JOÃO RENATO DE JESUS

MODELOS MATEMÁTICOS NA PREDIÇÃO DA CINÉTICA DE DESIDRATAÇÃO OSMÓTICA DE BATATA-DOCE

LAVRAS – MG

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

Orientador

Dr. Jefferson Luiz Gomes Corrêa

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APROVADA em 27 de fevereiro de 2015.

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RESUMO GERAL

Muitos estudos têm sido realizados visando o melhor entendimento dos mecanismos de transferência de massa durante a desidratação osmótica e a modelagem dos fenômenos envolvidos durante o processo. É um pré-tratamento que consiste na imersão de alimentos em uma solução hipertônica, ocasionando dois fluxos em contracorrente, um de perda de água do alimento para a solução e um segundo de incorporação de sólidos da solução pelo alimento. A batata-doce (*Ipomoea batatas* (L.)) é uma das cinco culturas de maior importância no suprimento de calorias e minerais em países em desenvolvimento, porém o estudo de desidratação osmótica nessa raiz tuberosa ainda é escasso. O presente trabalho foi elaborado com o objetivo de estudar o comportamento de fatias de batata-doce em processos de desidratação osmótica, utilizando-se sorbitol, sacarose e frutose como agentes osmóticos, em diferentes condições de atividade de água da solução e modelar a cinética de difusão de água e sólidos. Através das curvas de cinética de desidratação osmótica, observou-se que a sacarose foi muito efetiva na perda de água e a frutose forneceu maiores valores de ganho de sólidos e maior redução na atividade de água do produto. Soluções mais concentradas (menores valores de atividade de água) apresentaram maiores valores de perda de água e ganho de sólidos. As difusividades efetivas de água e solutos foram calculados de acordo com a solução analítica da Segunda Lei de Fick, considerando o encolhimento que ocorre durante o processo e sem essa consideração. Foram também calculadas as difusividades segundo os modelos propostos por Azuara e Barbosa Júnior. Em geral, os modelos apresentaram altos valores de coeficiente de correlação para os agentes osmóticos sacarose e sorbitol. A difusividade efetiva da água ficou na faixa de $3{,}60 \times 10^{-11}$ a $4{,}32 \times 10^{-10}$ $m^2\ s^{-1}$ e para sólidos ficou entre $2{,}20 \times 10^{-11}$ e $1{,}26 \times 10^{-10}$ $m^2\ s^{-1}$. Os dados experimentais de encolhimento como função do teor de umidade foram ajustados a sete diferentes modelos empíricos, sendo que a abordagem polinomial foi a que apresentou melhor ajuste. Além disso, houve o ajuste dos dados de encolhimento como função do tempo, sendo o modelo exponencial proposto adequado. Observou-se menor encolhimento em amostras tratadas com frutose e, independente da atividade de água da solução e maior encolhimento, foi observado quando a sacarose foi utilizada como agente osmótico.

Palavras-chave: *Ipomoea batatas* (L.), modelos empíricos, agentes osmóticos, encolhimento.

GENERAL ABSTRACT

Many studies have been conducted in order to better understand the mass transfer mechanisms the during osmotic dehydration and modeling of the phenomena involved in this process. It is a pretreatment consisting of the immersion of foods in a hypertonic solution, causing two counter-current fluxes, one of water loss from food to the solution and a second of incorporation of solids to solution from the food. The sweet potato (*Ipomoea batatas* (L.)) is one of the five most important cultures in supplying calories and minerals in developing countries, however, the study in osmotic dehydration of this root is scarce. The present work was elaborated with the objective of studying the behavior of sweet potato slices behavior during osmotic dehydration processes using sorbitol, sucrose and fructose as osmotic agents, in different conditions of water activity of the solution, and of modeling the diffusion kinetics of water and solids. By means of the kinetic curves of osmotic dehydration, we observed that sucrose was very effective in water loss and fructose provided higher solid gain and larger reduction of water activity of the product. More concentrated solutions (lower values of water activity) presented higher water loss and solid gain. The effective diffusivities of water and solutes were calculated according to the analytical solution of Fick's second law, considering the shrinkage that occurs during the process, and without this regard. We also calculated the diffusivities according to the models proposed by Azuara and Barbosa Junior. In general, the models showed high coefficient of correlation values for sucrose and sorbitol as osmotic agents. The effective diffusivity of water ranged from 3.60×10^{-11} to 4.32×10^{-10} m² s^{-1} and for solid, from 2.20×10^{-11} to 1.26×10^{-10} m² s^{-1}. The experimental shrinkage data as moisture content were adjusted to seven different empirical models, with the polynomial approach presenting the best adjustment. Moreover, we also adjusted the shrinkage data as function of time, with the exponential model proposed being appropriate. We observed lower shrinkage in samples treated with fructose, regardless of water activity of the solution and higher shrinkage was observed when sucrose was used as osmotic agent.

Keywords: *Ipomoea batatas* (L.), empirical models, osmotic agents, shrinkage.

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LISTA DE SIGLAS

DO	Desidratação osmótica
DOPV	Desidratação osmótica com pulso de vácuo
FAO	Food and Agriculture Organization
IBGE	Instituto Brasileiro de Geografia e Estatística
IG	Índice glicêmico
GS	Ganho de sólidos
MHD	Mecanismo hidrodinâmico
MW	Molecular weight
NMC	Normalized moisture content
OD	Osmotic dehydration
PA	Perda de água
RMSE	Root mean square value
SG	Solid gain
TACO	Tabela Brasileira de Composição de Alimentos
TSS	Total soluble solids
WL	Water loss

LISTA DE SÍMBOLOS

mg Miligramas

g Gramas

kHz Quilohertz

MHz Megahertz

X_0 Teor de umidade inicial (g água/100g de amostra)

X_t Teor de umidade no tempo t (g água/100g de amostra)

x_{st} Teor de sólidos no tempo t (g sólido/100g de amostra)

x_{s0} Teor de sólidos inicial (g sólido/100g de amostra)

t Tempo (s)

MR Teor adimensional de água

SR Teor adimensional de sólidos

i Número de termos da série

D_{eff} Difusividade efetiva ($m^2\ s^{-1}$)

L Comprimento característico

X_{eq} Quantidade de água no equilíbrio (g água/100g de amostra)

x_{seq} Quantidade de sólidos no equilíbrio (g sólido/100g de amostra)

D_{effw} Difusividade efetiva de água ($m^2\ s^{-1}$)

D_{effs} Difusividade efetiva de sólidos ($m^2\ s^{-1}$)

k_1 e k_2 Constantes do modelo de Peleg

PA_{eq} Perda de água no equilíbrio (g água/100g de amostra)

GS_{eq} Ganho de sólidos no equilíbrio (g sólido/100g de amostra)

PA_t Perda de água no tempo t (g água/100g de amostra)

GS_t Ganho de sólidos no tempo t (g sólido/100g de amostra)

S_1 Constante relacionada à perda de água ou ganho de sólidos no modelo de Azuara

$D_{eff\,(t)}$ Difusividade efetiva variável com o tempo ($m^2\ s^{-1}$)

$PA_{eq\ mod}$ Perda de água no equilíbrio obtida segundo o modelo proposto por Azuara (g água/100g de amostra)

$GS_{eq\ mod}$ Ganho de sólido no equilíbrio obtido segundo o modelo proposto por Azuara (g sólido/100g de amostra)

a_w Atividade de água

ºBrix Teor de sólidos solúveis

cm Centímetros

min Minutos

M_0 Peso inicial da amostra (g)

M_t Peso da amostra no tempo t (g)

$t_{1/2}$ Tempo de meia-vida (s)

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PRIMEIRA PARTE

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PRIMEIRA PARTE

1 INTRODUÇÃO GERAL

A batata-doce (*Ipomoea batatas* (L.)) é uma das culturas mais importantes das regiões tropicais e subtropicais com diversas formas de uso. É considerada de grande importância econômico-social, participando do suprimento de calorias e minerais na alimentação humana, principalmente das populações de baixa renda (ANTONIO et al., 2008; AZEVEDO et al., 2014).

Atualmente, a batata-doce (*Ipomoea batatas* (L.)) tem sido amplamente consumida por atletas e praticantes de musculação, uma vez que apresenta carboidratos de baixo índice glicêmico. Auxilia no emagrecimento, além de possuir fibras e potássio, que estimulam o intestino e auxiliam no controle da glicemia e do colesterol.

Após a colheita, esta raiz tuberosa é altamente perecível, devido à sua elevada atividade de água. Tem um curto período de vida para utilização adequada. O processamento é uma importante forma de conseguir um melhor e mais amplo aproveitamento, diminuindo o percentual de perdas.

O processo de desidratação representa a redução do teor de água, e da atividade de água dos alimentos. Por isso, é uma operação muito importante na obtenção de produtos que apresentem maior estabilidade e maior vida útil, além de uma manutenção das características químicas, sensoriais e nutricionais do produto.

A desidratação osmótica (DO) consiste na remoção parcial da água a partir de tecidos celulares pela imersão de um alimento em uma solução aquosa hipertônica. Durante o processo osmótico, a transferência de massa, em tecidos de plantas é complexa, sendo influenciada por diversos fatores, como estrutura da parede celular, composição química, temperatura, agitação, dentre outros.

A maioria das pesquisas consideram a DO sob aspectos macroscópicos, regidos pela Lei de Fick com a estimativa de coeficientes médios de difusão da massa (DERMESONLOUOGLOU; POURGOURI; TAOUKIS, 2008; GARCÍA-SEGOVIA et al., 2010; MERCALI et al., 2011). Para uma melhor representação da difusividade, o modelo matemático deve buscar retratar as condições experimentais, tanto com relação ao processo, quanto com relação às características do produto e as alterações que o mesmo sofre durante o processo. Desta forma, o coeficiente de difusão deve ser expresso como uma função da umidade da amostra, além de considerar-se as alterações na espessura e no volume (encolhimento) dos produtos submetidos à desidratação (PORCIUNCULA et al., 2013).

Neste trabalho, objetivou-se estudar a desidratação osmótica de batata-doce com a obtenção da cinética do processo com três agentes osmóticos (sacarose, frutose e sorbitol) e em duas condições de atividade de água da solução. Modelos matemáticos foram utilizados para o ajuste da cinética de perda de água e ganho de sólidos. Especial enfoque foi dado ao encolhimento da raiz durante a DO e a modelos que consideram a alteração do volume neste processo.

2 REFERENCIAL TEÓRICO

2.1 Batata-doce

A batata-doce (*Ipomoea batatas* (L.)) é uma raiz tuberosa possivelmente originária das Américas Central e do Sul, sendo encontrada desde a Península de Yucatam, no México, até a Colômbia. É uma cultura perene que serve como fonte de nutrientes para alimentação humana e animal, além de ser uma importante matéria-prima industrial (AHN et al., 2010).

Pertence à família *Convolvulaceae*, e ao genêro *Ipomoea*. Esta família apresenta cerca de 1000 espécies, sendo a batata-doce a única cultura de importância na alimentação. A batata-inglesa pertence à família *Solanaceae* e não está diretamente relacionada à batata-doce, apesar de serem designadas por termos semelhantes.

A batata-doce é cultivada em regiões tropicais e subtropicais, apresentando-se como uma cultura altamente tolerante a altas temperaturas, solos pobres, inundações, além de ser resistente a doenças e pragas.

Brasil é o principal produtor do continente latino-americano de batata-doce. Em 2013, a área total cultivada foi de cerca de 39.393 hectares, com produção média de 505.350 toneladas e rendimento médio de 13,1 toneladas por hectare (INSTITUTO BRASILEIRO DE GEOGRAFIA E ESTATÍSTICA, 2014). A batata é cultivada em todas as regiões, embora esteja mais presente nas regiões sul, sudeste e nordeste, notadamente nos estados do Rio Grande do Sul, São Paulo, Paraíba e Sergipe (INSTITUTO BRASILEIRO DE GEOGRAFIA E ESTATÍSTICA, 2014). No Estado de Minas Gerais foram produzidas em 2013 aproximadamente 30.999 t, em 1.966 hectares e rendimento médio de 15,7 toneladas por hectare (INSTITUTO BRASILEIRO DE GEOGRAFIA E ESTATÍSTICA, 2014).

De acordo com a Food and Agriculture Organization (FAO), a China é o maior produtor mundial de batata-doce, tendo alcançado em 2013, uma produção de 79.090.068 toneladas (71,41% da produção mundial) (FOOD AND AGRICULTURE ORGANIZATION, 2014). A batata-doce é a quinta cultura mais importante nos países em desenvolvimento, depois do arroz, trigo, milho e mandioca (ELAMEEN et al., 2007).

A batata-doce é consumida de diversas maneiras, mas principalmente na sua forma fresca, além de ser matéria-prima em processos industriais na obtenção de amido, pães, doces, farinhas, flocos e féculas (SANTOS; SOUSA;

SANTOS, 2009). Além disso, a partir do amido da batata-doce é possível a produção de etanol, apresentando como uma alternativa viável devido ao alto rendimento do tubérculo (em relação à cana-de-açúcar) e possibilita a inclusão social do agricultor familiar (PAVLAK et al., 2011).

A composição centesimal da batata-doce (Tabela 1), assim como a de demais hortaliças, varia significativamente de acordo com o tipo de solo, o clima, a cultivar e condições pós-colheita.

Tabela 1 Composição centesimal da batata-doce

Componentes ($g\ 100\ g^{-1}$)	(LEONEL; CEREDA, 2002)	TACO (2011)
Umidade	67,73	69,5
Proteínas	1,33	1,3
Lipídios	0,35	0,1
Fibras	1,39	2,6
Cinzas	1,32	0,9
Carboidratos	27,88	28,2

Quimicamente, a batata doce tem aproximadamente 31% de matéria seca, sendo, em média, 92% de carboidratos. A sacarose é o açúcar mais abundante na batata-doce *in natura.* Apresenta também pequena quantidade de glicose e frutose. Devido à sua relativa abundância, os carboidratos aumentam o seu valor calórico, sendo, o amido, a principal fonte de reserva (FONTES et al., 2012). É excelente fonte de vitaminas A, C, B2, B6 e E, bem como fibras dietéticas, minerais e apresenta baixo conteúdo de lipídeos (SHEKHAR et al., 2015). Dentre os minerais mais abundantes, destacam-se cálcio, ferro e potássio com 21; 0,4 e 340 mg $100g^{-1}$, respectivamente (NÚCLEO DE ESTUDOS E PESQUISAS EM ALIMENTAÇÃO, 2011).

Nutricionalmente, apresenta-se como alimento com baixo índice glicêmico. O índice glicêmico ou IG é um parâmetro cinético que reflete a capacidade dos carboidratos contidos nos alimentos em aumentar a glicose no sangue *in vivo* quando consumidos. Recentes experimentos clínicos relatam uma relação positiva entre consumos de alimentos que apresentam baixos índices glicêmicos e a redução de doenças crônicas, como a diabetes. Também, é muito apreciada por praticantes de atividades físicas, uma vez que liberam energia mais lentamente no sangue, fazendo com que o treino seja mantido por mais tempo (AMARAL, 2014; CHIU et al., 2011).

2.2 Desidratação osmótica

A desidratação osmótica (DO) consiste na imersão do alimento em uma solução hipertônica. Devido à diferença de concentração entre o agente osmótico e o alimento, são criados dois fluxos simultâneos em contra-corrente, através das membranas celulares: um da água que sai do alimento para a solução – o mais importante do ponto de vista da desidratação – e outro de soluto da solução para o alimento (SAGAR; SURESH-KUMAR, 2010). Há ainda, um terceiro fluxo de lixiviação de alguns solutos naturais (açúcares, ácidos, minerais, entre outros nutrientes) do produto para a solução de imersão, que, embora seja insignificante, pode ser importante para a qualidade sensorial (aroma, cor, textura) e nutricional do produto (NAHIMANA et al., 2011). É um dos pré-tratamentos mais empregados em processos na secagem de frutas e hortaliças, visando a obtenção de produtos com teores intermediários de umidade e atividade de água (CORRÊA et al., 2011; FALADE; SHOGAOLU, 2010; MAYOR; MOREIRA; SERENO, 2011).

A DO auxilia na manutenção das características físicas, químicas, nutricionais e sensoriais do produto desidratado, além de reduzir o tempo necessário à obtenção do produto seco, proporcionando assim uma economia energética (KAUSHAL; SHARMA, 2014). Durante o processo, ocorrem perdas de alguns nutrientes, como minerais e vitaminas, por lixiviação, além de incorporação do agente osmótico ao produto. Porém, apesar dessas alterações, o pré-tratamento proporciona vantagens durante as etapas posteriores de secagem, em relação ao processo sem pré-tratamento (AKTAS et al., 2007).

O produto de DO é parcialmente desidratado, sendo necessário um processamento posterior visando sua conservação como a secagem convectiva, secagem por micro-ondas e congelamento (CIURZYŃSKA; LENART; GRĘDA, 2014; CONTRERAS et al., 2008; SILVA; FERNANDES; MAURO, 2014).

Tem sido amplamente utilizada como pré-tratamento de alimentos perecíveis como goiabas (CORRÊA et al., 2010), abacaxi (CORRÊA et al., 2011), tomates (RIBEIRO, 2013), banana (PORCIUNCULA et al., 2013), yacon (FERNANDES; RODIGUES, 2011; MENDONÇA, 2014) e pequi (FIGUEIRA, 2014). É possível afirmar que a DO é uma alternativa para o aproveitamento do excesso de produção, além de possibilitar o consumo do produto nos períodos de entressafra. Portanto, o tratamento osmótico tem se apresentado como ferramenta tecnológica importante para o desenvolvimento de novos produtos derivados de frutos e vegetais, com valor agregado e propriedades funcionais (BELLARY; SOWBHAGYA; RASTOGI, 2011; DUARTE et al., 2012).

2.2.1 Variáveis do processo e características do produto

As taxas de perda de água e ganho de sólidos pelo produto dependem, de variáveis do processo e de características do produto. Rastogi et al. (2002) descrevem os parâmetros de processamento que interferem diretamente na transferência de massa durante a DO. Dentre esses fatores, destacam-se: a influência da temperatura do processo, da concentração do agente osmótico empregado e seu peso molecular, do grau de agitação, da proporção alimento:solução osmótica e do tempo de imersão.

A temperatura da DO tem uma importante influência na cinética, bem como na qualidade do produto final. O aumento da temperatura faz com que ocorra maior remoção de água e um decréscimo no tempo de tratamento. A taxa de desidratação aumenta com o aumento da temperatura, uma vez que, sob condições de altas temperaturas, ocorre um aumento na mobilidade da água, favorecendo a sua retirada. No entanto, um aumento na temperatura pode causar degradação e perda de compostos termossensíveis (CLEMENTE et al., 2014).

A concentração da solução osmótica e o tipo de soluto utilizado influenciam diretamente nas taxas de transferência de massa durante o processo de desidratação. Em geral, soluções osmóticas de maior concentração, apresentam uma maior pressão osmótica, favorecendo a retirada de água dos produtos. Uma maior diferença entre a concentração da solução e do produto cria dois fluxos simultâneos em contracorrente, através das paredes das células, uma de água, que se move para a solução, e o outro de sólidos a partir da solução para o produto, até que o equilíbrio de potencial químico seja estabelecido entre a solução e as células (ABRAÃO et al., 2013).

O peso molecular do agente osmótico interfere nos parâmetros de transferência de massa (perda de água e incorporação de sólidos). O uso de um agente osmótico com maior peso molecular pode provocar uma diminuição no ganho de sólidos e o aumento na perda de água, favorecendo a perda de massa. Carboidratos de baixo peso molecular, como a glicose e frutose, favorecem o ganho de sólidos devido à alta taxa de penetração das moléculas do soluto (KOTOVICZ et al., 2014; RUIZ-LÓPEZ et al., 2011).

A agitação afeta positivamente a saída de água do produto durante o processo. Isso indica a presença de uma resistência externa à transferência de massa da água na solução osmótica, provavelmente relacionada à sua viscosidade, sugerindo que a retirada de água não seja controlada apenas por um mecanismo interno de difusão. A utilização de maiores proporções de solução osmótica minimiza o efeito da diluição da solução, aumentando a eficiência da desidratação (NIETO et al., 2013; TONON; BARONI; HUBINGER, 2006).

Com relação ao tempo de imersão, diversos autores destacam que a maior perda de água durante a DO ocorre nos primeiros 120 minutos do produto (SILVA; FERNANDES; MAURO, 2014). Porém, deve-se avaliar cada processo, pois as transferências podem continuar em proporções relevantes após este período, até que o estágio de equilíbrio seja atingido (FERRARI et al., 2011). Quando o tempo de imersão do produto na solução for muito grande, pode haver um aumento da perda de moléculas bioativas, nutrientes e vitaminas solúveis, como vitamina C, que podem se difundir na solução osmótica juntamente com a perda de umidade. O efeito do tempo na DO pode ser explicado pela modificação do tecido vegetal, principalmente das membranas celulares que vão enfraquecendo com longos períodos de exposição à solução osmótica (KETATA; DESJARDINS; RATTI, 2013).

Dentre as características do produto que afetam o processo, destacam-se a compactação do tecido vegetal, sua porosidade e textura. Diferenças nas características do vegetal como massa inicial de substâncias não solúveis, enzimas presentes no produto, tamanho dos espaços intercelulares, presença de gás retido nos capilares e complexos de pectina e celulose e grau de geleificação, determinam a cinética do processo de desidratação. Fenômenos que modificam a permeabilidade dos tecidos da planta, como pré-tratamentos com substâncias químicas, como sulfitos, por exemplo, branqueamento ou congelamento, favorecem o ganho de sólidos em detrimento da perda de água (TORREGGIANI, 1993).

2.2.2 Tipos de DO

Para acelerar o processo de transferência de massa durante o processo osmótico, vários métodos têm sido aplicados, tais como utilização de energia ultrassônica, de pulso de vácuo e aquecimento ôhmico.

O pré-tratamento osmótico assistido por ultrassom envolve a imersão do alimento em água ou em uma solução aquosa hipertônica com aplicação de energia ultrassônica. O ultrassom é uma onda mecânica com uma frequência compreendida entre 20 kHz a 100 MHz, que pode propagar-se através de materiais sólidos, líquidos ou gasosos. O ultrassom de alta intensidade opera na faixa de kHz, promovendo a alteração das propriedades físicas e químicas de produtos alimentares. Em sistemas sólido-líquido, as ondas ultrassônicas induzem uma compressão e uma expansão do material, mecanismo normalmente referido como efeito esponja. Esse efeito influência diretamente a remoção de água do produto e a incorporação de sólidos. Além disso, são criados canais microscópicos no tecido dos frutos ou vegetais, afetando a transferência de massa e a permeabilidade da parede celular. Os gases contidos no interior dos poros sofrem uma expansão e são retirados, sendo esses espaços vazios preenchidos pela solução osmótica, o que aumenta a difusão de massa quando a desidratação osmótica assistida por ultrassom é utilizada. Dentre as vantagem da utilização dessa técnica, destacam-se a redução da degradação de componentes sensíveis à altas temperaturas, já que é realizado à temperatura ambiente e a não incorporação de sólidos ao produto quando a água destilada é utilizada como meio líquido de imersão (CÁRCEL et al., 2007; FERNANDES; RODRIGUES, 2011; NOWACKA et al., 2014).

A desidratação osmótica com pulso de vácuo (DOPV) consiste na aplicação de uma pressão reduzida a um sistema sólido-líquido, seguido da restauração da pressão atmosférica. Enquanto durante a DO conduzida à pressão atmosférica os mecanismos de transferência são baseados no gradiente de pressão osmótica entre a solução e o produto, na DOPV, além desse mecanismo, também ocorre um gradiente de pressão que é estabelecido através da utilização do vácuo. Como consequência, o processo, além de apresentar o transporte difusional, apresenta também um mecanismo hidrodinâmico (MHD) nos primeiros minutos, que auxilia na melhoria da transferência de massa graças à expulsão de gases e líquidos internos oclusos nos poros dos tecidos celulares, sendo substituídos pela solução osmótica. Dentre as vantagens desse processo, destacam-se a redução no tempo total do processo e a facilitação de impregnação controlada de compostos ativos no produto. A área de interface sólido-líquido e a transferência de massa entre as duas fases podem ser aumentadas, além de poder ser conduzido à temperatura ambiente (CORRÊA et al., 2010; FANTE et al., 2011; FITO et al., 2001; VIANA; CORRÊA; JUSTUS, 2014).

O aquecimento ôhmico é um processo térmico, no qual o calor é gerado internamente, através da passagem de uma corrente elétrica alternada por um material (como sistemas alimentares), que funciona como uma resistência elétrica. Em geral, os alimentos são condutores elétricos, graças à presença de inúmeros compostos de natureza iônica. Nos processos de aquecimento ôhmico, os componentes alimentares tornam-se partes do circuito elétrico através do qual os fluxos de correntes alternadas geram calor nos alimentos, de acordo com suas propriedades intrínsecas de resistência elétrica. O interesse na tecnologia de aquecimento ôhmico associada ao processo de DO baseia-se nas seguintes vantagens apontadas para esse processo: (i) uniformidade de aquecimento; (ii) aumento nas taxas de transferência de massa; e (iii) melhorias na qualidade final do produto e alterações mínimas nas características estruturais, nutricionais e sensoriais do produto (MORENO et al., 2011; MORENO et al., 2012).

2.2.3 Agentes osmóticos

A escolha do soluto é uma questão fundamental por estar relacionada às alterações nas propriedades sensoriais e ao valor nutritivo do produto final, além do custo de processo. Características do agente osmótico usado, como peso molecular e comportamento iônico, afetam significativamente a desidratação, tanto na quantidade de água removida quanto no ganho de sólidos (SAGAR; SURESH-KUMAR, 2010).

Se o objetivo da DO for a redução do teor de água, recomenda-se a utilização de solutos de alto peso molecular (como a sacarose), se o objetivo for a incorporação de sólidos, usualmente empregada na salga de produtos cárneos, solutos de natureza iônica e baixo peso molecular são indicados. Os agentes osmóticos mais comumente utilizados em processos de DO são a sacarose ($C_{12}H_{22}O_{11}$) e o cloreto de sódio (NaCl). Porém, outros tipos de agentes podem ser utilizados, como a frutose ($C_6H_{12}O_6$) e o sorbitol ($C_6H_{14}O_6$) (BROCHIER; MARCZAK; NOREÑA, 2014; HEREDIA et al., 2012; RUIZ-LÓPEZ et al., 2011; SCHMIDT; CARCIOFI; LAURINDO, 2009).

A sacarose é considerada um ótimo agente osmótico, devido à sua eficácia, conveniência e baixo preço, especialmente quando a DO é empregada como etapa preliminar à secagem convectiva, pois, por ser um inibidor enzimático da polifenoloxidase, previne o escurecimento enzimático e a perda de aromas. Esta prevenção é devida à presença de uma camada do dissacarídeo, formada na superfície do produto desidratado, que constitui um obstáculo ao contato com o oxigênio, minimizando ou impedindo o escurecimento enzimático, além da influência positiva sobre a manutenção de substâncias aromatizantes do alimento. Entretanto, devido à sua doçura, seu uso na desidratação de alguns tipos de vegetais é limitado (QI; LE MAGUER; SHARMA, 1998).

Segundo Ruiz-Lopes et al. (2011), a frutose é um monossacarídeo que apresenta moléculas menores, em comparação com a sacarose, apresentando maior mobilidade nos tecidos do fruto ou vegetal. O sorbitol é um poliól de baixo peso molecular com poder edulcorante utilizado como agente umectante na indústria. Apresenta sabor doce e agradável e, como agente osmótico, resulta em eficiente retirada de água e melhora a aceitabilidade sensorial do produto final. Devido à suas menores massas moleculares, a frutose o sorbitol favorecem a impregnação do alimento com o soluto em função da maior velocidade de penetração das moléculas no produto (ERBA et al., 1994).

2.3 Modelos matemáticos

A modelagem matemática é de extrema importância para avaliação do comportamento dos produtos durante a desidratação. Os modelos matemáticos podem ser divididos em dois grupos principais: os empíricos e os fenomenológicos. Os modelos empíricos geralmente são obtidos a partir de simples correlações matemáticas dos dados experimentais, e os seus parâmetros, normalmente, não possuem significado físico. Por sua vez, os modelos fenomenológicos consideram as etapas elementares de transferência de massa por difusão e convecção e os seus parâmetros, frequentemente, apresentam significado físico. Geralmente representam as tendências do processo, mesmo em condições diferentes das experimentais (COUTINHO et al., 2010; PARTHASARATHI; ANANDHARAMAKRISHNAN, 2014; SA-ADCHOM et al., 2011).

O tratamento matemático mais usual para a descrição do processo de transporte de massa, que tem a difusão como etapa principal, envolve o uso da equação de Fick da difusão para um sistema que não tem seu tamanho alterado durante o processo. Apesar de a desidratação causar encolhimento nas amostras, o habitual tratamento matemático dos resultados experimentais envolve a suposição de que o processo de secagem é controlado pela difusão interna em um sólido homogêneo e isotrópico sólido que não muda o seu tamanho. Baseado nestes pressupostos, o processo de difusão é normalmente descrito em termos de segunda lei de difusão de Fick (AGUERRE; TOLABA; SUAREZ, 2008; PORCIUNCULA et al., 2013).

O encolhimento que ocorre durante as etapas de secagem, afeta os parâmetros de transferência de calor e de massa, sendo um fator importante a ser considerado na modelagem. A maioria dos estudos considera um tamanho constante do produto durante a secagem, o que reduz a precisão dos modelos matemáticos. Assim sendo, a utilização de procedimentos, supondo que a espessura instantânea do sólido seja proporcional à quantidade de água evaporada, é uma alternativa viável para a obtenção de parâmetros com maior precisão (SILVA et al., 2014; PORCIUNCULA et al., 2013).

2.3.1 Modelo de difusão unidirecional

O modelo é baseado na equação de difusão unidirecional de Fick (CRANK, 1975), considerando a água, temos:

$$\frac{\partial X_t}{\partial t} = \frac{\partial}{\partial z}\left(D_{effw} \frac{\partial X_t}{\partial z} \right) \qquad (1)$$

Onde: X_t é o conteúdo de água no instante t, D_{effw} é a difusividade efetiva da água e *z* é uma coordenada genérica direcional.

Para a resolução da equação 1, utiliza-se condições iniciais e de contorno:

Como condição inicial das amostras a quantidade uniforme de água, isto é: X_t (z,o) = X_0. (2)

Para a utilização do modelo, considera-se o sólido com uma das três geometrias: cilindro infinito, esfera ou placa plana. Para a geometria de placa plana, considera-se o alimento como uma placa de espessura igual a 2L.

Desta forma, utiliza-se a condição de simetria de concentração em que no centro $\left.\frac{\partial X_t}{\partial t}\right|_{z=0} = 0$, e o teor de equilíbrio na superfície da amostra X(L,t) = X_{eq}.

Assim, a resolução de (CRANK, 1975) para a equação unidirecional de Fick torna-se:

$$MR = \left[\frac{8}{\pi^2} \sum_{i=1}^{\infty} \frac{1}{(2i+1)^2} \exp\left(-(2i+1)^2 \pi^2 D_{eff} \frac{t}{4L^2} \right) \right] \quad (3)$$

Onde: MR é o conteúdo adimensional de água; i é o número de termos da série; D_{eff} é o coeficiente de difusividade efetiva (m^2 s^{-1}); L é o comprimento característico (metade da espessura da amostra, em metros); e t é o tempo decorrido durante a secagem (s). A difusividade efetiva é obtida por estimativa não linear.

O conteúdo adimensional de água é dado pela expressão:

$$MR = \frac{X_t - X_{eq}}{X_0 - X_{eq}} \quad (4)$$

Onde: X_{eq} é o conteúdo de água no equilíbrio e X_0 é o conteúdo de água inicial.

A estimativa do conteúdo de água em condições de equilíbrio são baseadas no modelo de Peleg (PALOU et al., 1994), como demonstrado na equação abaixo (Eq. 5).

$$X_t \pm X_0 = \frac{t}{k_1 + k_2 t} \quad (5)$$

Onde k_1 e k_2 são parâmetros do modelo de Peleg calculados por regressão não-linear.

A constante k_2 está relacionada com o conteúdo de água no equilíbrio, de acordo com a equação 6.

$$X_{eq} = X_0 - \frac{1}{k_2} \tag{6}$$

De maneira análoga, a difusividade efetiva pode ser calculada para o conteúdo de sólidos. As substituições devem ser feitas na equação 1, substituindo X_t (conteúdo de água) por x_{st} (conteúdo de sólidos), sendo assim calculada a difusividade efetiva de sólidos (D_{effs}). Todas as condições feitas considerando o teor de água são também válidas para os sólidos. O conteúdo de sólidos adimensional (SR) e os valores de conteúdo de sólidos no equilíbrio são também calculados substituindo-se, nas equações 4 e 5, os valores de umidade iniciais no tempo t e no equilíbrio pelos conteúdos de sólidos iniciais (x_{s0}), sólidos no tempo t (x_{st}) e sólidos no equilíbrio (x_{seq}) respectivamente. Quando ocorre impregnação (ganho de sólidos), na equação 6, o sinal negativo deve ser substituído pelo sinal positivo.

2.3.2 Modelo de Azuara

Segundo Azuara et al. (1992), as equações utilizadas para descrever a cinética do processo osmótico são complexas e geralmente específicas para certas condições de processo e configurações geométricas, além de não predizerem o ponto de equilíbrio. Diante disso, os autores encontraram uma equação capaz de predizer a cinética de desidratação osmótica e o ponto final do equilíbrio, sem a necessidade de chegar, de fato, ao equilíbrio, utilizando apenas um curto período de processo. O modelo pode ser utilizado para caracterizar a desidratação osmótica de diferentes tipos de alimentos, sem restrições de geometria. Partindo-se de um balanço de massa no material que sofrerá desidratação, obtém-se as seguintes equações para a perda de água (PA) ou para o ganho de sólidos do produto (GS) em função do tempo, respectivamente (FERRARI et al., 2005).

$$PA = \frac{S_1 t (PA_{eq})}{1 + S_1 t} \qquad (7)$$

$$SG = \frac{S_1 t (SG_{eq})}{1 + S_1 t} \qquad (8)$$

Onde: S_1 é uma constante relacionada à perda de água ou ganho de sólido; PA_{eq} corresponde à perda de água no equilíbrio (g água/100g de amostra); e SG_{eq} corresponde ao ganho de sólidos, também no equilíbrio (g sólido/100g de amostra).

A partir da linearização das equações 7 e 8, obtém-se as seguintes:

$$\frac{t}{PA} = \frac{1}{S_1(PA_{eq})} + \frac{t}{PA_{eq}} \qquad (9)$$

$$\frac{t}{GS} = \frac{1}{S_1(GS_{eq})} + \frac{t}{GS_{eq}} \qquad (10)$$

Como as taxas de transferência de massa durante a desidratação osmótica são maiores no início do processo, é comum a utilização de versões simplificadas da equação de difusão de Fick, considerando um curto tempo de tratamento, regime transiente, difusão em um meio semi-infinito, concentração da solução osmótica constante e resistência externa à transferência de massa desprezível (FERRARI et al., 2005).

Esta equação é apresentada por Crank (1980)

$$\frac{PA_t}{PA_{eq}} = 2\sqrt{\left(\frac{D_{efft}}{\pi L^2}\right)} \qquad (11)$$

sendo PA_t a perda de água no tempo t.

Relacionando a equações 7 e 11, obtém-se uma expressão simples para calcular a difusividade da água a diferentes tempos:

$$D_{eff\,t} = \frac{\pi t}{4}\left[\left(\frac{S_1 L}{1+S_1 t}\right)\left(\frac{PA_{eq\,\mathrm{mod}}}{PA_{eq\,\mathrm{exp}}}\right)\right]^2 \tag{12}$$

onde $PA_{eq\ mod}$ é a perda de água obtida segundo a equação 7; e $PA_{eq\ exp}$ é a perda de água obtida a partir dos dados experimentais.

A difusividade média é calculada como uma média das difusividades obtidas em cada tempo (Equação 13)

$$D_{eff} = \frac{\sum_{i=1}^{N}\left(D_{eff\,t}\right)_i}{N} \tag{13}$$

De modo análogo, os valores de difusividade para sólidos são obtidos substituindo-se os valores de perda de água (PA) pelos valores de ganho de sólidos (GS) nas equações 11 e 12.

Para a predição dos valores de PA e GS no equilíbrio durante a desidratação osmótica de abóboras em solução ternária, Mayor et al. (2007) utilizaram o modelo proposto por Azuara et al (1992).

2.3.3 Modelo de Barbosa Júnior

Com o objetivo de considerar a difusividade variável com o tempo, Barbosa Júnior, Cordeiro e Hubinger (2013) propuseram uma modificação no modelo de Fick. Para tanto, na equação 3, os valores de difusividade efetiva foram considerados como variáveis com o tempo.

A condição de equilíbrio é obtida pela equação empírica proposta por Peleg (PALOU et al., 1994), sendo para perda de água descrita na equação 14

$$\left.\frac{dPA_t}{dt}\right|_{t=0} = \frac{1}{k_1} \tag{14}$$

Portanto, isso indica que existe uma relação entre as taxas de desidratação/impregnação e a força motriz do processo. O tempo de n-redução para a taxa de desidratação ($t_{1/2}$) é definido na equação 15:

$$\left.\frac{dPA_t}{dt}\right|_{t=t_{1/n}} = \frac{1}{n}\left.\frac{dPA_t}{dt}\right|_{t=0} \quad (15)$$

O processo começa com n = 1, e n = 2 é a taxa de "meia-vida" ($t_{1/2}$), isto é, o tempo necessário para redução na metade na taxa inicial de desidratação; quando n = ∞, o processo encontra-se em equilíbrio. Assim sendo, é formulada a equação 16.

$$t_{1/n} = \frac{k_1}{k_2}\left(\sqrt{n} - 1\right) \quad (16)$$

Onde, $t_{1/n}$ representa a relação entre PA_{eq} e a taxa inicial de perda de água.

Usando segunda lei de Fick, Crank (1975) sugere uma equação para a difusão em uma dimensão em placa plana, considerando uma quantidade infinita de solução osmótica, regime transiente e curtos períodos de tempo, que pode ser expressa por uma equação simplificada (AZUARA et al., 1992; CRANK, 1980).

$$\frac{PA_t}{PA_{eq}} = 2\sqrt{\frac{D_{efft}t}{\pi L^2}} \quad (17)$$

onde: D_{efft} é o coeficiente de difusividade instantâneo. Relacionando as equações acima apresentadas, D_{eff} (t) pode ser facilmente calculado em diferentes tempos (Equação 18).

$$D_{efft}(t) = \frac{\pi}{4}\left(\frac{L}{t_{1/n}/(\sqrt{n}-1)^{+t}}\right)^2 t \quad (18)$$

O significado físico dos parâmetros do modelo de Peleg ($t_{1/2}$) é a relação entre as constantes do modelo de Peleg. Finalmente, a partir das Equações 17 e 18:

$$\frac{PA_t}{PA_{eq}} = \left[\frac{t_{1/n}}{t(\sqrt{n}-1)} + 1\right]^{-1} \quad (19)$$

Para comparar a difusividade obtidos pelos modelos de difusão com o obtido pelo modelo de Barbosa Júnior et al. (2013), a difusividade média é estimada sobre o tempo em que X (t) vs √t são lineares de acordo com a Equação (20).

$$\overline{D_{eff}} = \frac{\int_0^t D_{eff}(t)dt}{\int_0^t dt} \qquad (20)$$

Procede-se de maneira similar para o cálculo de difusividade em sólidos, apenas substituindo os valores de perda de água (PA) pelos valores de ganho de sólidos (GS).

As difusividades efetivas são obtidas a partir de regressão não-linear, segundo o método de Quasi-Newton.

2.4 Encolhimento

O encolhimento é um fenômeno que ocorre durante a secagem da maioria dos produtos alimentícios, provocando alterações nas dimensões originais do material, relacionadas com as modificações ocasionadas em sua estrutura.

Os sistemas alimentares são materiais heterogêneos, podendo ser considerados como uma rede ou matriz sólida tridimensional contendo uma fase líquida, geralmente água. A estrutura particular do material e as características mecânicas dos seus elementos em equilíbrio definem o volume da amostra e determinam o seu tamanho e forma. Quando a água é removida do alimento, ocorre um desequilíbrio de pressão entre o interior do produto e a pressão externa, gerando tensões que levam ao encolhimento ou colapso do material, alterações na forma e fratura do produto (MAYOR; SERENO, 2004; NAHIMANA et al., 2011).

Durante a desidratação de sistemas alimentares, o encolhimento é um dos índices que caracterizam o desenvolvimento e as alterações da sua estrutura física do produto durante o processo. Deste modo, é necessário o acompanhamento de seu desenvolvimento, inclusive na predição dos perfis de perda de água e incorporação de sólidos durante a DO. Algumas propriedades do material, como porosidade, densidade, densidade da partícula, tamanho e distribuição dos poros são importantes para a quantificação da qualidade dos produtos alimentares desidratados e a caracterização de suas propriedades texturais (KOÇ; EREN; ERTEKIN, 2008; SOURAKI; GHAVAMI; TONDRO, 2014).

Em alimentos, três tipos de encolhimento podem ser observados: unidimensional (ocorrem em apenas uma dimensão), isotrópico (ocorre uniformemente em todas as direções), e anisotrópico ou arbitrário (não uniforme). Dentre os fatores que influenciam a magnitude do encolhimento durante a DO destacam-se para produtos alimentícios: o volume de água removido durante a operação, a taxa de secagem e a mobilidade da matriz sólida. O encolhimento aumenta com o volume de água removido, uma vez que quanto maior for a perda de água, maiores são as tensões de contração. Em alguns casos, o equilíbrio mecânico é atingido quando o encolhimento do material é igual o volume de água removida (MAYOR; SERENO, 2004; NAHIMANA et al., 2011).

Diversos modelos empíricos têm sido propostos para o encolhimento de frutas e vegetais durante operações de secagem. A modelagem é geralmente feita por modelos lineares e não-lineares. Contudo, alguns autores concluíram que a abordagem empírica não linear tem se mostrado eficaz e adequada para análise das alterações físicas do produto e o seu teor de umidade durante a desidratação (KOÇ; EREN; ERTEKIN, 2008; TOĞRUL; İSPIR, 2007).

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SEGUNDA PARTE
ARTIGO 1

Characterization and kinetics of osmotic dehydration of sweet potato (*Ipomoea batatas* (L.))

Running title: Effect of different osmotic agents on osmotic dehydration of sweet potato slices.

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Summary: Kinetics of osmotic dehydration and effects of sucrose, sorbitol and fructose impregnation of sweet potato (*Ipomoea batatas* L.) slices were investigated. Diffusivities of both water and solute were evaluated by using Crank's analytical solution to Fick's second law adapted to a semi-infinite plate geometry and according to Azuara and Barbosa Junior' s models. An analytical solution of Fick's second law was employed and the shrinkage effect was used to estimate the water and solid diffusion. Osmotic dehydration was carried out in six different treatments at constant temperature of 30 ± 0.5ºC. Two different water activities (0.900 and 0.940) of each osmotic agent (sucrose, sorbitol and fructose) were analyzed. All agents were effective on dehydration. Osmotic solution with higher water activity, presented less effect on mass transfer parameters (water loss and solid gain). The results showed that the water loss was greater when sucrose was employed, and the solid gain was lower. Fructose was the most effective on the water activity reduction. The effective diffusivities of water ranged from $3.60x10^{-11}$ to $4.32x10^{-10}$ m^2 s^{-1}, and solids ranged from $2.20x10^{-11}$ to $1.26x10^{-9}$ m^2 s^{-1}. When the shrinkage was considered lower water diffusivities and higher solid diffusivities values were obtained. All the the tested models presented good fit, but Barbosa Junior's model was the most suitable, with high R^2 values and low RMSE values. It was not capable to predict the D_{effs} when fructose was employed.

Keywords: Drying, Mass transfer, water activity.

1 Introduction

The sweet potato (*Ipomoea batatas* (L.)) is an excellent source of vitamins as well as dietary fibers, mineral and still has low lipid content. It is used for human consumption , animal feed and, to some extent, for industrial purposes (Mukhopadhyay et al. 2011; Azevedo et al. 2014). In the developing countries, it is the fifth most important crop after rice, wheat, maize and cassava (Rumbaoa et al. 2009). Brazil is the largest producer of sweet potatoes in the Latin American. In 2013, that country presented a cultivated area of about 39,393 hectares, with an average production of 505,350 t (IBGE, 2014). Like most vegetables, its shelf life is short and its storage, difficult. As a consequence, techniques for conservation are welcome.

Osmotic dehydration (OD) is a technique who provides partial water removal from a food product, with low energy consumption by been carried out at room or moderate temperatures. It is based on the immersion of pieces of fresh fruits or vegetables in a hypertonic solution. The process involves simultaneous counter-current water diffusion from the food to the solution and solute diffusion into the food, under the influence of osmotic pressure (Torreggiani 1993; Akbarian et al. 2014; Vieira et al. 2012).

OD is considered a pretreatment to many processes and preserves physical, chemical, nutritional, sensorial and functional properties of food with small changes on its integrity (Corrêa et al. 2011; Ferrari et al. 2011; Abbasi Souraki et al. 2012). Because of this, it can be an efficient complementary processing step to thermal dehydration process, before others drying techniques as convective (Silva et al. 2014), microwave (Botha et al. 2012), microwave-vacuum (Corrêa et al. 2011) and freeze drying (Ciurzyńska et al. 2014).

Solute choice and concentration depend on several factors, namely the effect on organoleptic quality properties, solute solubility and cell membrane permeability, which establish effect and cost. The osmotic agents employed during the process are responsible for quality traits, nutritional value, and for water loss and solid uptake of osmo dried vegetables. Sucrose, is the most used osmotic agent (mainly with fruits), since it is cheaper and has good results during dehydration. The addition of sucrose to the product, leds an increase in sweet flavor and caloric value of the dried product. There is another agents that can be employed like sorbitol and fructose. These agents found in fruits have a lower molecular weight than sucrose, sweet flavor and increase on caloric value are less pronounced too, and are good agents, aimed at reducing the product water activity (Ruiz-López et al. 2011; Toğrul & İspir 2007; Brochier et al. 2014; Rizzolo et al. 2007).

In the literature, there are few studies in relation to OD of sweet potato with non-ionic agents and considering the shrinkage that occurs during the process. Antonio et al. (2008) concluded that the ternary solution of sodium chloride and sucrose was the most effective in this process.

The aim of this study was to evaluate the kinetics of osmotic dehydration of sweet potatoes on different osmotic agents (sucrose, fructose and sorbitol) and fit three mathematical models with estimation of the water and the solids diffusion coefficients. An approach considering the variation occurred due to shrinkage was used for the analytical solution of Fick's law.

2 Material and methods

Characterisation of raw material

Sweet potato (*Ipomoea batatas* L.) Braslandia branca *cv.* used for the osmotic dehydration experiment were purchased in a local Market (Lavras, MG, Brazil) and stored in a refrigerator at 7±1ºC. The roots were selected based on their appearance, firmness and peel color. Fresh sweet potato was characterized with respect to chemical composition (moisture content, ether extract, ash, protein, crude fiber and carbohydrate) according to the methodology proposed by the AOAC (2005). Analyses of pH, total soluble solids (TSS) (Instituto Adolfo Lutz, 2008), water activity (Aqualab, Decagon Devices Inc., Pullman, WA, USA, 3-TE model) and color parameters (Minolta colorimeter CR 400, Japan) were also performed. All analyses were performed in three replicates.

Sample preparation

Sweet potato was washed, peeled and cut into slices of 2.00 cm length x 2.00 cm width x 0.50 cm thickness, with the aid of a stainless steel mold. The slices were immersed for 3 min in an aqueous citric acid solution 1% (w/v) to avoid undesirable changes (enzymatic browning) (Reis et al. 2012).

Osmotic solutions were prepared with distilled water and the osmotic agents, commercial sucrose, fructose and sorbitol at two different a_w, 0.940 and 0.900. The mass ratio between roots samples and osmotic solution was of 1:10 (w/w) in order to avoid significant changes on concentration of the solution during the experiments (Abraão et al. 2013).

Osmotic dehydration

Osmotic dehydration (OD) was performed at atmospheric pressure at 30.0 ± 0.5ºC. The osmotic agents used were sucrose, fructose and sorbitol. The solutions were prepared to give a final water activity of 0.940 and 0.900. The concentration (ºBrix) and the molecular weight (MW) of each solute are shown in Table 1.

Table 1 Molecular weight and concentration of osmotic agents at different water activities

Osmotic agent	MW [g mol^{-1}]	Concentration [ºBrix]	
		a_w=0.940	a_w=0.900
Sucrose	342.29	51.0	60.0
Fructose	180.16	40.2	52.1
Sorbitol	182.17	40.4	52.4

The samples were removed from solution at predetermined times (15, 30, 45, 60, 90, 120, 150, 180, 240 and 300 minutes). After this, the samples were immersed in a cold distilled water bath for 20 seconds to stop the osmotic process. The surface of the samples was gently dried with an absorbent paper, to remove the excess of solution (Fante et al. 2011; Viana et al. 2014). Finally, the samples were weighted and the moisture content was calculated according to AOAC (2005). All the experiments were performed in five replicates. Water loss (WL) and solid gain (SG) were calculated in accordance with Equations 1 and 2, respectively.

$$WL(\%) = \frac{(M_0 X_0) - (M_t X_t)}{M_0} x100 \quad (1)$$

$$SG(\%) = \frac{(M_t x_{st} - M_0 x_{s0})}{M_0} x100 \quad (2)$$

where: M_0 = weight of sample at time t= 0 (min); M_t = weight of sample at time t = t (min); X_0= initial moisture content, wet basis (w.b.); X_t = moisture content (w.b.) at time t = t (min); x_{st} = soluble solid content (w.b.) at time t= t(min) and x_{s0} = initial solid content wet basis (w.b.).

The reduction on thickness (shrinkage) was measured by a digital caliper. For each sample, there was five different points measured and the results were reported as average values.

Mathematical models

The experimental kinetics data of the WL and SG were fit by three mathematical models with estimation of the water and the solids diffusion coefficients. The tested models were the unidirectional diffusion model, based on Fick's 2nd law (Crank 1975), Azuara's model (Azuara et al. 1992) and the Barbosa Junior's model (Barbosa Júnior et al. 2013).

For the unidirectional diffusion model, based on Fick's 2nd law (Crank 1975), an approach considering the characteristic length variable during the process was employed, demonstrating the shrinkage that occurs during osmotic dehydration. The effective diffusivities were obtained using the non-linear regression (Quasi-Newton) from software Statistica 7.0® (Statsoft, Tulsa, OK), considering the expansion of the equation with five terms.

3 Results and discussion

Characterization of raw material

The results of characterization of fresh sweet potatoes are demonstrated in Tables 2 and 3. According to table 2, the sweet potato presents average moisture content of 68.13±1.93 g 100g^{-1} (w.b). This result is closer to obtained by Leonel and Cereda (2002) and by the Brazilian food composition table (2011).

Table 2 Centesimal composition of fresh sweet potato

Component (%)	Mean ± standard deviation (g $100g^{-1}$)
Moisture	68.130±1.93
Fat	0.310±0.02
Ash	0.961±0.04
Protein	3.460±0.14
Crude Fiber	0.976±0.10
Carbohydrate	26.157±1.22

Table 3 Physical and chemistry properties of fresh sweet potato

	Mean ± standard deviation
Water activity (a_w)	0.988±0.002
Soluble solids (°Brix)	9.33±0.40
L*	93.09±1.57
a*	-4.11±0.89
b*	21.28±3.02
pH	5.94±0.03

The sweet potato presents a high water activity (0.988±0.002). The water activity is an important parameter on fresh vegetables, once it is the site of many physicochemical and microbiological transformations that depend largely on the state of the water (Nieto et al. 2013; Chkir et al. 2015). The parameter that reflects the luminosity (L*) is high, indicating a strong trend to white coloring. The negative values of the parameter (a*) indicate weak tendency to green and positive values of (b*), indicate that the pulp has trend to yellow.

Kinetics of water loss (WL) and solid gain (SG)

The kinetics of WL and SG are shown in Figures 1 and 2. According to the figures, more concentrated osmotic solution (lower a_w) resulted in higher WL and SG. The samples osmotically dehydrated with solutions of a_w=0.940

(Figure 1(a)), presented lower WL than those treated with a_w=0.900 (Figure 1(b)), regardless of the osmotic agent employed.

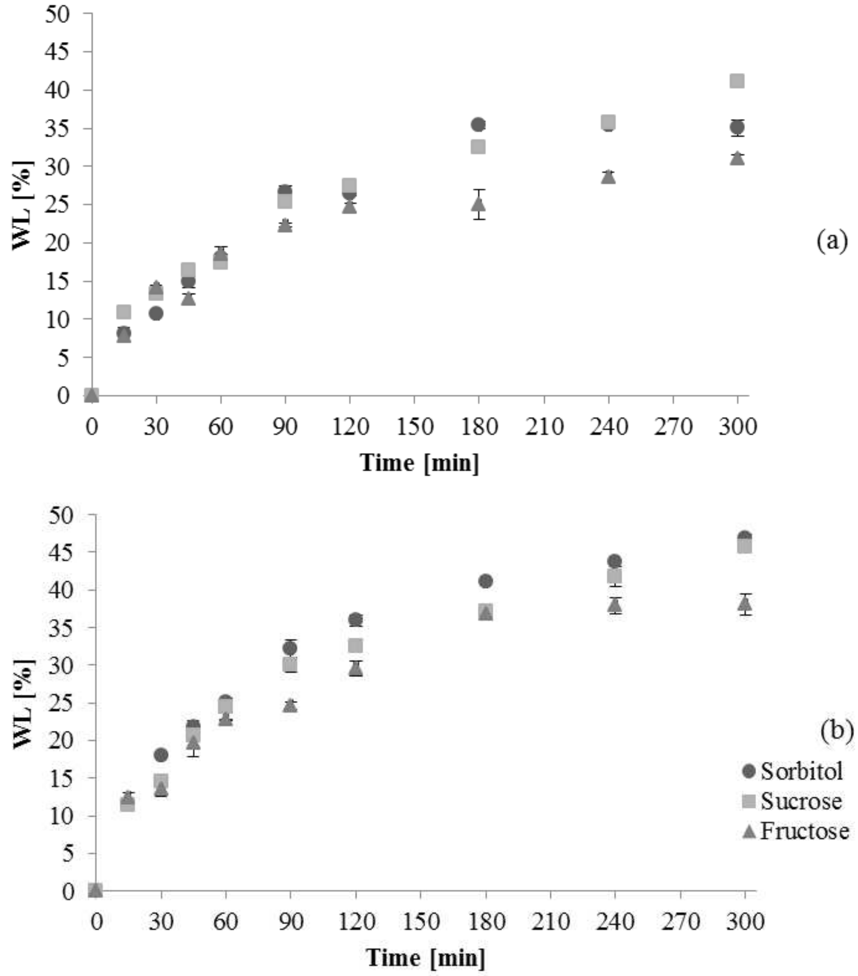

Figure 1 Kinetics of WL of sweet potatoes slices during osmotic dehydration by different osmotic agents. (a) a_w=0.940. (b) a_w= 0.900.

The WL was favored by higher solution concentrations, due to the increase of the osmotic pressure gradient between the food material and the osmotic solution. The presence of a large amount of solute causes a higher osmotic pressure that makes the WL easier (Pereira et al. 2006; Corrêa et al.

2010). The same phenomenon was also observed in the other works like the one of Mercali et al. (2011), using sucrose and sodium chloride on bananas. Silva et al. (2014) studying OD of pineapple in sucrose solution concluded that high WL occurred in the first two hours. Water loss was high at the beginning of the process and slowed down in later stages since the system was close to pseudoequilibrium. At initial times, osmotic driving force between the fresh potatoes and surrounding hypertonic solution was higher. This fact is also observed in this work for all osmotic agents.

According to the Figure 1, in the treatment of lower a_w, WL when sucrose or sorbitol were employed, were very close to and higher than when fructose was used.

Although the molecular weight of fructose and sorbitol are close, the two agents did not show the same effect on dehydration. In relation to fructose, from the 180 minutes, there is a tendency to equilibrium. High molecular weight solutes (as sucrose) were more easily blocked at the surface causing a greater osmotic concentration increasing the effect of dehydration during the osmotic process. Low molecular weight solutes (sorbitol and fructose) caused a gradual decrease in the concentration gradient due to rapid penetration of the solute and restricted water driving force, facilitating the rapid absorption of low molecular weight solutes. This process results in plasmolysis throughout the tissues, whereas high molecular weight solutes resulted in enhanced dehydration due to a marked difference in solute concentration between the surface and the interior of the product (Atarés et al. 2008; Chauhan et al. 2011).

According to El-Aouar et al. (2006), the WL was usually attributed to the influence of natural tissue membranes as well as the diffusive properties of water and solutes, as a function of their respective molecular weight.

Sucrose provided a higher WL, followed by sorbitol and fructose on solution’s water activity of 0.940. Between the osmotic agents, the molecules of sucrose present high molecular weight, and may remain in the intercellular space, improving the dehydration rather than impregnation phenomena (Kowalska et al. 2008).

In present work, when a_w was 0.900, the WL obtained by sorbitol was slightly higher than that observed for sucrose. The dehydration occurred when the osmotic agent was sorbitol was facilitated by the low molecular weight of the polyol. Moreover, the formation of a surface layer of sucrose also facilitated the WL.

As expected for OD process, WL was higher than SG. In the initial phase of the process, there was a significant water transfer from the vegetable to the solution, caused by the higher gradients of osmotic pressure (Porciuncula et al. 2013). Initially, for all the osmotic agents, the flow of solid inside the material was rapid for the first two hours, after that the flow of solid was gradually increased until the end of the process (Mayor et al. 2007). It was also found that the solid gain was increased with lower water activities of the different solutions (Figure 2).

When the water activity was 0.940, fructose presented higher solid gain, followed by sorbitol and sucrose. In solution with a_w=0.900, sucrose and sorbitol presented similar values of SG, lower than those obtained when fructose was employed (Figure 2). The gain of solids in the dehydration process is undesirable because it can impact changes in properties of the product. High SG affects negatively the quality and sensory characteristics of the dehydrated vegetable. When high levels of solids are infiltrated into the vegetable during osmotic dehydration, significant sensory alterations can occur, and the osmotically dehydrated product may present a different taste from the fresh (Rodrigues & Fernandes 2006).

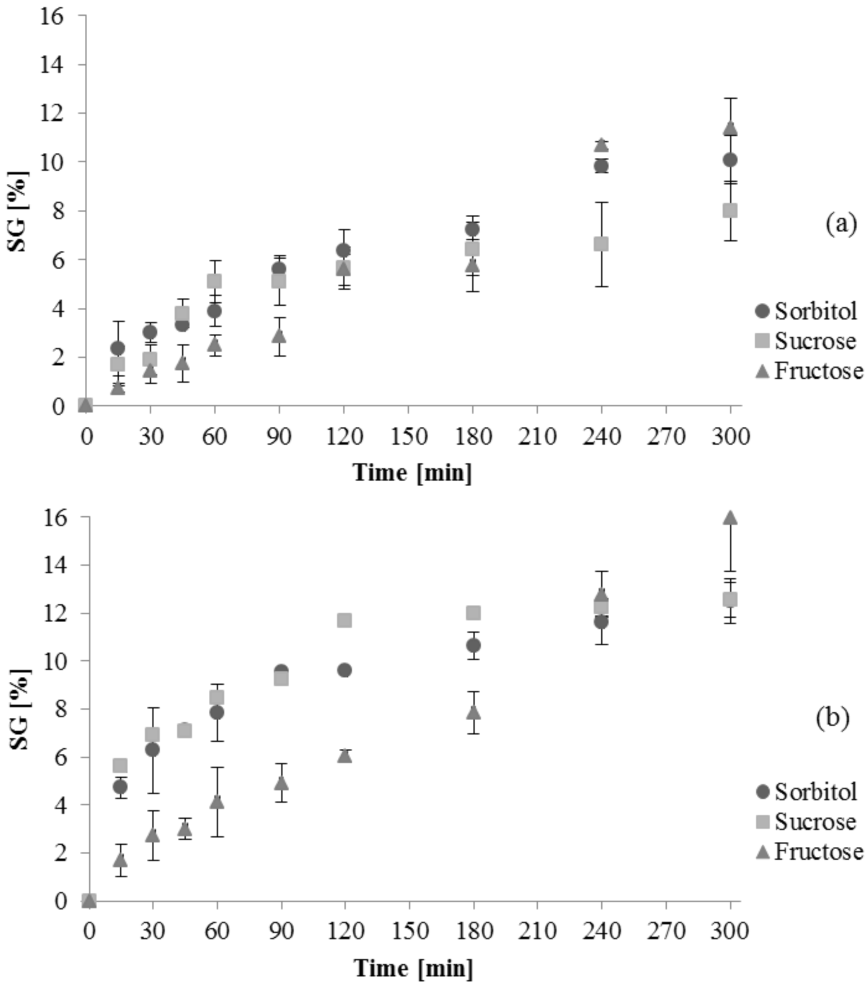

Figure 2 Kinetics of SG of sweet potatoes slices during osmotic dehydration by different osmotic agents. (a) a_w=0.940. (b) a_w= 0.900.

This study validates the result obtained by Ruiz-López et al. (2011) experiment, showing that the fructose provides biggest gain of solids than sucrose. Sritongtae, Mahawanich and Duangmal (2011) studying dehydration of cantaloupe slices, observed a higher gain of solids in the samples treated with sorbitol than those treated with sucrose. Increasing the molecular weight of the solute promotes high WL and low SG, favoring the weight loss and dehydration leading than incorporation of solutes.

At the end of the process, sucrose gave a lower SG than others solutes. It is well known that the sucrose promotes an increase on osmotic solution viscosity, which can lead to the formation of a boundary layer on fruit surface, limiting its impregnation on the product (Pereira et al. 2006).

The quality or goodness of the osmotic agent can be evaluated by different parameters, among them the capacity of decreasing the water activity in the dehydrated food. Figure 3, shows the development of the a_w for samples treated with different solutions, as a function of dehydration time. Osmotic dehydration of sweet potato in sorbitol, sucrose, and fructose solution reduced water activity in samples, independently on the kind of pretreatment applied.

By increasing the length of osmotic dehydration, the water activity showed a significant reduction. As the dehydration progresses, the driving force is reduced and the drop of a_w slow down. The best OD condition results from greater water loss and lower gain of solids and water activity (Silva et al. 2012; Corrêa et al. 2011; Atarés et al. 2008). Based on this concept, in the present study, it can be said that sucrose (regardless of concentration) is the agent which creates the best conditions for OD. This agent takes the lower solid gain and the higher water lost.

Once all the solutions employed have the same water activity, the molecular weight of each agent directly influenced the decrease of the a_w. Fructose has lower molecular weight, and provided a larger reduction in water activity of the product in both treatments, what made the slices less susceptible to microbiological reactions, but it is not effective on water lost like sucrose and sorbitol.

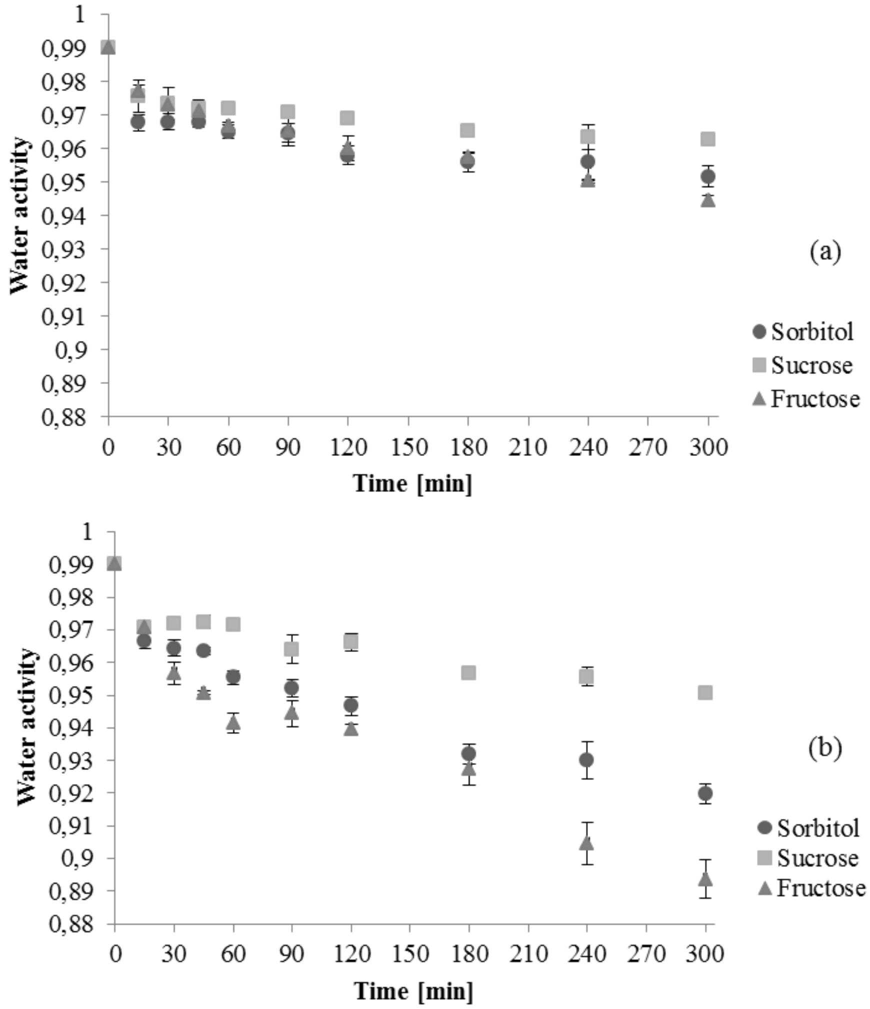

Figure 3 Kinetics of water activity reduction of sweet potatoes slices during osmotic dehydration by different osmotic agents. (a) a_w=0.940. (b) a_w= 0.900.

This agent also showed greater SG, which probably made it, too was highly effective in decreasing of a_w. In general way, it was also possible to note that for the mass transfer parameters analyzed, sorbitol had intermediate properties (WL, SG, a_w) to sucrose and fructose. Increasing concentrations of sucrose, sorbitol and fructose (lower a_w solution) led to an increase in WL, SG and a_w reduction.

Shrinkage during OD

The thickness ratio (L/L_0) of the samples during the OD was analyzed in function of water loss for the different treatments (Figure 4).

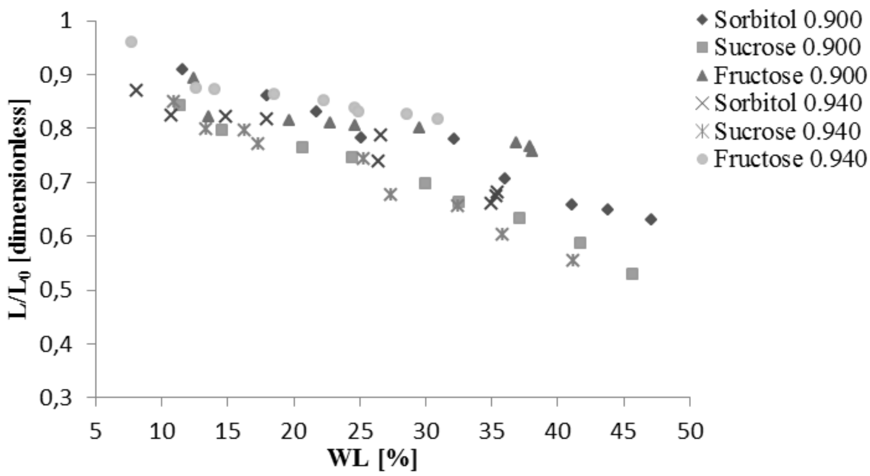

Figure 4 Thickness ratio in function of WL during OD

It was observed a linear decrease of thickness ratio with WL. A linear relationship between thickness ratio and moisture content was also observed by Souraki, Ghavami and Tondro (2014) in osmotic dehydration of apple in sucrose solution. They also observed that there was a reduction in thickness with WL increasing, and the reduction of moisture content, showing shrinkage.

According to Figure 4, the higher the WL, the higher the thickness. The higher shrinkage was observed for samples immersed in a sucrose solution at a_w= 0.900, with L/L_0 approximately equal to 0.52. The significant shrinkage points out the necessity to consider it in the modelling of osmotic dehydration.

According to the figures, it is possible to note that the kind of solute has more influence on thickness reduction than the water activity of the solution.

Regarding the osmotic agent, fructose solutions presented lower thickness reduction than other agents, regardless of the water activity. This fact can be explained by also less WL that this agent promotes during the OD. It is possible to note that lower thickness reduction was observed when sucrose was employed. This observation suggested that the thickness reduction is predominantly due to the amount of water removed, once sucrose presented a higher WL than sorbitol and fructose (Figure 1).

Osmotic dehydration modelling

For osmotic dehydration processes, several models have been proposed. A diffusional model is one of the most employed models to explain mass transfer during osmotic dehydration under certain assumptions. In general, effective coefficients of diffusion for water and osmotic solutes are obtained. These apparent diffusivities represent overall mass transport properties, including molecular diffusion (Seguí et al. 2010; Mayor et al. 2007).

The OD kinetics were adjusted by the models in the literature: unidirectional diffusion model, based on Fick's 2nd law (Crank 1975) with five terms, Azuara’s model (Azuara et al. 1992) and the Barbosa Junior’s model (Barbosa Júnior et al. 2013). The adjustment provided the diffusion coefficients of water and solids (Tables 4-7). In the tables we also observed statistical parameter: determination coefficient (R^2) and root mean square error (RMSE) for nonlinear equations.

Table 4 Effective diffusion coefficients of water (D_{effw}) in sweet potato slices during OD with and without considering shrinkage

Treatment	D_{eff} x10^{10} (m^2s^{-1})	R^2	RMSE	D_{eff} x10^{10} (m^2s^{-1})	R^2	RMSE	WL_{eq} (%)
	With shrinkage			Without shrinkage			
Sor(0.900)	0.84	0.9677	0.0315	2.08	0.9716	0.0598	58.82
Sor(0.940)	0.60	0.9565	0.0393	1.10	0.9425	0.0320	48.78
Suc(0.900)	0.36	0.9703	0.0406	0.64	0.8931	0.0380	55.37
Suc(0.940)	0.62	0.9661	0.0432	1.53	0.9775	0.0497	51.54
Fru(0.900)	1.00	0.9813	0.0296	1.64	0.9777	0.0322	46.73
Fru(0.940)	1.20	0.9650	0.0430	5.75	0.9597	0.0462	34.74

Table 5 Effective diffusion coefficients of solids (D_{effs}) in sweet potato slices during OD with and without considering shrinkage

Treatment	D_{eff} x10^{10} (m^2s^{-1})	R^2	RMSE	D_{eff} x10^{10} (m^2s^{-1})	R^2	RMSE	SG_{eq} (%)
	With shrinkage			Without shrinkage			
Sor(0.900)	12.16	0.9000	0.0859	5.05	0.9648	0.0509	13.26
Sor(0.940)	1.80	0.9663	0.0344	0.83	0.9484	0.0426	17.16
Suc(0.900)	12.64	0.9089	0.0829	6.22	0.9554	0.0581	13.91
Suc(0.940)	5.88	0.8803	0.0898	3.07	0.9437	0.0615	9.19
Fru(0.900)	2.72	0.8150	0.1059	0.98	0.7835	0.1371	20.41
Fru(0.940)	2.48	0.8265	0.0999	0.86	0.8020	0.1067	15.99

The effective diffusivities of water ranged from 3.60 x 10^{-11} to 1.20 x 10^{-10} m^2 s^{-1} when shrinkage was considered as a variable of the process. Without shrinkage, it ranged from 6.40 x 10^{-11} to 5.75 x 10^{-10} m^2 s^{-1}. As shown in Table 4, the values of effective diffusivities with shrinkage consideration are lower than those without considering this phenomenon. This fact demonstrates that Fick's second law of diffusion without considering shrinkage overestimates the transference of water by diffusion. This fact was also observed by Ponkham et al. (2012) and Souraki, Ghavan and Tondro (2014) studying dehydration of pineapple and apple respectively.

When the variation of thickness was considered in OD, lower diffusivities of water and higher diffusivities of solute were obtained. Antonio et al. (2008) studied osmotic dehydration of sweet potato in ternary solution of sucrose and NaCl, and the effective diffusion coefficients obtained from Fick's equation ranged from 3.82 x 10^{-10} to 7.46 x 10^{-10} m^2 s^{-1} for water and from 1.18 x 10^{-9} to 3.38 x 10^{-10} m^2 s^{-1} for solid. Even though, the diffusivity varies with the conditions of the experiments and it is difficult to compare (Corrêa et al. 2010), the range of the diffusivities obtained in the present work are near from the ones obtained by Antonio et al. (2008).

De Souza Silva et al. (2011) also reported similar results when pure sucrose solutions was employed, the coefficients of water diffusivity tended to decrease when the concentration of the osmotic solution increased (lower a_w), for fructose, this fact can also be observed, but not for sorbitol.

According to Table 5, D_{effs} ranged from 1.80 x 10^{-10} to 1.26 x 10^{-9} m^2 s^{-1}, considering shrinkage as variable during process. Without shrinkage, it ranged from 8.30 x 10^{-11} to 6.22 x 10^{-10} m^2 s^{-1}. For diffusivity of solids, a different behavior was observed with the inclusion of shrinkage as a variable in the process. In the model in which the shrinkage was used, higher values of diffusivity were observed. Toĝrul and Íspir (2007) studying OD of apricots in five different osmotic agents, found that effective diffusivity of solids with shrinkage is higher than those without shrinkage. When the change of thickness was considered in OD, the higher diffusion rates for solute were obtained.

Generally, the treatments performed in a higher water activity of solution, presented lower diffusivities of solid. It is because they are less concentrated than those with lower a_w. In solution with a_w=0.900, sucrose presented higher values, followed by sorbitol and fructose. When a_w=0.940, sucrose still presented high values, but sorbitol values was lower than fructose ones. Toĝrul and Íspir (2007) studying OD of apricots employing fructose, sucrose and sorbitol syrups as osmotic agents in a concentration of 70% (w/w), concluded that when shrinkage was used, sucrose presented a higher D_{effs}, but when the shrinkage was not employed, sorbitol presented high values. The WL and SG at equilibrium are obtained according to Peleg' model.

The results of Azuara's model parameters and diffusivity are demonstrated in Table 6 and 7, for WL and SG respectively.

Table 6 Diffusivity coefficient of water according to Azuara's model

Treatment	D_{effw}x10^{10} (m^2s^{-1})	R^2	RMSE	WL_{eq} (%)
Sor (0.900)	2.45	0.9970	0.1060	57.03
Sor (0.940)	1.95	0.9746	0.1764	47.24
Suc (0.900)	2.22	0.9904	0.1030	55.36
Suc (0.940)	1.91	0.9670	0.1111	50.36
Fru (0.900)	2.67	0.9848	0.1208	46.70
Fru (0.940)	2.76	0.9835	0.0910	35.87

Table 7 Diffusivity coefficient of solids according to Azuara's model

Treatment	D_{effs}x10^{10} (m^2s^{-1})	R^2	RMSE	SG_{eq} (%)
Sor (0.900)	3.93	0.9927	0.0555	13.59
Sor (0.940)	1.42	0.9183	0.0737	13.27
Suc (0.900)	0.41	0.9932	0.2336	13.92
Suc (0.940)	0.96	0.9505	0.1432	9.32
Fru (0.900)	0.30	0.7076	0.0896	31.37
Fru (0.940)	0.22	0.9600	0.0155	11.72

It is possible to note in Tables 6 and 7, that the model proposed by Azuara et al. (1992), can predict the osmotic dehydration kinetics satisfactorily. It is a suitable for this case (with these osmotic agents, concentrations and product), reflecting in higher values of R^2. The RMSE values were higher than those observed according to diffusional model. Diffusivities values were higher than those obtained according to Fick's law. D_{effw} ranged from 1.91 x 10^{-10} to 2.76 x 10^{-10} m^2 s^{-1}, and D_{effs} ranged from 0.22 x 10^{-10} to 3.93 x 10^{-10} m^2 s^{-1}. The water diffusivity was higher when fructose was employed (as observed in accordance with unidirectional model). In this model, solid diffusivity was higher when sorbitol was employed.

Comparison of diffusivities reported in the literature is difficult because of the different estimation methods and models employed together with the variation in food composition and physical structure (El-Aouar et al. 2003). Fructose and sorbitol (molecular weight = 180.16 g mol^{-1} and 182.17 g mol^{-1}) would be expected to exhibit higher diffusivities than sucrose (molecular weight = 342.29 g mol^{-1}) because of their smaller molecular size theoretically allowing a greater mobility in food media (Kowalska et al. 2008). Sucrose solutions have the greatest viscosities and mass concentrations in comparison with fructose or sorbitol, what might be able to explain why the lowest water diffusivities were estimated when sucrose was used as osmotic agent (an increased external resistance to mass transfer) (Ruiz-López et al. 2011). Water loss and solid gain at equilibrium were estimated according to Azuara's model.

The results of Barbosa Júnior's model parameters and diffusivity are demonstrated in table 8 and 9.

Table 8 Diffusivity coefficient of water according to Barbosa Junior's model

Treatment	$D_{effw}x10^{10}$ (m^2s^{-1})	R^2	RMSE	WL_{eq} (%)	$t_{1/2}$ (min)
Sor (0.900)	3.75	0.9928	0.0129	55.27	29.18
Sor (0.940)	3.39	0.9782	0.0079	51.51	37.75
Suc (0.900)	3.93	0.9972	0.0084	57.26	31.90
Suc (0.940)	3.42	0.9796	0.0079	48.60	40.22
Fru (0.900)	4.08	0.9795	0.0191	47.25	27.17
Fru (0.940)	4.33	0.9783	0.0040	35.62	24.10

Table 9 Diffusivity coefficient of solids according to Barbosa Junior's model

Treatment	$D_{effs}x10^{10}$ (m^2s^{-1})	R^2	RMSE	SG_{eq} (%)	$t_{1/2}$ (min)
Sor (0.900)	5.54	0.9686	0.0073	13.78	14.56
Sor (0.940)	3.91	0.9628	0.0018	9.24	64.68
Suc (0.900)	5.38	0.9839	0.0047	13.15	13.40
Suc (0.940)	2.42	0.9713	0.0004	15.14	29.50

This model presented good fit and lower RMSE values for WL in all treatments, but was not suitable for predict the kinetic of SG when fructose was employed. The prediction of SG in this osmotic agent was over estimated. Diffusivities values were higher than those obtained according to diffusional model and Azuara's model. D_{effw} ranged from 3.39 x 10^{-10} to 4.33 x 10^{-10} m^2 s^{-1}, and D_{effs} ranged from 2.42 x 10^{-10} to 5.54 x 10^{-10} m^2 s^{-1}. Fructose exhibited higher values of water diffusivity. Solutions of a_w=0.900 presented higher solids diffusivity.

The half-life of dehydration rate ($t_{1/2}$) ranged from 24.10 to 40.22 min. For impregnation process, the half-life rate ranged from 13.40 to 64.68 min. According to Barbosa Júnior et al. (2013) this parameter can be used as a criterion to define the osmotic process time, once it is the time required for a half reduction in dehydration/impregnation initial rate. The values at equilibrium were obtained according to descript in Barbosa Junior's work.

The values of effective diffusion coefficients of all models possess the same order of magnitude and among the tested models, Barbosa Júnior's model has higher R^2 values (0.97) and lower RMSE.

4 Conclusion

High values of WL and SG were observed in the first two hours of the osmotic process. All agents had a good effect on osmotic dehydration of sweet potato, sucrose was the best osmotic agent, with higher water loss and lower solids gain. Fructose showed greater efficiency in reducing the a_w of samples but it contributes with higher solids uptake. In this case, although the shrinkage has occurred significantly, its inclusion in the model for calculating the diffusivity coefficient was not adequate. All the models presented good fit. Between the models tested, Barbosa Junior's showed a good fit for water, with high R^2 values and low standard error, but was not capable to predict the D_{effs} when fructose was employed.

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ARTIGO 2

Volumetric shrinkage of sweet potato (*Ipomoea batatas* (L.)) during osmotic dehydration: osmotic agent influence and modelling

Running title: Effect of different osmotic agents and modelling during osmotic dehydration considering shrinkage.

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(artigo a ser submetido para publicação no International Journal of Food Science and Technology).

Summary: The osmotic dehydration is a common pretreatment for food drying. Even though the shrinkage during the process has been mentioned in several works, the mathematical treatment of this phenomenon is not current. In this work was studied the kinetics of shrinkage of sweet potato in osmotic dehydration by measuring the volumetric ratio during 300 minutes of process. The osmotic dehydration was performed in two different water activities (0.900 and 0.940) of the osmotic solution and with three different osmotic agents: fructose, sorbitol and sucrose. An exponential model was proposed in order to correlate the volume ratio and the time of the process. The reduction in volume was also related to moisture content ratio and modelled by using six mathematical models from the literature. It was showed that the shrinkage is higher for solutions with lower water activity. The shrinkage was higher for sucrose, followed by sorbitol and fructose. Comparisons were based on the coefficient of determination (R^2), reduced chi-square (χ^2) and root mean square error (RMSE). In general way, all the tested models presented good fit, and a polynomial approach was considered the best. An exponential model was very suitable for this process, presented high values of R^2.

Keywords: Dimensionless volume, sucrose, sorbitol, fructose

1 Introduction

Sweet potato (*Ipomoea batatas* (L.)) is the seventh food crop in the world. It is source of energy and phytochemicals for human and animal feed (Shekhar et al. 2015).

Osmotic dehydration (OD) is a partial dehydration process commonly used for fruits and vegetables. In the OD process, the food is immersed in a hypertonic solution (Silva et al. 2012; Souraki et al. 2013) and the cell membrane acts as a semi-permeable film. Two main fluxes, namely, the diffusion of water from the tissue to the osmotic solution due to osmosis and the uptake of solute from osmotic solution to the product happen in counter-current (Herman-Lara et al. 2013). During the dehydration, the product presents a volume reduction, once its water content is decreasing. This is due to mechanical disequilibrium caused by the removal of water. The wet product is generally regarded as a mix of two phases: a solid matrix and water in mechanical equilibrium (Katekawa & Silva 2007).

With the remotion of water from the interior of the solid matrix, the difference between the external and internal pressure leads to the development of strains with alterations in shape and dimensions, characterizing the shrinkage (Mayor & Sereno 2004; Nahimana et al. 2011). Food materials, considered as multicomponent and multiphasic systems, are organized in microstructural elements. The structural modifications during the dehydration are function of the food properties and of the process. Therefore, the mass transfer and the shrinkage are interrelated (Seguí et al. 2010; Seguí et al. 2012).

The shrinkage affects the sensorial quality of the product with loss of natural substances. The quantification of this phenomenon is important for a better understand of the dehydration process (Toğrul & İspir 2007; Nahimana et al. 2011; Moreira et al. 2008). Da Silva et al. (2014) in a study of OD in guavas, proposed a model that considers the shrinkage and variable effective moisture diffusivity, and observed that for experimental conditions, this model presented a good fit, and is therefore possible to predicts the moisture and sucrose distributions at any stipulated time.

Toĝrul and Ispir (2007) studied the variation in density and shrinkage of apricots during its OD with different osmotic agents and found that the shrinkage of apricots could be well correlated with the moisture content of the sample during OD. They concluded that mathematical modeling of food systems can provide better understanding of the structural changes (porosity, pore size and volume) during the process. In the calculation of the diffusion coefficients of water and solids, most of works does not consider the changes in structure (such as shrinkage) that occur during dehydration, providing inconsistent results with the reality of the phenomenon.

The objectives of this work were to investigate the shrinkage during OD of sweet potatoes employing three different osmotic agents, and to fit different mathematical models to shrinkage values as a function of moisture content and time.

2 Material and methods

Material

Sweet potato (*Ipomoea batatas* (L.)) Braslandia branca *cv.* used for the osmotic dehydration experiments were purchased in a local Market (Lavras, MG, Brazil) and stored in a refrigerator at 7±1ºC. The roots were selected based on their appearance, firmness and peel color. The average initial moisture content was 70% (w. b.), gravimetrically measured using a vacuum oven at 70ºC for 24 h (AOAC, 2005).

Preparation of samples and osmotic solution

The roots were washed, handly peeled and cut into slices of 2.00 cm length x 2.00 cm width x 0.50 cm thickness, with the aid of a stainless steel mold and then, they were subjected to osmotic treatment.

Six different osmotic treatments were tested: a_w= 0.940 binary solutions of sucrose (342.29 g mol^{-1}), sorbitol (182.17 g mol^{-1}) and fructose (180.16 g mol^{-1}) and a_w= 0.900 binary solutions of these osmotic agents. The osmotic solution was prepared with distilled water and the osmotic agents.

Osmotic dehydration (OD)

The OD was performed by immersing sweet potatoes' slices in 100 mL beakers containing the osmotic solution. The experiments were done at atmospheric pressure and temperature of 30±0.5ºC. The temperature was maintained constant in a chamber with temperature control (ELETROlab, EL 111/4 model). The solution:root ratio was 20:1 to avoid significant dilution during the process (Oliver et al. 2012).

At different times (15, 30, 45, 60, 90, 120, 150, 180, 240 and 300 minutes) samples were taken out of the osmotic solution, gently drained with tissue paper and characterized in terms of thickness and volume, then returned to the osmotic solution to continue the dehydration process. All the experiments were performed in five replicates and average values have been reported. To obtain the moisture content of the samples, a parallel experiment was conducted. At each set time, five samples were taken off from the solutions and the moisture was determined in an oven 70ºC for 24 h (AOAC, 2005).

Water loss (WL) and solid gain (SG) were calculated in accordance with Equations 1 and 2, respectively.

$$WL(\%) = \frac{(M_0 X_0) - (M_t X_t)}{M_0} x100 \quad (1)$$

$$SG(\%) = \frac{(M_t x_{st} - M_0 x_{s0})}{M_0} x100 \quad (2)$$

where: M_0 = weight of sample at time t= 0 (min); M_t = weight of sample at time t = t (min); X_0= initial moisture content, wet basis (w.b.); X_t = moisture content (w.b.) at time t = t (min); x_{st} = soluble solid content (w.b.) at time t= t(min) and x_{s0} = initial solid content wet basis (w.b.).

Analytical analysis

The shrinkage was determined by measuring the area and the thickness of the samples. Measurements of the area were obtained by analysis of images with the free software Image J® 1.45s (Seguí et al. 2010). The software provides the sample area by converting the pixels in the image in real dimensions, from a known scale. The thickness of each sample was measured in five locations with the aid of a digital caliper and the average data reported.

The volume of the samples was obtained by multiplying the surface area photographed by the average sample thickness, while the proportional volume was calculated as the ratio between the apparent volume of the dehydrated sample [m^3] and the fresh sample (initial) [m^3].

The dimensionless volume (β) was determined by the ratio between the apparent volumes after (V) and before (V_0) the OD, as shown in equation 3:

$$\beta = \frac{V}{V_0} \quad (3)$$

The water activity of the dehydration samples were obtained in the equipment Aqualab, Decagon Devices Inc., Pullman, WA, USA, 3-TE model.

Modeling of shrinkage

The models of the literature presented in Table 1 were used to fit the relationship between the shrinkage and the moisture content of the samples.

An exponential model of kinetics of shrinkage was proposed too. A non-linear regression analysis was conducted to fit the mathematical models by the Quasi-Newton method using the software Statistica 7.0® (Statsoft, Tulsa, OK).

Table 1 Mathematical models applied to predict shrinkage in sweet potatoes

Name	Equation, ($\beta=V/V_0$)	Reference
Rahman equation	$\beta=1-a(X-X_0)$	Yaldiz and Ertekin (2001)
Adapted Bala and Woods	$\beta=1-a(1-\exp(b(X-X_0)))$	Yaldiz, Ertekin and Uzun (2001)
Correa et al. model	$\beta= 1/ (a + b \exp(X))$	Ertekin and Yaldiz (2004)
Ratti equation	$\beta= a + b(X/X_0)$	Ratti (1994)
Adapted Lozano-2	$\beta= a + b(X/X_0) + c\exp(d(X/X_0))$	Lozano, Rotstein and Urbicain (1983)
Ratti equation 2	$\beta= a + bX + cX^2 + dX^3$	Ratti (1994)

where X is the moisture content and a, b, c and d are adjustment parameters.

The initial criteria for evaluating the adequacy of the model to the dehydration curve was the correlation coefficient value (R^2). In addition to this, reduced chi-square (χ^2) and root mean square error (RMSE) were used to determine the quality of the fit. These statistical parameters can be calculated as shown in equations 4 and 5.

$$\chi^2 = \sum_{i=1}^{N} \frac{\left(I_{\exp,i} - I_{pred,i}\right)^2}{N-n} \tag{4}$$

$$RMSE = \sqrt{\left[\frac{1}{N}\sum_{i=1}^{N}(I_{pre,i} - I_{\exp,i})^2\right]} \tag{5}$$

where: $I_{exp,i}$ stands for the experimental values and $I_{pre,\ i}$ denotes predicted values, which are calculated using the model for these measurements. N is the number of observations and n is the number of constants.

The experimental data were adjusted to an exponential model relating the volume ratio and the time of process.

3 Results and discussion

In order to investigate the shrinkage of sweet potatoes during OD with different osmotic agents, the change in volume ratio ($\beta = V/V_0$) were determined during the process. Figure 1 presents the relationship between normalized moisture content (X/X_0) and volumetric shrinkage (V/V_0).

Such relation was in a linear manner for samples osmodehydrated with sucrose and sorbitol, but not for the ones dehydrated with fructose. Mayor and Sereno (2004) observed that there was a linear relation between X/X_0 and V/V_0, when carrots and apples were osmodehydrated in sucrose solutions.

When plant tissue is placed in a hypertonic solution, water will leave the cell by osmosis. As a result the vacuole and the rest of the protoplasm will shrink, causing the plasma membrane to pull away from the cell wall. This phenomenon is accompanied with a loss in the turgor pressure, shrinkage and deformation of cells (cell wall and plasma membrane), and concentration of the protoplasmatic liquid phase (Mayor et al. 2008). Cellular shrinkage during OD has been observed in isolated apple cells immersed in hypertonic solution (Seguí et al. 2010). They noted that the protoplast detached from the cell wall. During membrane detachment some points which more strongly linked the plasma membrane to the cell wall were identified, the membrane-to-wall linkers try to resist the stretching and through the effect of the generated stretching forces, the cell wall becomes deformed. Microscopic changes in the cells affect directly structural changes on osmodehydrated products.

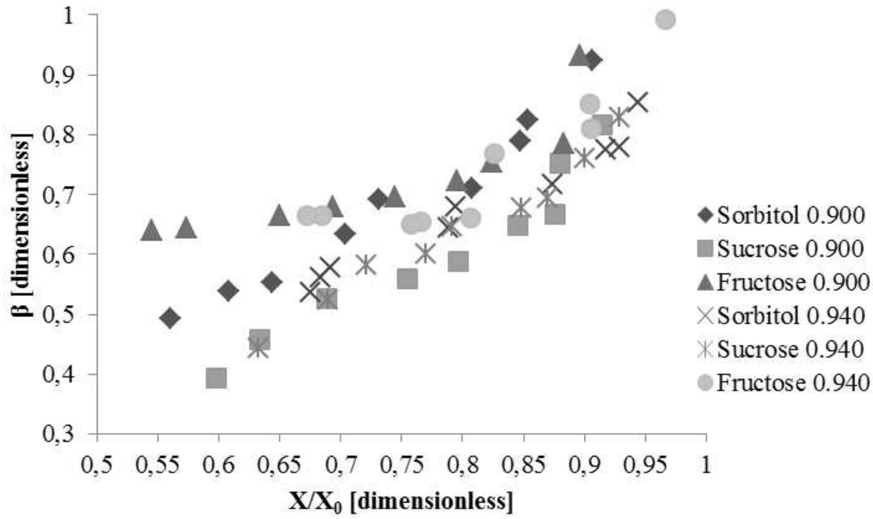

Figure 1 Variations of the volume ratio and normalized moisture content (X/X_0) during OD of sweet potato slabs.

Six models were employed in order to evaluate the volume reduction of osmodehydrated sweet-potatoes. The results of the non-linear regression analyses are shown in Table 2.

Table 2 The relationships between volume shrinkage and moisture content

Constants	Sorbitol (0.900)	Sucrose (0.900)	Fructose (0.900)	Sorbitol (0.940)	Sucrose (0.940)	Fructose (0.940)
Model 1: $\beta=1-a(X-X_0)$						
a	-0.2672	-0.3474	-0.2696	-0.3881	-0.4214	-0.3732
R^2	0.9820	0.9715	0.7736	0.9233	0.9220	0.8870
χ^2	5.00×10^{-4}	9.16×10^{-4}	3.79×10^{-3}	1.60×10^{-3}	1.97×10^{-3}	2.16×10^{-3}
RMSE	2.13×10^{-2}	2.87×10^{-2}	5.84×10^{-2}	3.80×10^{-2}	4.21×10^{-2}	4.41×10^{-2}

Model 2: β=1-a(1-exp(b(X-X_0)))

a	499.992	499.999	500.000	500.000	499.999	500.000
b	5.35×10^{-4}	6.95×10^{-4}	$5.4 \times 10^{-4}0$	7.70×10^{-4}	8.43×10^{-4}	7.47×10^{-4}
R^2	0.9822	0.9716	0.7736	0.9234	0.922	0.887
χ^2	5.69×10^{-4}	1.03×10^{-3}	4.27×10^{-3}	1.80×10^{-3}	2.21×10^{-3}	2.42×10^{-3}
RMSE	2.13×10^{-2}	2.86×10^{-2}	5.84×10^{-2}	3.80×10^{-2}	4.21×10^{-2}	4.40×10^{-2}

Model 3: β= 1/ (a + bexp(X))

a	1.7580	2.1014	1.6885	1.9787	2.1292	1.8080
b	-0.0449	-0.0627	-0.0394	-0.0946	-0.1097	-0.0815
R^2	0.7663	0.8618	0.8212	0.9398	0.9088	0.8311
χ^2	7.47×10^{-3}	5.00×10^{-3}	3.37×10^{-3}	1.42×10^{-3}	2.60×10^{-3}	3.63×10^{-3}
RMSE	7.73×10^{-2}	6.33×10^{-2}	5.19×10^{-2}	3.37×10^{-2}	4.56×10^{-2}	5.38×10^{-2}

Model 4: β= a + b(X/X_0)

a	0.2001	0.0409	0.2772	0.1608	0.0838	0.0821
b	0.8211	0.9189	0.6827	0.9690	1.0596	1.1504
R^2	0.9860	0.9837	0.7953	0.9571	0.9518	0.8907
χ^2	4.48×10^{-4}	5.90×10^{-4}	3.86×10^{-3}	1.01×10^{-3}	1.37×10^{-3}	2.35×10^{-3}
RMSE	1.89×10^{-2}	2.17×10^{-2}	5.55×10^{-2}	2.84×10^{-2}	3.31×10^{-2}	4.33×10^{-2}

Model 5: β= a + b(X/X_0) + cexp(dX/X_0)

a	0.1669	0.0999	-11.1140	0.2268	0.1844	-66.1004
b	0.9716	0.8085	-5.4188	0.6770	0.6787	-9.1889
c	-0.0127	0.0000	11.9473	0.0000	0.0000	66.5691
d	2.3278	10.7516	0.3866	18.0977	8.7318	-0.1368
R^2	0.9876	0.9946	0.8630	0.9812	0.9776	0.8994
χ^2	5.25×10^{-4}	2.60×10^{-4}	3.44×10^{-3}	5.91×10^{-4}	8.48×10^{-4}	2.88×10^{-3}
RMSE	1.77×10^{-2}	1.25×10^{-2}	4.54×10^{-2}	1.88×10^{-2}	2.25×10^{-2}	4.16×10^{-2}

Model 6: β= a + bX + cX^2 + dX^3

a	0.2809	-0.4187	3.4916	-1.1514	-0.6246	7.6743
b	0.1074	1.1804	-4.3087	2.9298	1.9473	-12.2466
c	0.1138	-0.5049	2.0477	-1.6503	-1.1058	6.8722
d	-0.0223	0.0919	-0.2958	0.3368	0.2429	-1.2204
R^2	0.9878	0.9968	0.8971	0.9837	0.9829	0.9655
χ^2	$5.17x10^{-4}$	$1.50x10^{-4}$	$2.59x10^{-3}$	$5.12x10^{-4}$	$6.50x10^{-4}$	$9.89x10^{-4}$
RMSE	$1.76x10^{-2}$	$9.49x10^{-3}$	$3.94x10^{-2}$	$1.75x10^{-2}$	$1.97x10^{-2}$	$2.43x10^{-2}$

The equations that best described empirical osmotic dehydration data due to high R^2 values and small values of χ^2 and RMSE is the sixth model (β= a + bX + cX^2 + dX^3). According to the tests applied, the model presented the followed mean values: $R^2 \geq 0.8971$, $\chi^2 \leq 0.00259$ and RMSE$\leq$0.0394.

The worst adjustment was observed when fructose was used as osmotic agent (as observed in Figure 1). It is possible to note that when the model 2, presented values of $R^2 = 0.7736$, to fructose (a_w=0.900) and the model 3, showed values of R^2=0.8311 to fructose (a_w=0.940). In a general way, the third model was the worst, with small values of R^2 and high values of χ^2 and RMSE.

For treatments with sucrose and sorbitol (a_w=0.900), all the models (excluding model 3) presented a correlation coefficient higher than 0.970, indicating the adequacy of the predicting models. Empirical models showed an acceptable fit to experimental data for all the materials tested, being the exponential model the one leading to larger deviation between experimental and predicted values.

Toĝrul and Ispir (2007), also proposed the application of these models in the prediction of apricots shrinkage in five different osmotic agents, and also concluded that most of the models presented showed good fit to the experimental data.

Figures 2 to 4 present the kinetics of volume ratio and of inverse of water loss (1/WL). It could be observed that the curves inverse of V/V_0 and 1/WL presented similar decreasing behavior.

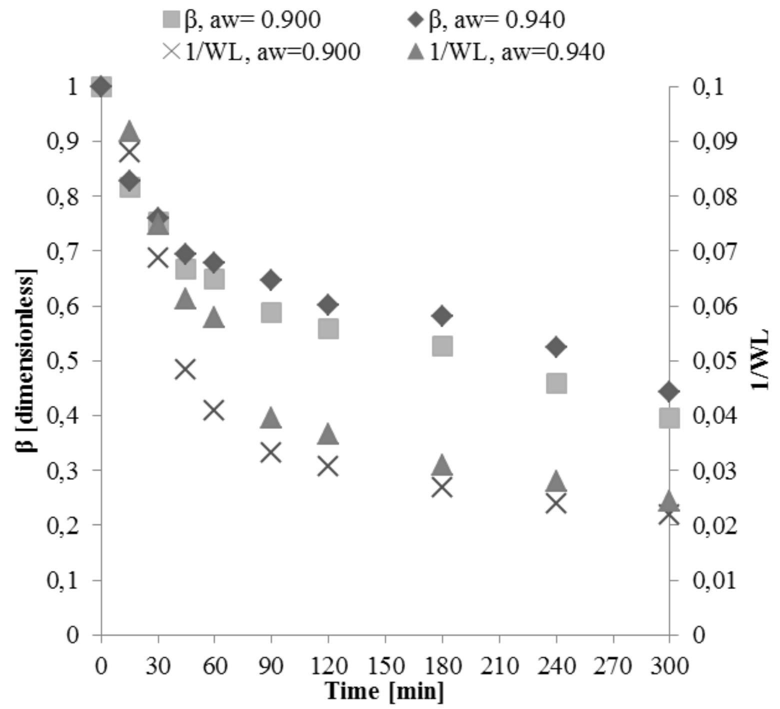

Figure 2 Variations of the volume ratio during OD of sweet potato slabs in sucrose solution

According to Mayor and Sereno (2004), at high moisture, when the material is in the rubbery state, shrinkage almost entirely compensates for moisture loss, and volume of the material decreases linearly with moisture content. At low moisture contents, the glass transition temperature (T_g) increases, allowing the material to pass from rubbery to glassy state, and the rate and extension of shrinkage decreases significantly. This behavior may explain deviations from linearity observed by several authors in the relative change of sample volume vs. the relative change of moisture content.

According to the figures 2 to 4, each osmotic agent caused various effects on its volume. Fructose solutions presented a lower shrinkage than other agents, regardless of the water activity (Figure 4). It is possible to note that higher volume ratio was observed when sucrose was employed (Table 3). This observation suggested that the volume change (shrinkage) is predominantly due to the volume of water removed, once sucrose presented a higher water loss than sorbitol and fructose in a_w=0.940. In solution with a_w=0.900, sucrose and sorbitol presented similar water loss. A linear shrinkage is a behavior considered suitable when V/V_0 is related to X/X_0. That is, the volume reduction was proportional to the amount of water removed.

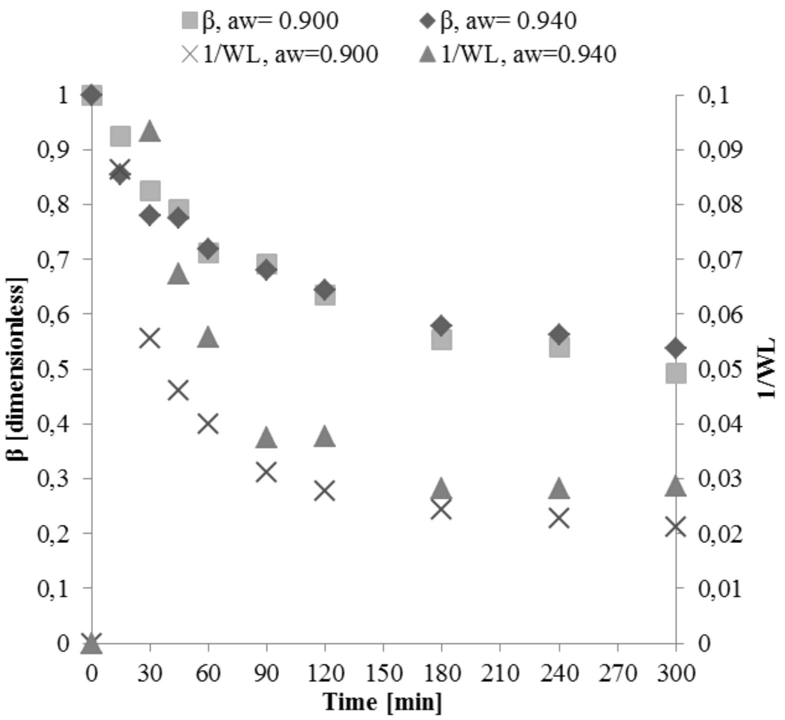

Figure 3 Variations of the volume ratio during OD of sweet potato slabs in sorbitol solution.

According to Béttega et al. (2014) the porosity of the material remains constant over the dehydration course.

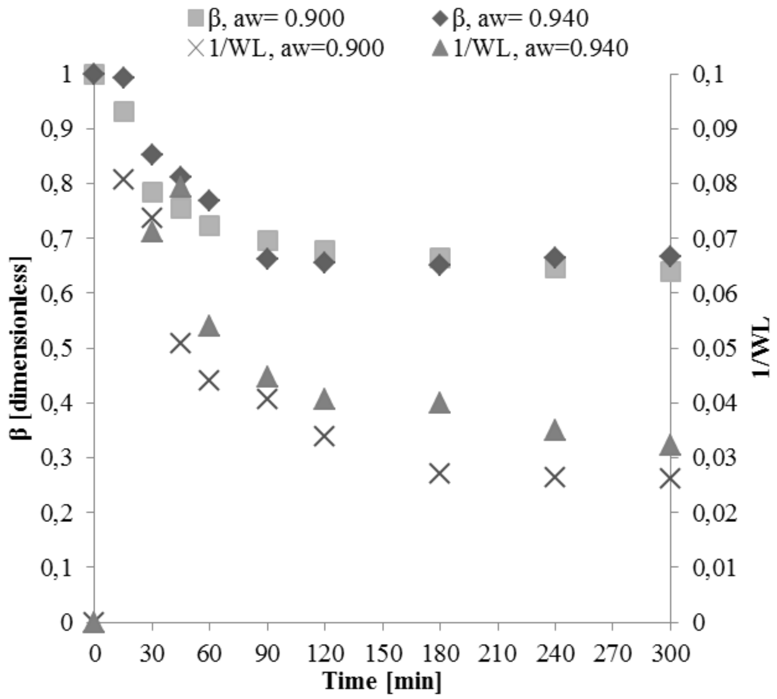

Figure 4 Variations of the volume ratio during OD of sweet potato slabs in fructose solution.

It is also possible to observe that the samples treated with sucrose, in the course of processing time, water loss, and therefore volumetric shrinkage, continue increasing (Figure 2). When sorbitol and fructose were employed, there is a tendency to reach equilibrium, around 180 minutes of the process, resulting in lower water loss and constant shrinkage (Figures 3 and 4).

On table 3, are summarized some results obtained in the end of the osmotic process (300 minutes) for all treatments.

Table 3 Water activities (a_w) of the samples, volume ratio (β), water loss (WL) and the relation between WL and SG (solid gain) at final process (300 minutes). Average values ± stardard deviation

Treatment	a_w	β	WL	WL/SG
Sor (0.900)	0.919±0.003	0.492±0.044	47.138±0.524	3.609±0.236
Suc (0.900)	0.950±0.000	0.394±0.065	45.665±0.258	3.580±0.292
Fru (0.900)	0.893±0.006	0.639±0.093	38.082±1.335	2.411±0.269
Sor (0.940)	0.951±0.002	0.537±0.072	35.018±1.062	3.492±0.277
Suc (0.940)	0.962±0.001	0.443±0.099	41.173±0.536	4.232±1.097
Fru (0.940)	0.944±0.001	0.665±0.049	30.975±0.607	2.683±0.061

As shown on table 3, the solutions with lower water activity performed lower a_w in the samples for all osmotic agents, lower volume ratio and higher water loss. In general way, sorbitol presented intermedial values for all results, fructose was very effective on a_w reduction, but it is not so effective on water loss, reflecting the higher volume ratio (low shrinkage).

The aim of osmotic dehydration is to remove water from plant tissue in such a way that the penetration of osmoactive substance from solution to the material is severely reduced, so, higher relation between water loss and solid gain is advantageous. According to table 3, in solution with water activity lower, sucrose and sorbitol presented a higher and similar relation, demonstrating that they are more indicating than fructose. When the water activity is higher, sucrose presented a good relation between these two mass transfer parameters, and fructose presented low results for this. The net result is that increased concentrations seem to favor sugar uptake (sucrose and fructose cases), leading to decreased WL/SG ratios. It seems that in the case of higher sucrose concentrations (lower a_w), massive sugar uptake results in the development of a concentrated subsurface solids layer which upsets the osmotic gradient across the sample-solution interface and decreases the driving force for water flow (Kowalska et al. 2008).

The exponential model proposed for ratio volume (dimensionless) and time (min), the constants and statistical analyses results for all treatments are shown in Table 4

Table 4 Model constants of volume ratio and statistical analyses results

Model: $\beta = \exp(-kt^n)$	Sorbitol (0.900)	Sucrose (0.900)	Fructose (0.900)	Sorbitol (0.940)	Sucrose (0.940)	Fructose (0.940)
k	0.0267	0.0625	0.0651	0.0523	0.0640	0.0368
n	0.5826	0.4651	0.3556	0.4410	0.4304	0.4589
R^2	0.9815	0.9901	0.9109	0.9930	0.9860	0.8266
RMSE	0.0218	0.0161	0.0348	0.0114	0.0178	0.0546

The proposed empirical model presented good fit when sucrose and sorbitol were used as osmotic agent, with $R^2 \geq 0.9815$ and RMSE$\leq$ 0.0546, demonstrating that the exponential model would give a good representation for the phenomena when this agents were in the osmotic solution. The treatments where fructose were employed, presented higher values of RMSE and lower values of R^2, but still is possible to explain the relation between the ratio volume and the time. An exponential approach is extensively employed to demonstrate the relation between the dimensionless moisture and the time during the dehydration, and it is known by Page's model (1949). This model presented much better adjusts parameters than other equations. There are several works that reports the adequation of the exponential equation to drying data (Alibas 2007; Arslan & Özcan 2011).

4 Conclusion

Mathematical modeling is a powerful tool to solve significant problems arising in food dehydration. A similar behavior is observed on volume ratio and the inverse of water loss for all osmotic agents employed. An exponential model was obtained for predicting the relation between the shrinkage and the time of process and present good fit. Between the six proposed models for sweet potato shrinkage data versus moisture content, a polynomial approach presented good agreement with the experiment. The lower water activity, lower shrinkage was obtained for all osmotic agents. Among the agents used, sucrose showed a reduction in the volume of osmotically dehydrated samples.

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