cell adhesion and firmness. According to the microscopic images, the middle lamella may become solubilized during blanching in hot water. Figures 3D and 3E show that blanching caused damage to the cell structure with membrane breakage and partial solubilization of the middle lamella, which resulted in high moisture diffusivity. Similar results have been found for apples (GÓNZALEZ-FÉSLER et al., 2008; NIETO et al., 1998) and plums (NUNES et al., 2008).

Coated samples presented a cellular arrangement similar to fresh samples (Figure 3F and Table 10). However, dissolution of some parts of the middle lamella was verified.

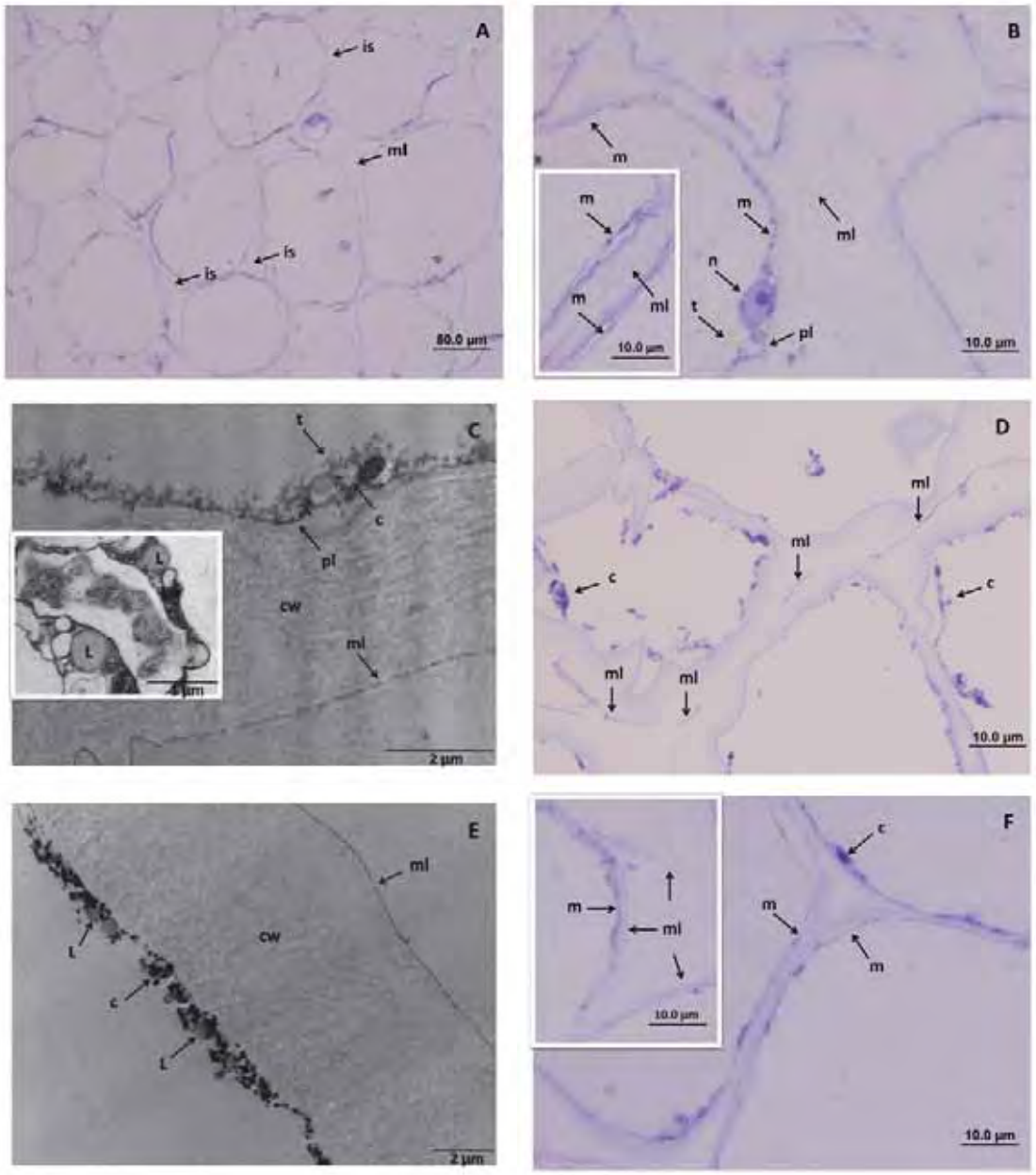

Figure 3. Light (A, B, D and F) and TEM (C and E) micrographs from fresh (A-C), blanched (D-E) and coated (F) papaya tissues. c: cytoplasm; cw: cell wall; is: intercellular space; L: lipid inclusions; m: membranes (plasmalema plus tonoplast); ml: middle lamella; n nucleus; pl: plasmalema; t: tonoplast.

The cell walls of fresh samples were greatly deformed and folded after drying (Figures 4A and 4B). As compared with fresh samples, dried fresh tissues had lower optical density. Disorganization of the cell wall arrangement in dried fresh tissues was verified (Figures 3C and 4B), and some poor residues of the middle lamella were present in these tissues. Intense rupturing of cellular membranes occurred in all the visualized cells, which caused the intracellular membranous content (referred as intracellular content) to spread out (Figure 4B).

Figure 4 shows LM and TEM images of coated samples after drying, which were similar to images of fresh fruits after drying. Microscopic images of dried coated samples did not show a protective effect of the coating against tissue structural changes. However, the coating provided a protective effect for nutritional compounds during drying because the redness and yellowness increased compared with the fresh samples and the retention of vitamin C in coated fruits reached 95% after drying.

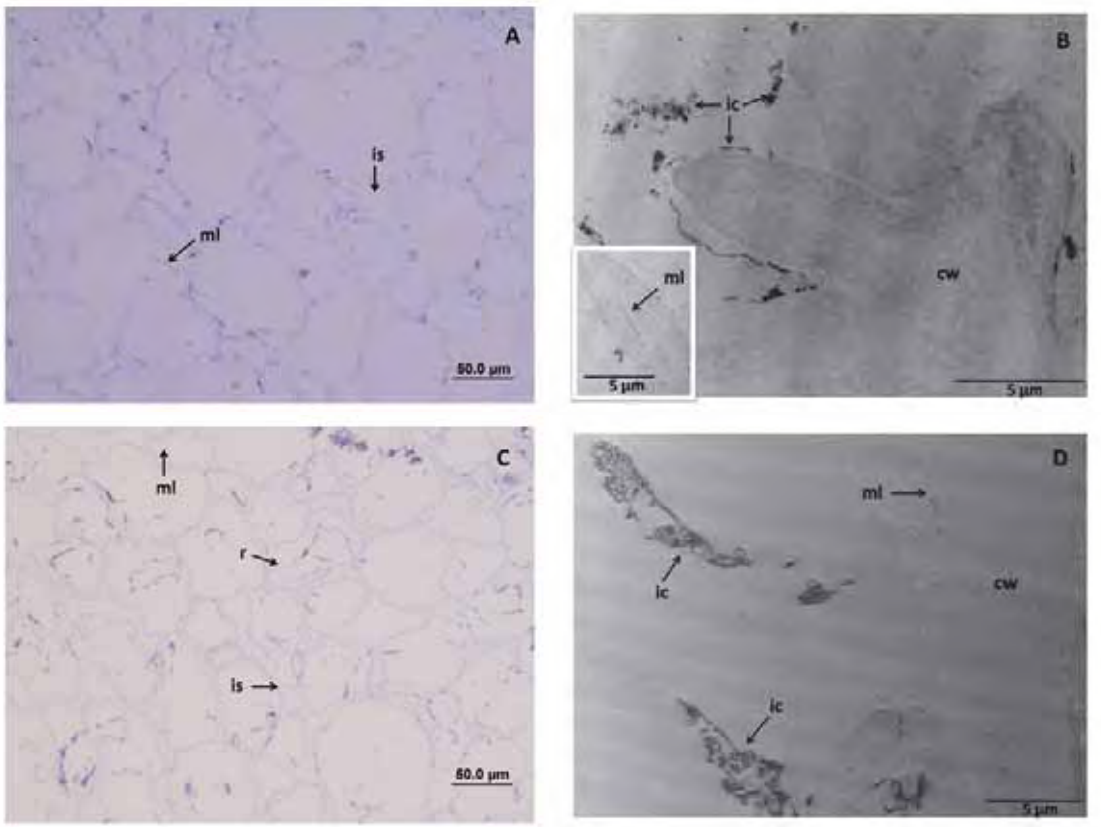

Figure 4. Light (A and C) and TEM (B and D) micrographs from fresh (A and B) and coated (C and D) papaya tissues dried at 70 °C for 300 min. cw: cell wall; ic: intracellular content; is: intercellular spaces; ml: middle lamella; r: rupture of cellular membranes.

Table 10 shows the internal diameters (in µm) of the cells measured from light micrographs using image analyzer software.

The fresh cells had a round shape with an internal diameter of 82.55 µm. The pretreatments did not affect the size or shape of the cells. Blanched cells had an internal diameter of 81.62 µm, and coated cells had an internal diameter of 79.65 µm. In addition to the visualized tissue damage, drying robustly influenced the size of the cells resulting in severe shrinkage as shown by the density results (Tables 2 and 3). Fresh cells had an internal

diameter of 69.36 μm after drying, which was 84% lower than the internal diameter of raw material. Coating did not affect the drying behavior of fresh samples because cells of dried coated tissue had an average internal diameter of 67.47 μm, which was 84.7% lower than cells of dried fresh tissue.

Table 10. Internal diameters (in μm) of the cells measured from light micrographs using image analyzer software.

	Diameter (μm)
Fresh	82.55 ± 20.11
Blanched	81.62 ± 15.56
Coated	79.64 ± 21.49
Fresh – dried	69.36 ± 16.74
Coated – dried	67.47 ± 17.02

4. Conclusions

This study showed that modeling the water diffusion during the drying of papaya slices using analytical solutions of Fick´s Second Law presented a better fit when shrinkage was incorporated into the equation.

Drying blanched samples showed that blanching damaged the cell tissues, which resulted in higher moisture diffusion coefficients and lower drying time as compared with dried fresh fruits. Dried blanched fruits presented lower L*, a* and b* parameters than dried fresh papayas.

Coated fruits had cellular structures similar to those of fresh samples after drying. The moisture effective diffusion coefficients of coated papayas were higher than those of fresh papayas, due to the greater diffusivity of water within the pectin coating.

Pectin coating exerted a protective effect against color and vitamin C degradation during drying because coated papaya slices had higher or similar color parameters than the fresh samples and a 95% retention of vitamin C. The coating did not affect the drying efficiency, which suggests that this technology is a promising alternative to enhance the quality of dried foods.

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CAPÍTULO V

EFFECT OF OSMOTIC DEHYDRATION ON CONVECTIVE DRYING AND QUALITY OF PAPAYA (*Carica papaya*) OF FORMOSA CULTIVAR

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Abstract

The effect of osmotic dehydration and coating applying on air-drying kinetics and quality of papaya of Formosa cultivar was evaluated. A simplified model based on the solution of Fick's Law with shrinkage was used to estimate effective diffusion coefficients during air-drying. Pre-treatments enhanced mass transfer during air-drying. Osmo-treated coated papaya slices had the highest effective moisture diffusivities, followed by the osmotically dehydrated fruits. The relative deviations between predicted and experimental OD and drying data were reduced if using variable thickness in the model. Non-significant changes in color of pre-treated and dried samples were verified. The greater vitamin C losses occurred during osmotic pre-treatment when compared to drying process. In addition, coating did not enhance vitamin C retention after drying. Microscopic observations showed that lactic acid damaged cellular tissue of dehydrated papaya when added to osmotic solution, instead calcium promoted structural preservation of the osmotically treated fruits. Osmo-treated air-dried tissues presented deformed and folded but great preservation of middle lamella was verified due to calcium impregnation.

Keywords: Osmotic dehydration, edible coating, drying, calcium impregnation, vitamin C, microscopic features

1. Introduction

Drying is one of the oldest methods of food preservation. The objective of drying is to remove water from the food materials in order to considerably reduce the microbiological activity of the material, slow down the action of enzymes and minimize many of the water mediated deteriorative reactions (DOYMAZ; GÖL, 2011; DOYMAZ, 2010).

Treatments applied before convective drying are generally used to reduce the initial moisture in food and the quality degradation resulted from drying (SILVA et al., 2011; DOYMAZ, 2010). Osmotic dehydration of fruits and vegetables is a pre-treatment based on their immersion in an aqueous concentrated solution containing one or more solutes, resulting in partially water removal from the product. Simultaneous flow of water and solutes are verified: water and low molecular weight solutes diffuse from the food to the concentrated solution and solutes from the osmotic solution flow into the food (FERNANDES et al., 2009; GARCIA et al., 2007).

Dehydration of foodstuffs by immersion in osmotic solutions previously to convective air-drying seems to improve the quality of the final product since it can minimize color and nutrient losses (MURATORE et al., 2008) and enhance vitamin C retention after drying (SANTOS; SILVA, 2008).

Osmotic dehydration has traditionally been used to incorporate compounds of interest in the food matrix. The use of calcium salts in the osmotic dehydration solutions has been shown to be important in the structural preservation of the processed vegetable products, since it helps maintaining structural integrity of membranes and cell wall, as a result of cross-linking with pectic components of the middle lamella and binding to the cell wall (PEREIRA et al., 2009; ANINO et al., 2006).

Edible coatings are also an alternative pre-treatment that could enhance quality properties of dried food. Traditionally, edible coatings have been used in the fresh-cut industry to reduce the deleterious effects that minimal processing imposes on intact vegetable tissues (DUAN et al., 2011; ROJAS-GRAÜ et al., 2009). More recently, coatings have been used prior to osmotic dehydration, intending to minimize the uptake of osmotic solids (MITRAKAS et al., 2008; KHIN et al., 2006; MATUSKA et al., 2006).

The barrier properties of coatings mostly depend on their composition and the method used for their fabrication. Coatings formed from polysaccharides materials, as low methoxylated pectin, present high oxygen barrier under low moisture conditions, preventing

the oxidation of nutritional compounds and improving the quality of dried product (ZHAO; CHANG, 1995).

The objectives of this work were as follow: a) to determine convective drying characteristics of fresh, osmotically dehydrated and osmo-dehydrated pectin coated papaya slices of Formosa cultivar; b) to compute effective moisture diffusivities of fresh and pre-treated fruits during drying; c) to evaluate the effect of pre-treatments and drying processes on color and retention of vitamin C and d) to clarify the effects of the processes on cell structure modifications.

2. Materials and methods

2.1. Samples preparing

Papaya of Formosa cultivar from São Paulo State (Brazil) were purchased at São José do Rio Preto Supplying Center (CEAGESP, São José do Rio Preto, São Paulo, Brazil). Ripe fruits with orange peels and average reducing sugar contents of 4.25% ± 0.13 were used in the experiments.

Papayas were longitudinally cut into four pieces, and the seeds were removed. Each portion was sectioned into cylinders (diameter of 3.6 cm) with a manual cutter. These pieces were then cut into slices with a thickness of 0.9 cm. The samples were kept and mixed in a plastic bag until the slices were randomly removed to be used in the experiments.

2.2 Osmotic dehydration

Osmotic solutions were prepared with commercial sucrose (refined sugar) purchased in a local market, food grade pentahydrate calcium lactate in powder (PURAC Synthesis, Brazil) in a concentration of 1% w/w, food grade L (+)-lactic acid in 85% solution (PURAC Synthesis, Brazil) in a concentration of 0.1 M and distilled water.

Fresh papayas of Formosa cultivar were dehydrated in 50% w/w sucrose aqueous solutions with 1% w/w of calcium lactate and 0.1 M of lactic acid for 120 min.

OD experiments were conducted into a stainless steel vat with an external jacket for water circulation at 27 °C. Osmotic solutions were continuously stirred using a 1.6 kW mechanical stirrer (Marconi, model MA-261, Brazil) with a 10 cm diameter propeller and rotation at 1000 rpm. The ratio between sample and solution masses were at least 1:10.

After osmotic treatments samples were removed from the solutions, washed with distilled water for 10 s and dried with absorbed paper.

Total solids and reducing and total sugars contents, water activity, color and vitamin C were determined in fresh and osmotically dehydrated fruits.

2.3. Edible coating application

Low methoxylated amidated pectin (GRINDSTED® LA 210; degree of methoxylation of 0.34; degree of amidation of 0.17; Danisco, Brazil) was used to coat the fresh papaya slices. A 2% (w/w) pectin solution was prepared at 70 °C and applied to the surface of the samples at 40 °C by immersion for 1 min. Gelling was activated with subsequent placement of samples in a 2.8% (w/w) aqueous solution of food grade calcium lactate pentahydrate (PURAC Synthesis, Brazil) for 30 s. To remove excess coating, samples were washed in distilled water for 30 s.

The average mass of the pectin coating was determined (0.62 g) and used to calculate the initial thickness of the coat by considering the solution density to be approximately 1000 $kg \cdot m^{-3}$.

2.4. Convective drying

Osmotically dehydrated, coated or not and fresh papaya slices were dried in convective driers at 60 and 70 °C with air velocity of 1.0 $m.s^{-1}$ until a water activity minor than 0.6 was achieved. Osmo-treated samples were placed into driers and dehydrated in parallel (osmo-treated *versus* osmo-treated coated papaya slices). Fresh fruits were dried in a subsequent experiment. The air flowed parallel to the bed, which consisted of three wire nets. Approximately 330 g of samples were dried. Samples were weighted every 20 min during the first hour of drying and every 30 min during the remaining time. At the weighting time, the trails inside the driers were rotated. After drying time, three slices of each sample were kept into the driers for more 2-3 h to determine the equilibrium water content. Total solid contents, reducing sugar contents, total sugar contents, water activity, color and vitamin C contents were determined in fresh and dried (treated or untreated) fruits.

2.5. Microscopic observations

Conventional microscopy techniques were used to reveal structural changes in papaya slices due to osmotic treatment and drying. The following samples were analyzed: a) Fresh, b) osmotically dehydrated in 50% w/w sucrose solution for 120 min, c) osmo-dehydrated in 50% w/w sucrose solution with 0.1 M of lactic acid for 120 min, d) osmo-treated in 50% w/w sucrose solution added with 1% w/w of calcium lactate for 120 min and e) osmotically

dehydrated in 50% w/w sucrose solution added with 0.1 M of lactic acid and 1% w/w of calcium lactate for 120 min papaya slices. Fresh and dehydrated in the last condition of osmotic treatment papayas were also analyzed after convective drying at 70 °C for 300 and 200 min, respectively.

Thin parallelepipeds from the internal zone of the samples were fixed in 4% w/w glutaraldehyde solution overnight at room temperature, postfixed in 1% w/w OsO_4 solution for 2 hs and dehydrated in a graded acetone series prior to being embedded in Araldite 502 resin. For light microscopy (LM), the sections were stained with toluidine blue in borax solution and examined in an Olympus BX60 (Olympus, Hamburg, Germany) photomicroscopic with Olympus DP71 camera. For transmission electron microscopy (TEM), ultrathin sections were stained with uranyl acetate and lead citrate and examined under a Zeiss EM906 Transmission Electron Microscope (Zeiss, Cambridge, England) at 80 kW.

The images were digitized using Image-Pro Plus software (version 6.0, Media Cybernetics Incorporation, Singapore).

2.6. Analytical methodologies

The solid contents of fresh, osmotically dehydrated and osmo-treated coated slices were gravimetrically determined in triplicate by drying the samples in a vacuum oven at 60 °C and 10 kPa until a constant weight was achieved. The reducing and total sugar contents of fresh and osmotically dehydrated fruits were determined in triplicate by an oxidation-reduction titration (WILLIAM, 1970). Water activity of the samples was measured in triplicate at 25 °C in a hygrometer (AW SPRINT; NOVASINA, Switzerland). The color of fresh and osmo-treated either dried or not dried was evaluated in six replicates using a Colorflex spectrophotometer (HunterLab, USA) and version 4.10 of the Universal software with the following settings: illuminant D65, observer at 10° and reading of the absolute values of L*, a* and b*.

The vitamin C content of the samples was determined in duplicate using the modified method described by Benassi and Antunes (1988). Samples (25 g) were homogenized in 50 mL of extractor solution (oxalic acid at 2%; w/w) using Turratec equipment (TECNAL, TE-102 model) for 1 min. An aliquot (20 g) was volumetrically diluted with the extractor solution to 50 mL. Ten milliliters of the diluted solution was titrated with 2.6-dichlorophenolindophenol. The calculations of vitamin C contents of dried papaya slices were based on the total solids contents of the samples. The calculations and the retention of vitamin C related to the fresh samples are presented at Appendix II.

2.7. Calculations

2.7.1. Control of OD

Through the experimental data, total mass variation in relation to initial mass during papaya OD was calculated according to Equation 1.

$$\Delta M = \frac{M - M^0}{M^0} \quad (1)$$

where ΔM represents total mass variation in relation to initial mass; M is the mass at time t, in kg; M^0 is the initial mass at time t = 0, in kg.

Water loss (*WL*) and reducing sugar gain (*RS*) in relation to initial mass were calculated for the OD, through the mass balances shown in Equations 2 and 3.

$$WL = \frac{\left(w_w M\right) - \left(w_w^0 M\right)}{M^0} \quad (2)$$

$$RS = \frac{\left(w_{red} M\right) - \left(w_{red}^0 M^0\right)}{M^0} \quad (3)$$

where w_w and w_{red} represent, respectively, water and reducing sugar contents after a time t of OD. w_w^0 and w_{red}^0 are these contents before OD (t = 0).

During OD of fresh samples sucrose was not detected, due to invertase activity, so calculations considered the reducing sugar contents.

2.7.2. Effective diffusion coefficients

The effective diffusion coefficients of moisture (D_m) were determined according to Fick's Law applied to an infinite slab. The diffusion model has been applied to the drying of biological materials by changing the fractional contents to express the moisture on a dry basis (db) (GARCIA et al., 2007). The analytical solution of the following diffusion equation (Equation 4) has been previously described by Crank (1975):

$$\boldsymbol{X} = \left(\frac{\overline{X}(t) - X^{eq}}{X^0 - X^{eq}} \right) = \frac{8}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{(2n-1)^2} \exp\left[-(2n-1)^2 \frac{\pi^2 D_m t}{z^2} \right] \quad (4)$$

where *eq* indicates equilibrium; *0* indicates initial moisture (at t = 0); $\boldsymbol{X}$ is the fractional or residual moisture, dry basis (dimensionless); X represents moisture (db); $\overline{X}$ is the average moisture (db) at time t; D_m is the effective diffusion coefficient of moisture ($m^2.s^{-1}$) and z is the thickness of the samples.

The coefficients in Equation 4 were determined by considering *z* as the following parameters: a) initial thickness of the slices (0.9 cm), b) an average thickness calculated using the initial and final measurements and c) a variable thickness assuming *z* as a linear function of ***X*** (Table 1) to incorporate shrinkage in the analytical solution. Table 1 presents the linear functions used in the calculations.

Table 1. Linear functions used in the calculations to incorporate shrinkage in the solutions.

Sample after drying	**60 °C**	**70 °C**
Fresh	$z = 0.407\boldsymbol{X} + 0.492$	$z = 0.461\boldsymbol{X} + 0.438$
Osmotically dehydrated	$z = 0.411\boldsymbol{X} + 0.489$	$z = 0.392\boldsymbol{X} + 0.507$
Osmo-treated coated	$z = 0.487\boldsymbol{X} + 0.412$	$z = 0.389\boldsymbol{X} + 0.510$

For long drying times, the terms in Equation 4 were expected to converge rapidly, so only a few terms were required to perform the calculations (DOYMAZ; GÖL, 2011).

2.8. Statistical analysis and fitting

The data were statistically analyzed by an analysis of variance (ANOVA) and Tukey's test at a 5% significance level using Excel 2007 (Microsoft, USA).

Effective diffusion coefficients were calculated from the experimental data according to Equation 2 using Statistica 7.0 software. The fitting efficiency was evaluated by the determination coefficient (R^2) and the mean relative error root square (*RRMS*). RRMS was calculated with Equation 3 as follows:

$$RRMS\ (\%) = 100\left\{\frac{1}{N}\sum_{n=1}^{N}\left[\frac{\left(x^{calc} - x^{\exp}\right)}{x^{calc}}\right]\right\}^{1/2} \quad (5)$$

where x^{calc} represents moisture content on a dry basis and was calculated according to Equation 4; x^{exp} is the experimental value and *N* represents the number of observations.

3. Results and discussions

3.1. Effect of osmotic dehydration on convective drying of papaya slices

The main changes in the weight and composition of papaya slices during osmotic dehydration are summarized in Table 2. Moisture content and water activity of fresh papaya diminished during the osmotic treatment, in accordance with the potential chemical gradient existing between the product and the osmotic solution, while the initial reducing sugar content increased.

Chan and Kwok (1975) verified that invertase is active in papayas of Solo. Authors showed that the enzyme must be inactivated because after manipulation, which involved removing of skin and seeds, cutting and crushing, half of the sucrose was inverted to reducing sugars in 2.66 min. Papaya of Formosa cultivar also presented the activity of invertase, so monitoring of sugar gain during osmotic treatment was performed analyzing reducing sugar content (*RS*) once sucrose was not detected (Table 2).

As expected, mass variation during osmotic treatment was reproducible and similar values of ΔM, *WL* and *RS* were verified in both OD treatments (Table 2).

Initial moisture content of fresh fruits was very similar in both processes (Table 2). As a result of osmotic treatment, moisture of osmotically dehydrated papaya slices was significantly lower than the moisture of fresh samples at the starting time of drying. Osmo-dehydrated fruits gained some water during immersion in coating aqueous solution and significantly increasing their moisture and water activity, since the coating has approximately 98% water content (Table 2). When water activity of foods is approximately 0.6 the growth of microorganisms is reduced if not disabled (YAN et al., 2008). All the dried samples presented satisfactory water activity regarding microorganisms growth (Table 2).

Table 2. Moisture content (w, in g/100 g) and water activity (a_w) of fresh, osmotically dehydrated (OT), osmo-treated coated (OT-CO) and dried papaya slices (60 and 70 °C). Variation in mass (ΔM, in g/100 g), water loss (WL, in g/100 g) and reducing sugar gain (RS, in g/100 g) in relation to the initial mass (M^0), during osmotic treatment (50% sucrose solution with 1% of calcium lactate and 0.1 M of lactic acid, 120 min).

60 °C							
	Before Drying		**After Drying**		**Mass variation during osmotic treatment**		
	w	***a_w***	***w***	***a_w***	***ΔM***	***WL***	***RS***
Fresh	90.35 ± 0.03	0.992 ± 0.001	13.24 ± 1.05	0.510 ± 0.009			
OT	80.84 ± 0.00	0.977 ± 0.004	10.17 ± 0.20	0.466 ± 0.001	-25.94 ± 0.87	-28.87 ± 0.71	10.00 ± 0.17
OT-CO	84.03 ± 0.03	0.981 ± 0.003	12.30 ± 0.22	0.537 ± 0.003			
70 °C							
	Before Drying		**After Drying**		**Mass variation during osmotic treatment**		
	w	***a_w***	***w***	***a_w***	***ΔM***	***WL***	***RS***
Fresh	91.23 ± 0.04	0.991 ± 0.000	13.24 ± 0.86	0.565 ± 0.006			
OT	79.70 ± 0.15	0.978 ± 0.002	15.11 ± 0.31	0.618 ± 0.004	-25.70 ± 0.23	-29.25 ± 0.18	9.86 ± 0.04
OT-CO	83.30 ± 0.12	0.981 ± 0.003	13.41 ± 0.36	0.577 ± 0.005			

The drying curves of moisture content *versus* the drying time are presented in Figure 1, showing the effects of osmotic treatment and coating on the drying time of papaya slices.

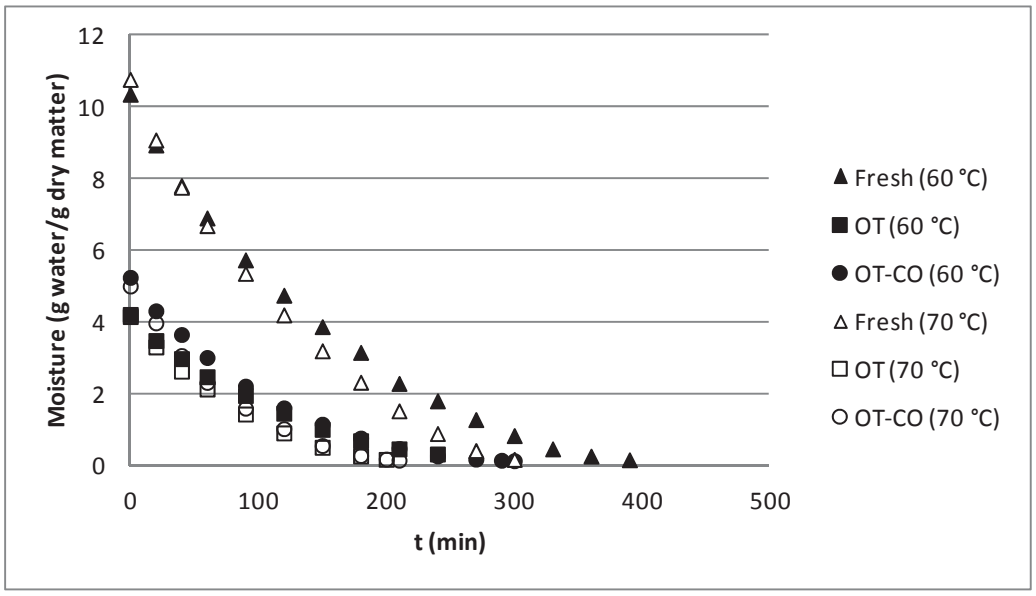


Figure 1. Variations of moisture with drying time of fresh, osmotically dehydrated (OT) and osmo-treated coated (OT-CO) papaya slices at 60 and 70 °C.

Drying temperature significantly affected the moisture change of both fresh and osmotically dehydrated papaya slices. As a result of a larger driving force for heat transfer, drying at higher temperature was faster to achieve a specific moisture (Figure 1). The influence of air temperature on drying time was noticed for all fresh and pretreated samples. With increase in the air temperature the drying curves become more accentuated (Figure 1) and caused the reduction of the final product drying time (SHI et al., 2008; EL-AOUAR et al., 2003).

The reduced drying time of sugar-infused papaya slices was a result of their low initial moisture content, which was much lower than that of fresh fruits (Table 1). Coating did not affect the drying behavior of osmotically dehydrated papaya slices, but increased drying time.

The difference in drying times of pre-dehydrated coated samples was 10% and 5% longer than the osmotically dehydrated fruits dried at 60 and 70 °C, respectively.

The drying process of the samples occurred in the range of the falling-rate period, which indicated that internal diffusion was the dominant physical mechanism governing the moisture movement in the samples. Similar results were obtained by Doymaz (2010) for apples, Shi et al. (2008) for blueberries and Nieto et al. (2001) for mangos.

Table 3 presents the moisture effective diffusion coefficients, calculated according to Equation 4, considering *z* as the following parameters: a) initial thickness, b) average thickness (between initial and final state) and c) variable thickness (a linear function of water content, Table 1). In both the second and third (b and c) diffusivity determinations, the thickness *z* was estimated considering similar shrinkage in all dimensions. Consideration of variable thickness in calculations better predicted the moisture content. Good fitting was obtained with this assumption because the values of R^2 were higher than 0.99 and *RRMS* values were the lowest (Table 3). Even though *RRMS* values have been high, it can be explained due to small moisture content at the last drying stages that increased relative deviations in Equation 6.

D_m values of fresh and pre-treated fruits dried at 70 °C were higher than the ones obtained at 60 °C (Table 3), as more heating energy speeds up the movement of water molecules enhancing diffusivities (DOYMAZ, 2010; SHI et al., 2008; GARCIA et al., 2007). The effect of the predehydration on convective drying enhanced the water transfer (Figure 1). Moreover, the effective diffusion coefficients were higher for pretreated than untreated samples (Table 3). However, pretreatment of fruits in sugar solution is expected to reduce the water diffusion during convective drying (SIMAL et al., 1997; KARATHANOS et al., 1995; RAHMAN; LAMB, 1991) due to the higher water retention capacity by the sucrose (CHIRIFE et al., 1980) when compared to cellulosic compounds (PAPADAKIS et al., 1993) at low water content. The higher diffusivities of pretreated papaya were related to the reduction in the effect of shrinkage and surface hardening due to the osmotic treatment (GARCIA et al., 2007, PARK et al., 2002). On the other hand, the fast drying of the fresh papaya slices surface possibly formed areas of hardness on the surface and hence reduced the drying rates, as verified for pumpkins by Garcia et al. (2007). Additionaly, it is possible that papaya provides a natural protective barrier to oxidation during air drying, due to its latex compounds which are partially liberated by the pulp after cutting. Latex from papaya is a rich source of plant proteases (ABDELKAFI et al., 2009; AZARKAN et al., 2004).

Moisture effective diffusion coefficients of the coated papayas were higher than the fresh and osmotically treated samples (Table 3), due to the strong hydrophilic nature of the pectin (McHUGH; KROCHTA, 1994).

Table 3. Effective diffusion coefficients of moisture ($D_m \times 10^{10}$, in $m^2 \cdot s^{-1}$), calculated according Equation 4, considering initial, average and variable thickness of fresh, osmotically dehydrated (OT) and osmo-treated coated (OT-CO) papaya slices after convective drying at 60 and 70 °C.

60 °C									
	Fresh			**OT**			**OT-CO**		
	Initial thickness	**Average thickness**	**Variable thickness**	**Initial thickness**	**Average thickness**	**Variable thickness**	**Initial thickness**	**Average thickness**	**Variable thickness**
D_m	7.95	4.81	3.60	12.09	7.11	5.32	13.59	7.03	5.39
R^2	0.9576	0.9576	0.9978	0.9599	0.9599	0.9984	0.9591	0.9591	0.9982
RRMS (%)	39.77	39.77	22.77	56.10	56.10	40.02	51.39	51.39	33.03
70 °C									
	Fresh			**OT**			**OT-CO**		
	Initial thickness	**Average thickness**	**Variable thickness**	**Initial thickness**	**Average thickness**	**Variable thickness**	**Initial thickness**	**Average thickness**	**Variable thickness**
D_m	9.51	5.27	3.83	16.11	8.57	6.72	17.42	9.26	7.17
R^2	0.9514	0.9514	0.9979	0.9536	0.9536	0.9974	0.9609	0.9609	0.9968
RRMS (%)	20.87	20.87	19.77	37.48	37.48	17.15	62.84	62.84	38.71

Effective diffusivities reported in the literature present different estimation methods and models employed, besides the food composition and physical structure variations, becoming difficult the comparisons. El-Aouar et al. (2003) found values around 10^{-9} $m^2 \cdot s^{-1}$ for moisture diffusivities during air-drying of osmotically dehydrated papaya, higher than the coefficients obtained in this work.On the other hand, values around 10^{-10} $m^2 \cdot s^{-1}$ had been found for several vegetables and fruits (DOYMAZ, 2007; GARCIA et al., 2007; AKPINAR et al., 2003; NIETO et al., 2001).

3.2. Effect of processing on quality of papaya slices

3.2.1. Color

The osmotic dehydration practically did not change the color of the papaya (Table 4). Also, the air-drying of osmotically dehydrated papayas, with or without coatings, did not significantly changed the L*, a* and b* color parameters in a confidence level of 95%.

A tendency of increasing lightness, redness and yellowness when applying the osmotic treatment is reported by other researchers (FALADE et al., 2007; TONON et al., 2007; RODRIGUES et al., 2003), probably due to the concentration effect. The immersion of the fruits into the osmotic solution can protect the surface from the oxygen and hence minimize oxidation of color pigments. However, in the present work neither the osmotic dehydration nor the air drying influenced the color parameters after processing.

3.2.3. Vitamin C

The effects of the osmotic treatment, coating and the drying temperatures on the degradation of vitamin C are shown in Table 4.

As seen in Table 5, ascorbic acid, a hydrophilic vitamin, diffused from the fruit to the osmotic solution. About 15-22% of the initial vitamin C was lost during osmotic treatment. Loss of vitamin C during drying of osmotically treated samples was verified by Vial et al. (1991) for kiwi and by Heng et al. (1990) for papaya.

Table 4. L*, a* and b* color parameters of fresh, osmotically dehydrated (OT) and osmo-treated coated (OT-CO) papaya slices.

	60 °C					
	Before drying			**After drying**		
	L*	**a***	**b***	**L***	**a***	**b***
Fresh	49.64 ± 1.52	33.06 ± 1.05	38.30 ± 1.52	ND	ND	ND
OT	51.77 ± 1.55[a]	33.47 ± 1.59[a]	41.64 ± 1.47[a]	45.59 ± 1.63[a]	33.00 ± 1.60[a]	39.73 ± 1.73[a]
OT-CO	49.64 ± 1.41[a]	32.58 ± 0.64[a]	39.91 ± 2.11[a]	50.08 ± 3.55[a]	32.53 ± 3.38[a]	40.58 ± 3.83[a]
	70 °C					
	Before drying			**After drying**		
	L*	**a***	**b***	**L***	**a***	**b***
Fresh	47.77 ± 2.20	33.86 ± 0.83	37.03 ± 1.08	ND	ND	ND
OT	47.46 ± 3.32[a]	34.22 ± 1.16[a]	39.48 ± 2.81[a]	50.33 ± 1.19[a]	31.97 ± 0.78[a]	42.87 ± 1.09[a]
OT-CO	52.67 ± 2.66[a]	32.54 ±2.57[a]	40.52 ± 1.46[a]	52.28 ± 3.14[a]	30.93 ± 1.23[a]	40.89 ± 3.28[a]

Different letters for the same parameter in the same line indicate statistically significant differences at $p < 0.05$.

ND: Non-determined.

Table 5. Vitamin C contents, in mg/100 g of sample, of fresh, osmo-dehydrated (OT) and osmotically treated coated (OT-CO) papaya slices before and after drying at 60 and 70 °C and retention (%) of the referred vitamin related to the fresh sample after osmotic dehydration (OD) and drying (DR).

	60 °C				**70 °C**			
	Before drying		**After drying**		**Before drying**		**After drying**	
	Vitamin C	**Retention (OD)**	**Vitamin C**	**Retention (DR)**	**Vitamin C**	**Retention (OD)**	**Vitamin C**	**Retention (DR)**
Fresh*	44.30 ± 1.69	-	330.86 ± 8.19	83.03	55.61± 1.00	-	480.52 ± 27.97	87.30
OT	63.86 ± 3.66	77.78	275.07 ± 2.54	71.44	76.12 ± 0.7	84.88	309.86 ± 23.49	78.79
OT-CO			262.20 ± 32.43	65.78			292.27 ± 5.47	69.90

ND: Non-determined.

*independent experiment.

Osmotic treated fruits presented retention of vitamin C during drying (Table 5). However, the vitamin C is soluble in water solutions and vitamin C losses during the osmotic dehydration together with the losses during the drying, resulted in lower retention than that found in the fresh dried papaya (Table 5). On the other hand, for non-soluble components, this process seems interesting. Moreover, the nature of the tissue probably is an important factor on nutrients retention and papaya seems to exert a protective effect on vitamin C in comparison to other foods. Vitamin C retention in air dried papaya at 45°C was 75% while in guavas was 25% (HAWLADER et al., 2006).

Coating application on papaya slices prior to drying was expected to improve quality of dried product due to its barrier properties against oxygen. However, coating on osmosed fruits did not enhance vitamin C content after drying (Table 5). Vitamin C retention of osmo-treated coated samples was lower than that of the osmotically dehydrated papaya slices after drying at both studied temperatures (Table 5), showing that osmotic treatment itself could provide dried papayas with improved quality.

The studied air drying temperatures did not have a significant effect on vitamin C degradation of sugar infused dried papaya slices which was strongly influenced by the retention during osmotic treatment.

3.2.3. Microscopic features

In order to analyze the structural alterations associated with the osmotic processes and drying of papayas, microscopical studies were conducted using LM and TEM techniques (Figures 2 and 3).

As seen in Figure 2, dehydrated papaya tissues in four different osmotic solutions for 120 min were microscopically evaluated as follow: a) 50% w/w sucrose solution, b) 50% w/w sucrose solution with 0.1 M of lactic acid, c) 50% w/w sucrose solution with 1% w/w of calcium lactate and d) 50% w/w sucrose solution with 0.1 M of lactic acid and 1% w/w of calcium lactate. The last condition was used in the osmotic experiments of the present work and the others were prepared to evaluate the effects, at structural level, of adding calcium salt and acid to the dehydrating solution.

As seen in LM images (Figure 2 A-B), fresh tissue cells (control) and intercellular spaces were arranged in a net-like pattern. Cells showed round shape and turgid, with an apparent consistent cell wall structure. The large amount of cell volume was occupied by the central vacuole. The cytoplasm, bounded by the plasmalemma and the tonoplast, was present as a thin layer, lining the cell surface, where the nucleus of the fresh cell was observed.

Various shapes and sizes were verified for the intercellular spaces. A well defined middle lamella was cementing adjacent cells.

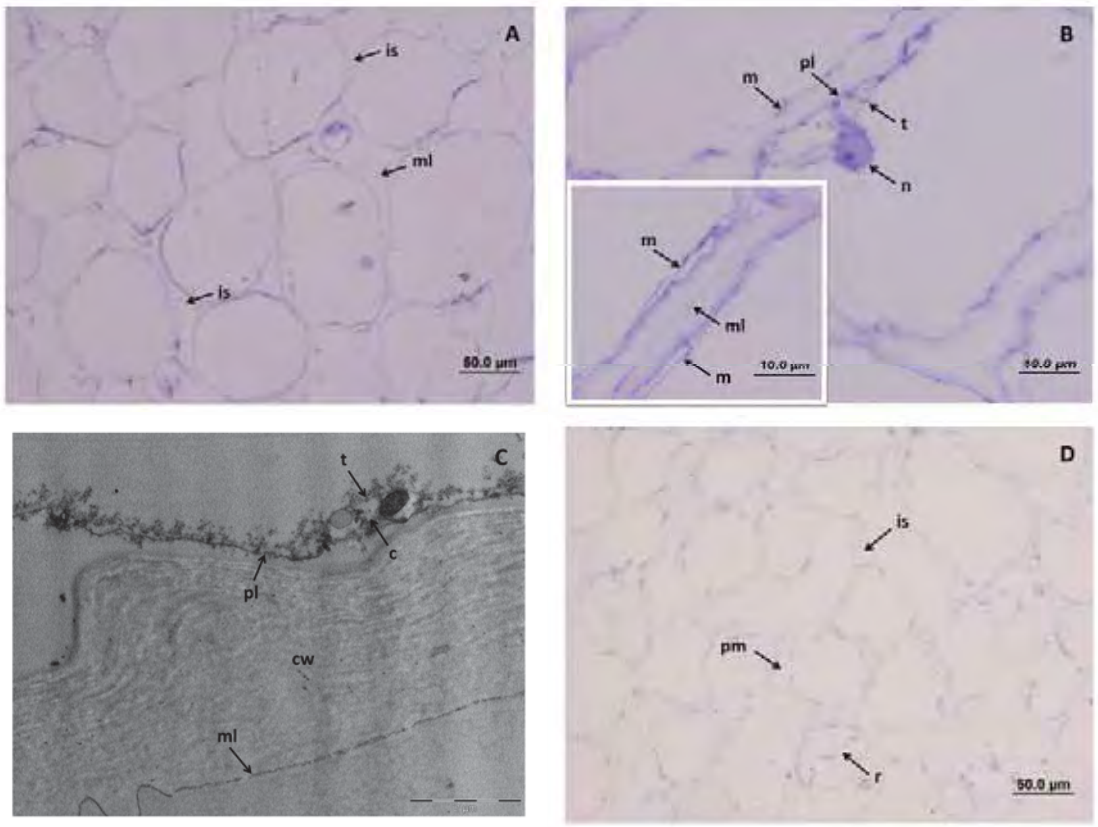
Stained cell walls with a clear longitudinal fibrillar pattern were verified in TEM micrographs of fresh tissue (Figure 2 C). Cell walls presented with a higher intensity toward the margin and in the central zone of the middle lamella, which was intact (Figure 2 C). The arrangement of cytoplasm was marginal and tonoplast and plasmalemma appeared intact (Figure 2 C).

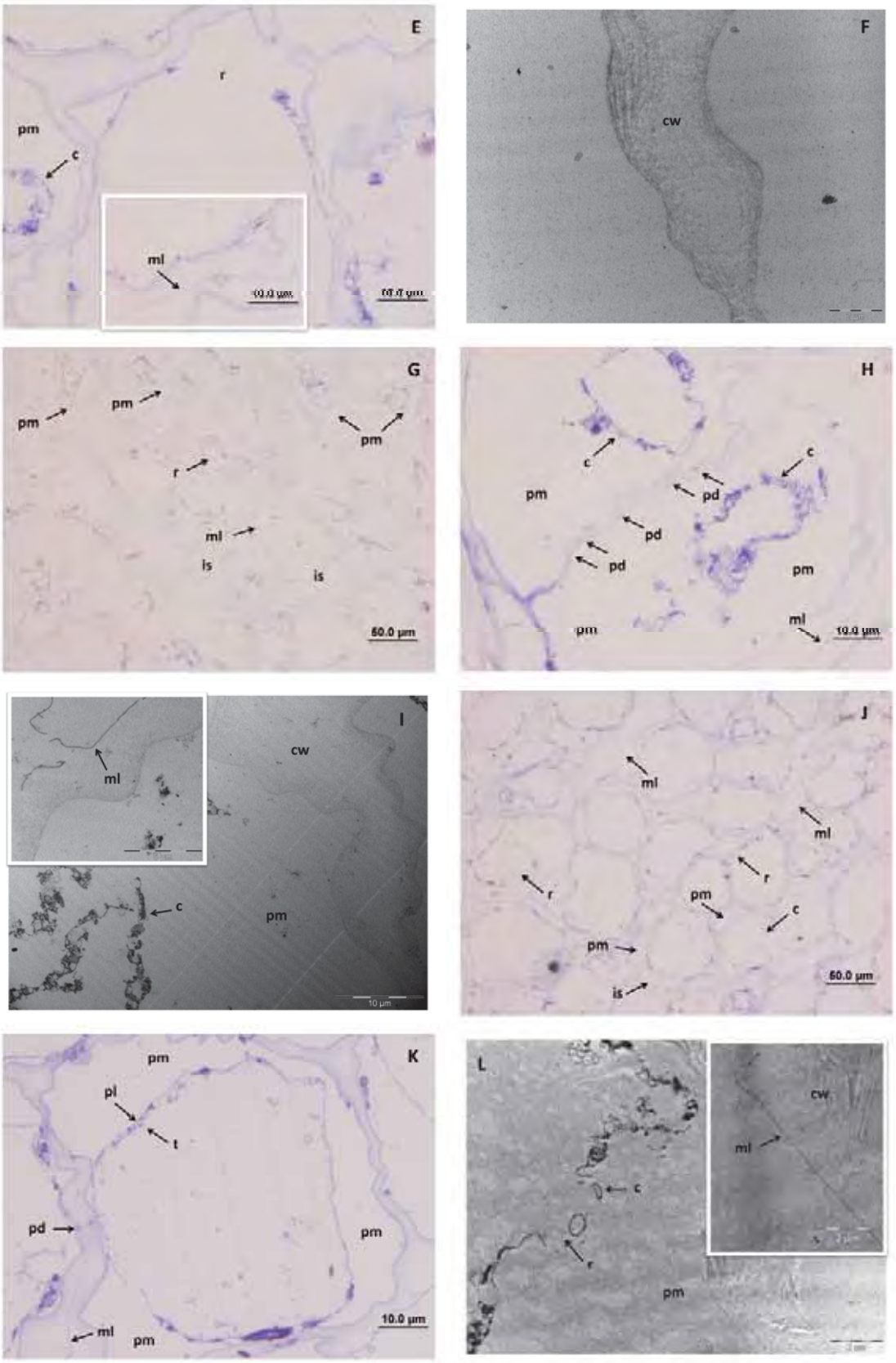
After 120 min of osmotic dehydration in 50% w/w sucrose solution, cell structure appeared affected by plasmolysis (Figure 2 D-E). The occurrence of plasmolysis during osmotic dehydration was also noticed by Martínez et al. (2007) and Nieto et al. (2004) working with apples. Some loss of the cell turgor, shrinkage of the tissue and reduction of intercellular spaces associated to water loss were observed. In some cells, rupture of cellular membranes was also noticed (Figure 2 D-E). Cell walls were deformed and became distorted compared to the fresh tissue, indicating some ultrastructural cell wall impact by the treatment (Figure 2 D-E). Miofibrillar disorganization of cell wall which presented lower optical density compared to the fresh tissue was verified (Figure 2 F). Although residues of the middle lamella were present, solubilization of pectin was observed (Figure 2 D-F). Similar results were found by Fernandes et al. (2009) for melon and by Fernandes et al. (2008) for pineapple. After osmotic treatment in sucrose solution added with lactic acid, cell structure appeared strongly affected by plasmolysis (Figure 2 G-I), probably resulting in enhanced osmotic diffusivities, as noticed by other researchers (ARGADOÑA et al., 2002; HENG et al., 1990). Loss of the cell turgor, shrinkage of the tissue and reduction of intercellular spaces were again verified (Figure 2 G-I), but with more intensity than in sucrose dehydrated cells (Figure 2 D-F). Rupture of cellular membranes was noted in some cells, but plasmolysis was predominant (Figure 2 G-I). Figure 4 G-I showed deformation of cell walls which became distorted in all regions of the samples. More pronounced miofibrillar disorganization of cell wall was verified (Figure 2 I) than that for sucrose treated samples (Figure 2 C). Solubilization of pectin was observed regardless of residues of the middle lamella were present (Figure 2 G-I).

Adding of calcium ions to the osmotic solution resulted in linkages between these ions and the pectin of the cell walls and middle lamella, promoting structural preservation of the osmotically dehydrated papayas. Similar results were verified by other researchers (PEREIRA et al., 2009; GÓNZALEZ-FÉSLER et al., 2008). As the other osmotic treatments, plasmolysis and some rupture of the cellular membranes were verified with adding calcium (Figure 2 J-L). Apparently, shrinkage of these reinforced cells (Figure 2 J-L) was lower than that of the

osmotically dehydrated with acid (Figure 2 G-I) and similar to the osmo-dehydrated in sucrose solution (Figure 2 D-F) tissues. Calcium incorporation resulted in stained cell walls with a longitudinal fibrilar pattern similar to the raw material, but folding of cell walls occurred (Figure 2 L and C). Unlike osmotic treatments in sucrose and in sucrose plus acid solutions, preservation of middle lamella was high due to the bounding of calcium salt with pectin (Figure 2 L).

Intense plasmolysis and rupture of cellular membranes, as verified for the samples osmotically dehydrated in sucrose solution added with lactic acid, were verified for the fruits osmo-dehydrated in sucrose solution added with calcium salt and lactic acid (Figure 2 M-O) Cell walls presented deformed and density of staining similar to the sucrose dehydrated cells (Figures 2 O and F) but with a longitudinal fibrillar pattern similar to the raw material (Figures 2 C and O). Despite calcium incorporation, some solubilization of chelator-soluble pectin occurred, resulting in reduced structural damages and high osmotic diffusivities due to solutes addition. As well as cells treated in sucrose solution with calcium lactate, preservation of middle lamella was high due to the bounding of calcium salt with pectin (Figure 2 M-O and D-F).

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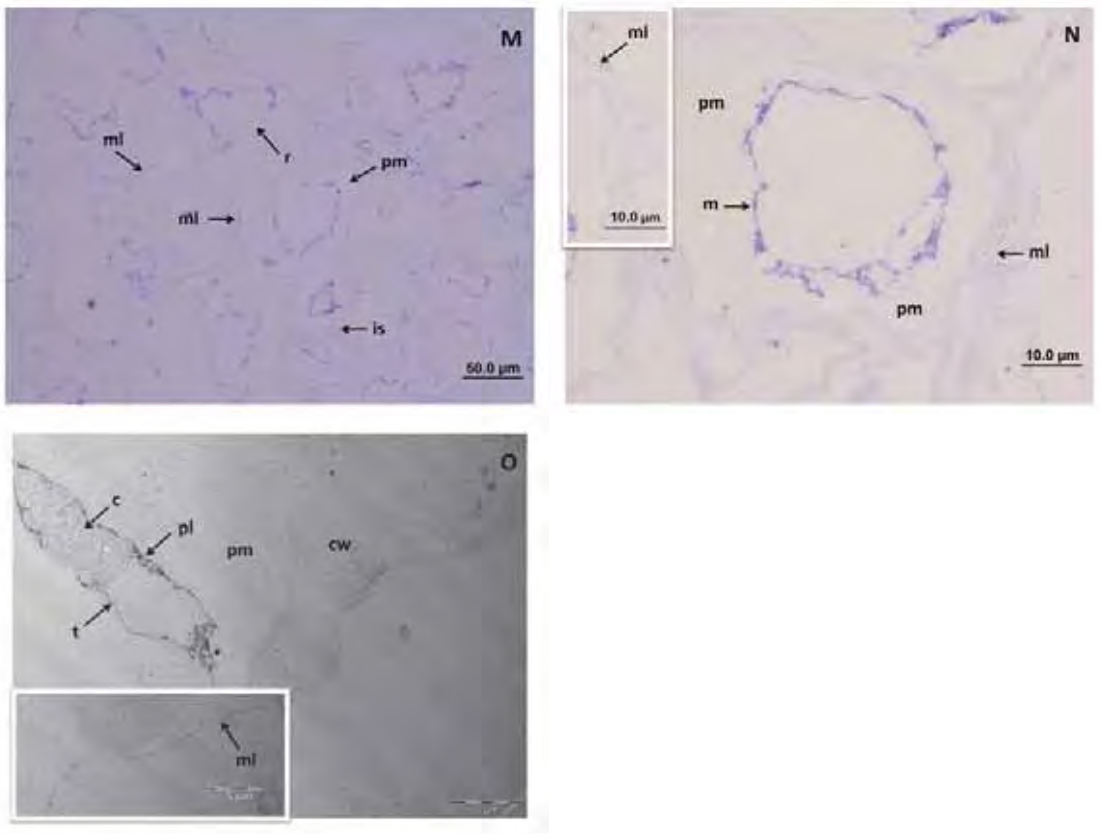

Figure 2. LM (A, B, D, E, G, H, J, K, M, N) and TEM (C, F, I, L, O) micrographs of papaya from fresh and osmotically tissue treated in different conditions for 120 min. A-C: fresh papaya; D-F: osmotically dehydrated fruits in 50% w/w sucrose solution; G-I: osmo-dehydrated papaya in 50% w/w sucrose solution with 0.1 M of lactic acid; J-L: osmo-treated samples in 50% w/w sucrose solution added with 1% w/w of calcium lactate; M-O: osmotically dehydrated fruits in 50% w/w sucrose solution added with 0.1 M of lactic acid and 1% w/w of calcium lactate. c: cytoplasm; cw: cell wall; is: intercellular space; m: membranes (plasmalemma plus tonoplast); ml: middle lamella; n nucleus; pl: plasmalemma; pm plasmolysis; pd: plasmodesmatas; r: rupture of cellular membranes; t: tonoplast.

Figure 3 presents LM and TEM micrographs of papaya tissue, fresh and osmotically dehydrated in sucrose solution with calcium lactate and lactic acid (condition of the present work) after convective drying at 70 °C for 300 and 200 min, respectively.

Figure 3 A-C showed that drying of fresh papaya resulted in strongly deformed and folded cell walls. Compared to fresh sample, lower optical density and disorganization of cell wall arrangement were verified (Figures 2 C and 3 C). Some poor residues of middle lamella were present and intense rupture of cellular membranes occurred in all the visualized cells. This intracellular membranous content was called intracellular content.

Osmotically dehydrated dried samples presented more deformed and folded cell wall than dried fresh samples (Figure 3). Figure 3 C showed that this treatment caused disorganization of the cell wall fibrillar pattern. Despite that, disruption of cell walls was not observed. Calcium incorporation also resulted in a great preservation of middle lamella (Figure 3 D and E). As a result of the intense heat treatment used, great rupture of cellular membranes occurred leading to a dispersed membranous intracellular content (Figure 3 B-F).

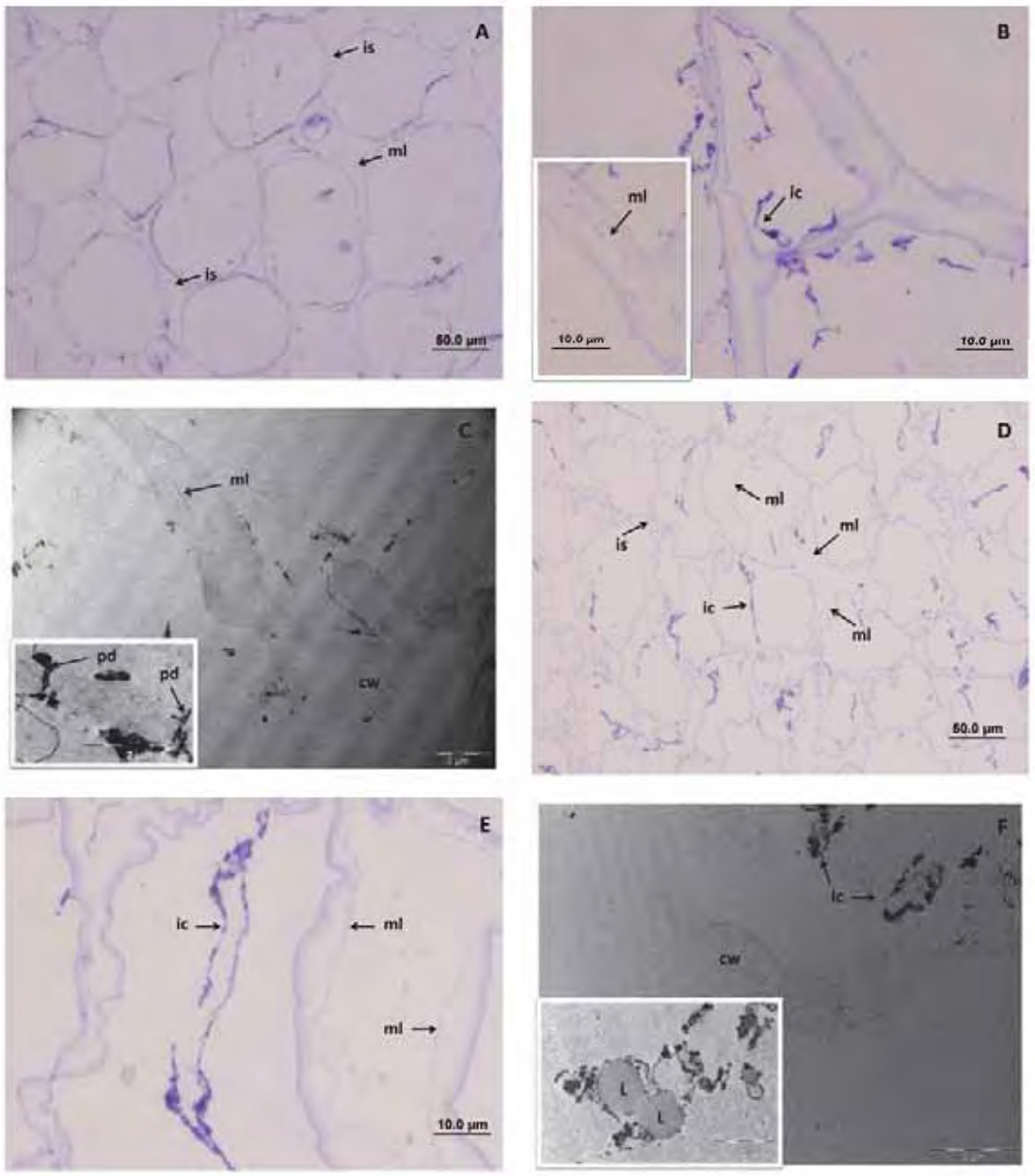

Figure 3. LM (A, B, D, E) and TEM (C, F) micrographs from fresh and osmotically dehydrated (50% w/w sucrose solution with 0.1 M of lactic acid and 1% w/w of calcium lactate) papaya tissues after air-drying at 70 °C for 300 and 200 min, respectively. A-C: dried

fresh papaya; D-F: dried osmotically dehydrated papaya. cw: cell wall; ic: intercellular content; is: intercellular space; L: lipid inclusion; ml: middle lamella; pd: plasmodesmatas.

3. Conclusions

The water effective diffusion coefficients increased with the drying temperature. Pre-treatments enhanced the water transfer during drying and moisture diffusion coefficients resulted higher than those for the non-treated fruits. This behavior was related to the fast surface drying of the fresh samples as well as to the latex compounds on the fresh surface papaya. Coated fruits presented the highest water diffusion coefficients during convective drying and slightly increased the drying time. Osmotic dehydration reduced the drying time.

The variations in color due to pre-treatments and drying were not significant at a 95% confidence level. However, the results suggested that osmotic treatment was efficient to avoid oxidation of color pigments when compared to non-treated samples.

During osmotic treatment, 15-20% of the initial vitamin C was lost. Osmotic treatment itself could not provide dried papayas with improved quality at both studied temperatures and coating did not enhanced vitamin C content after drying. More investigation should be carried out to determine nutrient stability during storage.

Fresh papaya seemed to provide a natural protective barrier to oxidation during air-drying, probably related to the latex compounds present in the pulp, which are partially liberated after cutting. This barrier probably was responsible for the lower drying efficiency of fresh papaya in comparison to osmotically dehydrated samples. More studies should be carried out to elucidate these questions.

Microscopic observations showed that lactic acid caused severe damages to the fresh cellular tissue of papaya when added to osmotic solution. However, calcium lactate promoted structural preservation of the osmotically dehydrated papayas.

Intense plasmolysis and rupture of cellular membranes was verified for the samples osmotically dehydrated in sucrose solution added with acid and calcium salt. Some solubilization of chelator-soluble pectin occurred, but preservation of middle lamella was high, resulting in reduced structural damages, as a result of calcium incorporation.

Osmotically dehydrated dried samples presented more deformed and folded cell wall than dried fresh samples. Cell walls presented density of staining similar to the fresh sample and great preservation of middle lamella, probably due to calcium impregnation during osmotic treatment.

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4. CONCLUSÕES GERAIS

A avaliação da desidratação osmótica (DO) de mamões frescos e branqueados em solução de sacarose com adição de lactato de cálcio e ácido láctico demonstrou que diferentes concentrações de sacarose e tempos de desidratação não alteraram significativamente a cor das fatias frescas e branqueadas de mamão Formosa, concluindo-se que pigmentos foram preservados durante o processo. Para as frutas branqueadas, foi encontrada uma expressão na qual o tempo de processamento apresentou maior influência no decréscimo da atividade de água do que a concentração da solução desidratante. Para utilização da DO como tratamento prévio à secagem, selecionou-se a solução com 50% de sacarose, 1% de lactato de cálcio e 0,1 M de ácido lático, e tempo de processo de 120 min para as frutas frescas e 105 min para as branqueadas. Secagem convectiva de mamão fresco e branqueado, desidratado osmoticamente ou não e revestido ou não com cobertura comestível à base de pectina, mostrou que o branqueamento não foi um pré-tratamento apropriado na redução da atividade de água e preservação de cor. A redução de atividade de água durante a secagem foi mais efetiva em amostras desidratadas osmoticamente, com ou sem cobertura, mostrando o potencial da DO para obtenção de produtos mais estáveis, com menor gasto energético.

Isotermas de dessorção de mamão fresco, branqueado termicamente e desidratado osmoticamente foram bem representadas pelo modelo de GAB, nas temperaturas de 30, 50 e 70 ^{o}C. As frutas branqueadas mostraram comportamento não usual. Calores isostéricos de sorção indicaram que a dessorção da água das frutas desidratadas osmoticamente requereu maior quantidade de calor que das frescas e, por fim, das branqueadas. A teoria de compensação entalpia-entropia foi satisfeita, demonstrando que os processos de dessorção são controlados entalpicamente.

A avaliação da cinética da desidratação osmótica de mamão branqueado e de mamão fresco demonstrou grande efeito do tratamento térmico. Enquanto imagens de microscopia do tecido vegetal fresco mostraram plasmalema e tonoplasto íntegros e presença de lamela média intacta, verificou-se, no tecido branqueado, rompimento do plasmalema e do tonoplasto, assim como muitos pontos de solubilização da lamela média, o que resultou no amolecimento das fatias de mamão. Como resultado, durante a DO dos mamões, as frutas branqueadas apresentaram maior perda de água e ganho de solutos, assim como maiores coeficientes de difusão da água e da sacarose em comparação às fatias frescas. O ganho de soluto das frutas branqueadas durante a DO foi excessivo, chegando ao dobro do ganho das fatias frescas, mostrando que a combinação branqueamento-DO não é vantajosa do ponto de vista

nutricional. Também se constatou presença de enzima invertase nos mamões frescos, devido à ausência da sacarose nessas amostras. Nas amostras não tratadas termicamente, a difusividade efetiva do açúcar, neste caso medido como açúcar redutor, resultou maior que da água, o que foi atribuído à atividade da enzima, que alterou a composição de açúcares durante o processamento. A caracterização da enzima mostrou que o mamão apresenta elevada concentração da mesma.

Durante a secagem convectiva de mamão, verificaram-se maiores coeficientes efetivos de difusão da água para frutas branqueadas, quando comparadas com frescas. O tempo de secagem dos mamões branqueados também foi menor que dos frescos. As frutas branqueadas escureceram durante a secagem e a retenção da vitamina C, após sua secagem, foi menor que das amostras frescas. As atividades de água das fatias branqueadas de mamão, após a secagem, foram mais elevadas que as das frutas *in natura* e que as das desidratadas osmoticamente, revelando que o branqueamento não foi um pré-tratamento adequado para reduzir a atividade de água e melhorar as características nutricionais das amostras.

Imagens microscópicas do tecido vegetal fresco, após a secagem, mostraram que esse processo danificou intensamente o tecido vegetal, cuja parede celular apresentou-se bastante deformada. Intensa ruptura das membranas celulares, espalhadas pela célula, foi verificada.

A secagem convectiva de mamão desidratado osmoticamente mostrou que esse pré-tratamento intensificou a perda de água das amostras durante a secagem, resultando em maiores coeficientes de difusão da água e menores tempos de processamento em comparação às frutas frescas. Esse comportamento não usual foi relacionado à secagem rápida da superfície das amostras frescas, assim como ao látex presente nessa fruta, com consequente formação de áreas endurecidas que restringiriam a secagem. Verificou-se, através de imagens, que após a secagem, as células das frutas desidratadas osmoticamente apresentaram maior preservação da lamela média que as das frutas frescas secas, devido à impregnação de cálcio. O cálcio, por sua vez, também demonstrou, através da microscopia, capacidade de preservar melhor a estrutura celular do tecido de mamão durante a desidratação osmótica. A maior eficiência de secagem das amostras desidratadas osmoticamente mostrou que esse processamento pode ser útil na obtenção de produtos estáveis e na economia energética. Por outro lado, as perdas de vitamina C durante o tratamento osmótico reduzem seu conteúdo no produto desidratado, em comparação com o conteúdo das frutas frescas secas.

Conforme verificado nas imagens de microscopia, a aplicação da cobertura comestível não alterou o tecido celular das frutas frescas, mesmo após a secagem. Porém, devido à natureza hidrofílica da cobertura, as difusividades efetivas dos mamões cobertos foram

maiores do que as das frutas frescas e das desidratadas osmoticamente. Quando adicionada às amostras frescas, a cobertura comestível protegeu contra a oxidação da vitamina C, cuja retenção alcançou 95%, durante a secagem. Por outro lado, a adição de cobertura sobre as frutas desidratadas osmoticamente não melhorou a retenção dessa vitamina durante a secagem. Ainda que amostras frescas com cobertura tenham apresentado valores elevados de retenção de vitamina C após a secagem, também foram encontrados, em amostras frescas sem cobertura, conteúdos relativamente altos dessa vitamina no produto desidratado, o que pode estar relacionado ao látex presente na polpa do mamão, que agiria como uma barreira protetora natural contra a oxidação durante a secagem. Essa barreira, provavelmente, foi responsável pela baixa eficiência de secagem dos mamões frescos, em comparação aos desidratados osmoticamente. Estudos futuros devem ser realizados para esclarecer o papel desempenhado pelo látex presente no mamão durante a secagem, uma vez que o presente estudo sugeriu influência desse composto sobre a transferência de massa durante o processo.

5. PROPOSTAS PARA ESTUDOS FUTUROS

Mais estudos devem ser conduzidos para avaliar a possibilidade de utilização do mamão para extração da invertase, principalmente objetivando o aproveitamento das perdas na colheita e pós-colheita.

Estudos de armazenamento também devem ser realizados visando a avaliação do conteúdo nutricional das frutas secas, assim como sua segurança microbiológica. Além disso, o papel desempenhado pelo látex durante o armazenamento deve ser esclarecido.

A avaliação sensorial do produto final também será uma análise importante para detectar o comportamento do consumidor em relação a esses produtos desidratados.

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APÊNDICE I:

MATERIAIS E METODOLOGIAS

1. Matéria-prima: Mamões

Mamões compridos, cultivar Formosa, provenientes do Estado de São Paulo (Figura 1), foram adquiridos no CEASA de São José do Rio Preto e, quando não utilizados imediatamente, ficaram armazenados sob refrigeração à temperatura de 4 °C por um período máximo de 96 horas. Frutas maduras com a casca levemente alaranjada foram utilizadas nos experimentos. Nos ensaios realizados para determinação das melhores condições de DO e secagem (Capítulo I), nos de cinética de DO (Capítulo II) e para a determinação das isotermas de sorção (Capítulo III), as frutas frescas apresentaram teor médio de açúcares redutores de 8,00% ± 0,00 e de açúcares totais de 8,04 ± 0,04. As frutas *in natura* usadas nos ensaios de secagem (Capítulos IV e V) apresentaram teor médio de açúcares redutores de 4,87% ± 0,08 e de açúcares totais de 4,86% ± 0,01.

Figura 1. Mamões utilizados nos experimentos.

2. Corte e preparo das amostras

Mamões apresentando casca levemente alaranjada foram selecionados aleatoriamente para os ensaios de DO.

Os mamões foram cortados longitudinalmente em quatro, as sementes foram retiradas (Figura 2) e cada parte foi seccionada com um cortador manual de 3,6 cm de raio interno (Figura 3). Os cilindros de mamão foram cortados em fatias de aproximadamente 1,0 cm de espessura (Figura 4). As amostras foram armazenadas e misturadas em um saco plástico (Figura 5) do qual, aleatoriamente, foram retirados os pedaços a serem utilizados nos experimentos.

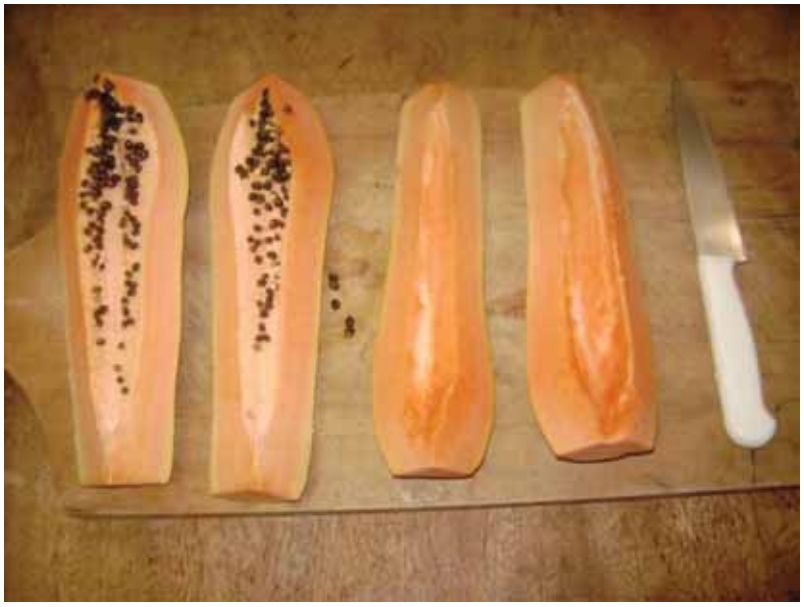

Figura 2. Corte longitudinal das frutas selecionadas para os experimentos.

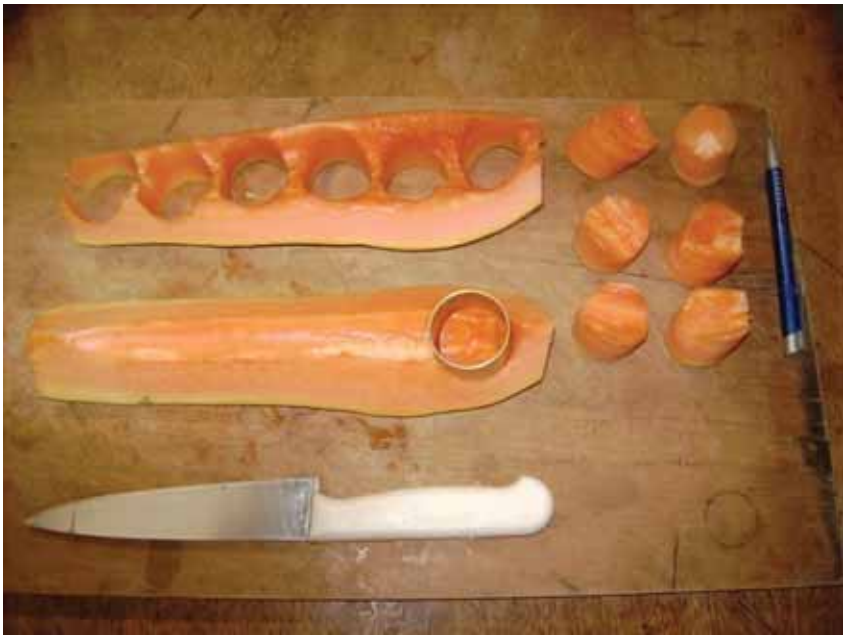

Figura 3. Corte seccional dos mamões no diâmetro de 3,6 cm.

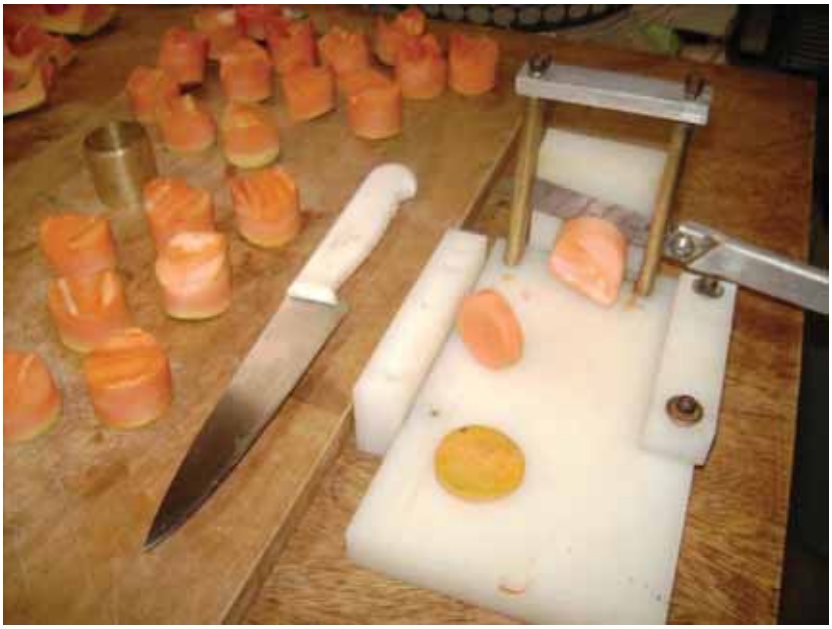

Figura 4. Corte das fatias de mamões na espessura de 1,0 cm.

Figura 5. Armazenamento das amostras de frutas para utilização nos experimentos.

3. Branqueamento térmico

O branqueamento das fatias de mamão foi realizado em cesto de aço inoxidável (Figura 6) por imersão em água fervente (98,3 °C em São José do Rio Preto) por 2 min com posterior resfriamento em água corrente por 1 min.

Figura 6. Cesto contendo as fatias de mamão a serem branqueadas.

4. Desidratação Osmótica

O açúcar utilizado nos ensaios de DO foi a sacarose (açúcar refinado comercial), adquirida em mercado local. L (+)-ácido láctico grau alimentício em solução 85% e lactato de cálcio penta-hidratado grau alimentício em pó da PURAC Sínteses – Brasil foram adicionados às soluções desidratantes na concentração de 0,1 M e 1% p/p, respectivamente.

Os ensaios de DO foram conduzidos em uma cuba de aço inoxidável com camisa externa de circulação de água (Figura 7) a 27 °C, bombeada a partir de um banho ultratermostatizado. As soluções foram mantidas sob agitação constante de 1000 rpm conduzida por um agitador mecânico. Na cuba foram dispostos 4 cestos de nylon contendo 2 compartimentos cada um, que continham as amostras de frutas branqueadas ou não (Figuras 8 e 9).

Foram utilizadas soluções nas concentrações de 40, 50 e 60% p/p de sacarose. Os tempos de processamento foram de 60, 120 e 180 min para os ensaios de com frutas frescas; e de 30, 105 e 180 min para os ensaios com mamões branqueados.

Os ensaios de equilíbrio foram conduzidos em recipientes de vidro com tampa (Figura 10), contendo 600 g de solução osmótica, os quais foram inseridos em incubadora refrigerada (Marconi, modelo MA 830/A) com plataforma de agitação orbital (117,5 rpm) e temperatura controlada (27^{o}C) (Figura 10). Aproximadamente 50 g de fatias de mamão foram desidratadas, em duplicata, por 48 h nas soluções a 50 e 60% e por 72 h nas soluções a 40%.

Figura 7. Cuba de DO contendo solução osmótica.

Figura 8. Cestos de nylon contendo as amostras.

Figura 9. Cuba de DO contendo os cestos de nylon com as amostras.

Figura 10. Recipiente de vidro e incubadora utilizados nos ensaios de equilíbrio.

Após os tempos de processamento, as fatias foram retiradas das soluções, lavadas em água destilada por 10 s e secadas em papel absorvente (Figura 11).

Figura 11. Amostras sendo secadas em papel absorvente.

5. Cobertura Comestível

Utilizou-se pectina amidada (grau de amidação: 0,17) de baixa metoxilação (grau de metoxilação: 0,34) GRINDSTED® LA 210 (Danisco - Brasil) a 2% p/p para recobrir as fatias de mamão fresco e desidratado osmoticamente. A solução foi preparada a 70 °C. As frutas foram dispostas em cestos e imersas na solução de pectina mantida a 40 °C em um banho termostatizado (Figura 12). Solução a 2,8% p/p de lactato de cálcio penta-hidratado grau alimentício em pó da PURAC Sínteses – Brasil foi usada como agente gelificante por imersão (30 s) das fatias cobertas (Figura 13). Em seguida, as amostras foram lavadas em água destilada por 30 s para remover o excesso de cobertura (Figura 13) e dispostas sobre tela de nylon até sua utilização nos experimentos (Figura 14).

Figura 12. Disposição das fatias de fruta em cesto e imersão na solução de pectina mantida a 40 °C.

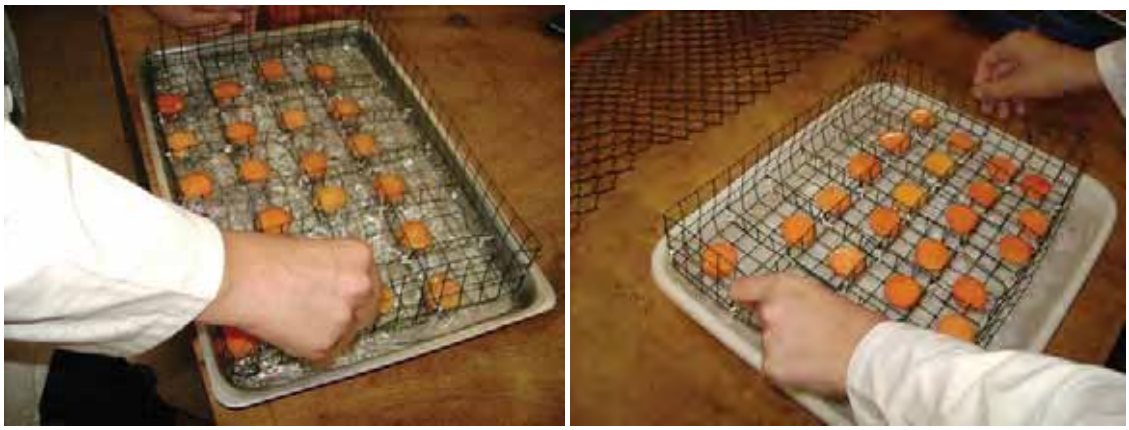

Figura 13. Gelificação da cobertura por imersão em solução de lactato de cálcio e lavagem das amostras em água destilada.

Figura 14. Disposição das amostras cobertas em tela de nylon.

6. Secagem convectiva

Aproximadamente 330 g de amostras foram secas em secadores convectivos a 60 e 70 °C com velocidade do ar de 1,0 $m.s^{-1}$ por tempo suficiente para atingir atividade de água suficientemente baixa para garantir a segurança microbiológica das amostras após a secagem (a_w < 0,6). Dois experimentos foram realizados em paralelo a cada temperatura para trabalhar com o mesmo lote de frutos frescos. O ar fluiu paralelamente às amostras que estavam dispostas em bandejas metálicas (Figura 15), as quais foram pesadas a cada 20 min durante a primeira hora de experimentos e a cada 30 min após esse tempo (Figura 15). Após as pesagens foi realizada a rotação das bandejas no secador.

Figura 15. Disposição das amostras em bandejas dentro do secador convectivo e pesagem.

7. Métodos analíticos

7.1. Determinação dos Sólidos Totais

Os sólidos totais foram determinados em triplicata a 60 °C em estufa a vácuo, 10 kPa, até atingir peso constante.

7.2. Determinação dos Açúcares

Os açúcares totais e redutores foram determinados em triplicata por titulação de oxi-redução (WILLIAM, 1970) em equipamento Redutec (TECNAL – Brasil).

7.3. Determinação da Atividade de Água

A atividade de água das amostras frescas e processadas foi determinada em triplicata a 25 °C em equipamento modelo Aw Sprint da Novasina (Axair Ltd. – Switzerland).

7.4. Determinação da Cor

Nos ensaios para avaliar as melhores condições de DO das frutas frescas, a cor foi determinada em replicata (6 amostras) com câmera digital instalada em cabine de luz apropriada (Figura 16) e determinação dos parâmetros L*, a* e b* com auxílio do software acadêmico (V-01 LensEye) para análise de cor (LUZURIAGA et al., 1997). Foram utilizados os padrões laranja (Figura 16a) e laranja amarelado (Figura 16b).

Figura 16. Cabine de luz e padrões para análise de cor.

Nos experimentos seguintes a cor das amostras foi determinada em um espectrofotômetro modelo ColorFlex (HunterLab, Estados Unidos), adquirido após o início dos estudos desta tese. Aproximadamente seis fatias de frutas frescas ou processadas foram acomodadas em um copo de vidro, de maneira a não sobrar espaços não cobertos por amostras, e tampadas. O aparato de vidro foi coberto por um copo plástico preto para a leitura da cor das amostras (Figura 17).

Figura 17. Espectrofotômetro ColorFlex, aparato de vidro com tampa e copo plástico.

O software Universal versão 4.10 foi o utilizado nas análises do ColorFlex, com as configurações: iluminante D65, observador a 10° e leitura dos valores absolutos de L*, a * e b*.

A comparação das respostas de L*, a* e b* obtidos pelas duas diferentes metodologias, permitiu verificar que o padrão utilizado para a análise de cor pelo software LensEye, padrão laranja (L* = 62,661; a* = 36,067; b* = 57,096), não era o mais adequado para a avaliação dos valores de L* e b* das amostras. Assim, testando-se vários padrões de cor, concluiu-se que para a avaliação dos valores de L* e b*, o padrão laranja amarelado (L* = 71,941; a* = 19,363; b* = 67,857) seria o mais adequado, e que, para a análise de a*, seria o padrão laranja (L* = 62,661; a* = 36,067; b* = 57,09).

7.5. Determinação da Textura

A textura das amostras foi determinada em replicata (7 a 9 amostras) utilizando o método TPA (Texture Profile Analysis), em um texturômetro Universal (TA-XT2i Texture Analyser, Stable Micro System, Surrey, UK). Amostras cilíndricas (10 mm de altura e área transversal de 260,155 mm^2), foram comprimidas individualmente até 30% de deformação por 5 s a uma taxa de 1 $mm.s^{-1}$, através de uma ponta de prova acrílico de 35 mm de diâmetro. Os resultados foram expressos como dureza.

7.6. Determinação da Vitamina C

A vitamina C das fatias frescas e processadas de mamão foi determinada em duplicata por titulação conforme metodologia proposta pela AOAC modificada por Benassi e Antunes (1988). 25 g de amostras foram homogeneizadas em 50 mL de solução extratora (ácido

oxálico a 2% p/p) em equipamento Turratec (TECNAL, modelo TE-102) por 1 min. Uma alíquota de 20 g foi volumetricamente diluída com solução extratora a 50 mL e 10 mL da solução diluída foi titulada com 2,6-diclorofenolindofenol.

7.7. Determinação da Densidade

A densidade das fatias frescas e processadas de fruta foi determinada em duplicata por deslocamento de volume usando um kit da Gehaka (GEHAKA, modelo BK-300) acoplado à balança semi-analítica (Figura 18). Metanol foi utilizado como fluido de preeenchimento.

Figura 18. Kit Gehaka para determinação de densidade em balança semi-analítica.

7.8. Microscopia

Para as análises de microscopia de luz e eletrônica de transmissão, os cilindros de mamões frescos e processados foram cortados no sentido de sua altura em fatias com menos de 2 mm de espessura (Figura 19a), as quais foram cortadas longitudinalmente. Os cortes das superfícies das amostras (Figura 19b) foram preparados para as análises de microscopia.

As amostras cortadas foram fixadas em solução de glutaraldeído 4% por 24 hs e, em seguida, em solução 1% de tetróxido de ósmio por 2 hs. Após a lavagem das fatias com água destilada, procedeu-se à sua desidratação em soluções de acetona a 10, 20, 30, 40, 50, 60, 70, 80 e 90% por 15 min. A desidratação das amostras em acetona (100%) foi realizada em triplicata por 15 min. As amostras foram pré-infiltradas por 12 hs em solução de acetona/resina (Araldite 502) e, então, incluídas na resina de araldite previamente adicionada dentro de pequenos moldes de silicone para formar os blocos. Os moldes foram colocados em estufa a 60 °C por 48 hs para polimerização. Os cortes dos blocos foram realizados em micrótono Leica.

Para as análises de luz, as lâminas das diferentes amostras foram coradas com azul de toluidina em bórax a quente. Utilizou-se fotomicroscópio Olympus Bx 60 com câmera Olympus DP71, nos aumentos de 20X (escalas de 50 μm) e de 100X (escalas de 10 μm) para as análises.

Os cortes ultra-finos dos blocos pré-selecionados através da microscopia de luz foi realizado em ultra-micrótono Leica Ultracut UCT. Estes cortes foram contrastados por 20 min em acetato de uranila e citrato de chumbo e analisados em um microscópio eletrônico de transmissão Zeiss EM906 (Zeiss, Cambridge, England) a 80 kW.

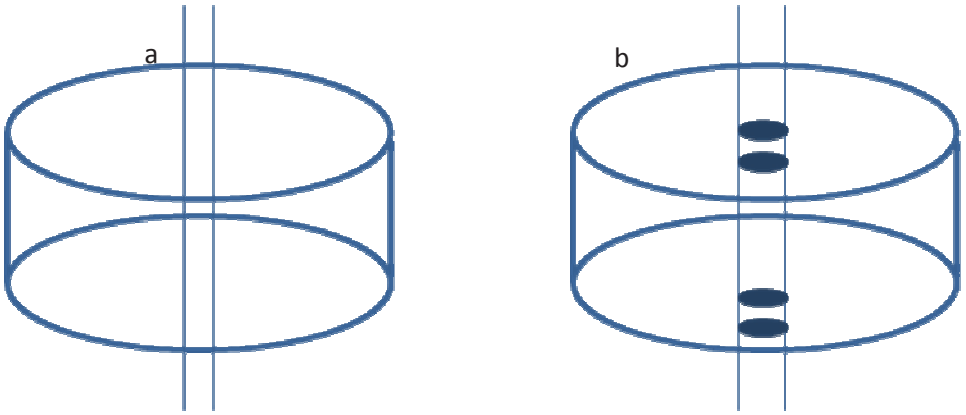

Figura 19. Cortes longitudinal (a) e transversal (b) das fatias de mamão.

8. *Isotermas de sorção*

A construção das curvas de isotermas foi baseada no método estático gravimétrico proposto por Jowitt et al. (1987). Aproximadamente 2 a 5 g de amostras frescas, branqueadas e desidratadas osmoticamente foram pesadas em cápsulas de plástico, em triplicata. As cápsulas de plástico contendo as amostras foram colocadas sobre uma estrutura de acrílico, acima da solução, dentro dos potes de vidro contendo as soluções salinas. As umidades relativas foram obtidas com o uso de soluções salinas saturadas correspondentes à faixa de atividade de vapor de água de 0,04 a 0,90. Os sais utilizados foram NaOH, LiCl, CH_3COOK, $MgCl_2$, K2CO_3, NaBr, $NaNO_2$, NaCl, KCl e $BaCl_2$. Formol comercial (40%) foi utilizado para limpeza dos recipientes de vidro e das estruturas em acrílico para evitar a presença de contaminantes, em especial fungos. Os frascos foram fechados hermeticamente e acondicionados em estufas com temperatura controlada (30, 50 e 70°C). Pesagens periódicas das cápsulas foram realizadas até as amostras entrarem em equilíbrio com o ambiente, com base na mudança do peso, que, expresso em base seca, não deverá exceder 0,1% (0,001 g/g

sólidos secos) num período de 72 hs. Sólidos totais foram analisados após as amostras atingirem equilíbrio.

APÊNDICE II:

CÁLCULOS DO CONTEÚDO E DA RETENÇÃO DA VITAMINA C

1. Cálculo do conteúdo de vitamina C das frutas frescas, branqueadas, cobertas e desidratadas osmoticamente

Para calcular o conteúdo de vitamina C nas frutas frescas, branqueadas, cobertas e desidratadas osmoticamente utilizou-se a Equação 1:

$$\frac{Vit\,C\,(g)}{100\,g\,amostra} = \left[\frac{V_{DCFI\,amostra}(mL)}{V_{DCFI\,padrão}(mL)} \frac{100\,(g)}{m_{reidratado}(g)} \frac{(m_{solvente} + m_{reidratado})(g)}{m_{extrato}(g)} \frac{50\,(mL)}{V_{alíquota}(mL)} \right] \quad (1)$$

em que: *Vit C* representa o conteúdo de vitamina C, em g/100 g de amostra; $V_{DCFI\ amostra}$ é o volume de 2,6-diclorofenolindofenol 0,01 % gasto na titulação da amostra; $V_{DCFI\ padrão}$ é o volume de 2,6-diclorofenolindofenol 0,01 % gasto na titulação do padrão de vitamina C (125 mg de ácido ascórbico P.A. diluídos em 50 mL de ácido oxálico 2% p/V); $m_{amostra}$ corresponde à massa da amostra, em g; $m_{solvente}$ corresponde à massa, em g, de 50 mL do solvente (aproximadamente 50 g); $m_{extrato}$ corresponde à massa de amostra, em g, tomada após a diluição em 50 mL de solvente (aproximadamente 20 g) e $V_{alíquota}$ é o volume de 2,6-diclorofenolindofenol 0,01 % utilizado nas titulações (5 mL).

2. Cálculo do conteúdo de vitamina C das amostras após a secagem convectiva

As frutas secas foram reidratadas em 20 mL de água destilada por 20 min para serem trituradas para a determinação do conteúdo de vitamina C. O teor de vitamina C das frutas após a secagem convectiva, apresentado nos Capítulos IV e V, foi determinado com base nos **sólidos totais**, considerando $m_{amostra} = m_{reidratado}$ (Equação 2).

$$\frac{Vit\,C\,(g)}{100\,g\,amostra} = \left[\frac{V_{DCFI\,amostra}(mL)}{V_{DCFI\,padrão}(mL)} \frac{100\,(g)}{m_{reidratado}(g)} \frac{(m_{solvente} + m_{reidratado})(g)}{m_{extrato}(g)} \frac{50\,(mL)}{V_{alíquota}(mL)} \right] (1-w) \quad (2)$$

em que: *Vit C* representa o conteúdo de vitamina C, em g/100 g de amostra; $V_{DCFI\ amostra}$ é o volume de 2,6-diclorofenolindofenol 0,01 % gasto na titulação da amostra; $V_{DCFI\ padrão}$ é o volume de 2,6-diclorofenolindofenol 0,01 % gasto na titulação do padrão de vitamina C (125 mg de ácido ascórbico P.A. diluídos em 50 mL de ácido oxálico 2% p/V); $m_{reidratado}$ corresponde à massa da amostra seca e da água adicionada, em g; $m_{solvente}$ corresponde à massa, em g, de 50 mL do solvente (aproximadamente 50 g); $m_{extrato}$ corresponde à massa de reidratado, em g, tomada após a diluição em 50 mL de solvente (aproximadamente 20 g); $V_{alíquota}$ é o volume de 2,6-diclorofenolindofenol 0,01 % utilizado nas titulações (5 mL) e *w* é a umidade da amostra seca, em base úmida.

3. Cálculo de retenção da vitamina C

3.1. Cálculo da retenção da vitamina C das frutas branqueadas e cobertas, secas ou não

A retenção da vitamina C foi determinada em relação à amostra **fresca**. Para as frutas cobertas e branqueadas, secas ou não, considerou-se que as amostras não ganharam nem perderam massa durante esses pré-processamentos. O conteúdo médio de vitamina C das amostras frescas, cobertas e branqueadas, antes e após a secagem, foi calculado segundo a Equação 1. Os resultados foram expressos em base seca e o valor obtido para as frutas pré-processadas, secas ou não, foi dividido pelo das amostras *in natura*. A Tabela 1 apresenta um modelo do cálculo da retenção da vitamina C para essas amostras.

Tabela 1. Modelo do cálculo de retenção da vitamina C para as amostras cobertas e branqueadas, secas ou não.

Amostra	Conteúdo médio de vitamina C			Retenção (%)**
	(mg/100 g amostra)*	%, (g/100 g amostra)	%, (base seca)	
Fresca	44,3	0,0443	0,459	-
Fresca seca	56,82	0,0568	0,381	83,03
Fresca-CO seca	65,64	0,0656	0,437	95,17

CO: amostra coberta com pectina amidada de baixa metoxilação.

*calculado segundo a Equação 1.

**em relação à amostra fresca.

3.2. Cálculo da retenção da vitamina C das amostras desidratadas osmoticamente, secas ou não

Durante a desidratação osmótica ocorre variação de massa, portanto, os cálculos de retenção da vitamina C devem considerar essas variações. A retenção da vitamina C durante a DO foi calculada segundo as Equações 3 e 4.

$$Ret_{DO} = 1 - Perda_{DO} \quad (3)$$

$$Perda_{DO} = \left(\frac{1 - \overline{Vit}_{DO}}{\overline{Vit}_{Fresca}} \right) (1 + \Delta M) \quad (4)$$

em que: Ret_{DO} é a retenção da vitamina C durante a DO; $Perda_{DO}$ é a perda da vitamina C durante a DO; $\overline{Vit}_{DO}$ é o teor médio de vitamina C nas amostras desidratadas osmoticamente, em g/100g de amostra; $\overline{Vit}_{Fresca}$ é o teor médio de vitamina C nas frutas frescas, em g/100 g de amostra e ΔM é a variação total de massa durante a DO.

Após a secagem das frutas desidratadas osmoticamente, a retenção da vitamina C foi calculada considerando a variação de massa total durante a DO e a secagem, segundo as Equações 5 e 6.

$$M_T = (1 + \Delta M_{DO})(1 + \Delta M_{Sec}) \quad (5)$$

$$Ret_{Sec} = \frac{M_T \cdot \overline{Vit}_{Sec}}{\overline{Vit}_{Fresca}} \quad (6)$$

em que: M_T é um adimensional que representa a fração de massa seca total em relação à massa inicial; ΔM_{DO} é a variação total de massa durante a DO; ΔM_{Sec} é a variação total de massa durante a secagem (tirada do secador); Ret_{Sec} é a retenção da vitamina C durante a secagem; $\overline{Vit}_{Sec}$ é o teor médio de vitamina C nas amostras desidratadas osmoticamente após a secagem, em g/100g de amostra; $\overline{Vit}_{Fresca}$ é o teor médio de vitamina C nas frutas frescas, em g/100 g de amostra.