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LUCIANE FANTE

ESTUDO DA CINÉTICA DE BRANQUEAMENTO E DE SECAGEM POR AR QUENTE E LIOFILIZAÇÃO DO ALHO (*Allium sativum L.*)

PORTO ALEGRE

2011

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Dissertação apresentada ao Curso de Pós Graduação em Ciência e Tecnologia de Alimentos como um dos requisitos para obtenção do grau de Mestre em Ciência e Tecnologia de Alimentos.

Orientador: Prof. Dr. Caciano Zapata Noreña

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Luciane Fante
(Engenheira de Alimentos/Universidade de Caxias do Sul)

DISSERTAÇÃO

ESTUDO DA CINÉTICA DE BRANQUEAMENTO E DE SECAGEM POR AR QUENTE E LIOFILIZAÇÃO DO ALHO (*Allium sativum L.*)

Submetida como parte dos requisitos para obtenção do grau de

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Aprovada em: ...../...../.....
Pela Comissão Examinadora:

Prof. Dr. Caciano Zapata Noreña
Orientador – PPGCTA/UFRGS

Prof. Dr. Adriano Brandelli
Banca – UFRGS

Profa. Dra. Vânia Regina Nicoletti Telis
Banca – UNESP

Profa. Dra. Ligia Damasceno Ferreira Marczak
Banca – UFRGS

Homologada em: ...../...../.....
Por:

José Maria Wiest
Coordenador do Programa de Pós Graduação em Ciência e Tecnologia de Alimentos (PPGCTA)

VITOR MANFRÓI
Diretor do Instituto de Ciência e Tecnologia de Alimentos. ICTA/UFRGS

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“Para ser grande, sê inteiro:
Nada teu exagera ou exclui.
Sê todo em cada coisa.
Põe quanto és no mínimo que fazes,
assim em cada lago a lua toda brilha, porque alta vive.”

Fernando Pessoa

RESUMO

O alho (*Allium sativum L.*) originário das zonas temperadas da Ásia Central é uma planta herbácea da família Alliaceae; possui como principais constituintes funcionais a alicina e a inulina. A alicina é um componente ativo e responsável pelo cheiro característico e atividade antimicrobiana, enquanto que a inulina é classificada como prebiótico e como fibra alimentar solúvel por ser resistente à digestão na parte superior do trato intestinal. Neste trabalho foram estudadas as propriedades físico-químicas do alho *in natura*, além do processo de branqueamento em água e vapor e a desidratação por ar quente e liofilização no alho. Os bulbos de alho foram limpos e selecionados considerando a ausência de injúrias visuais e infecções, bem como a uniformidade de tamanho e cor. O alho in natura apresentou umidade de 64,15±0,09 %, atividade de água de 0,986±0,001, sólidos solúveis de 36,00±0,85 °Brix e pH de 6,41±0,008. As concentrações de inulina, glicose e frutose foram de 56,62±0,89, 2,37±0,03 e 2,23±0,05 g/100g de matéria seca respectivamente. Os bulbilhos do alho foram descascados e cortados em rodelas com diâmetros de 15±2,40 mm e espessuras de 1±0,35 mm. A seguir as amostras passaram pelo processo de branqueamento em água previamente aquecido a 80 e 90 °C e em vapor a 100 °C à pressão atmosférica. Foram empregados tempos de 1, 2, 4, 6, 8 e 10 minutos em ambos os casos. Nesta etapa verificou-se o efeito do tempo e temperatura de branqueamento sobre a atividade das enzimas peroxidase, polifenoloxidase e inulinase e, mediram-se os parâmetros de cor *L*, a* e b**, avaliando-se a cinética de inativação dessas enzimas e das mudanças de cor. A análise das concentrações de inulina, glicose e frutose foram realizadas para as melhores condições de branqueamento utilizando Cromatografia Líquida de Alta Eficiência. A melhor condição de branqueamento foi obtida no vapor por 4 minutos, onde não se observaram mudanças na textura, com redução da atividade enzimática de 93,53 %, 92,15 % e 81,96 % para a peroxidase, polifenoloxidase e inulinase respectivamente. Nesta condição a concentração da inulina diminuiu 3,72 %, enquanto que a glicose aumentou 0,67 % e a frutose 0,55 %, quando comparadas ao alho in natura, devido à atividade residual da inulinase. Durante o branqueamento em água e vapor o parâmetro de cor *L** aumentou com o tempo, tornando as amostras mais claras, enquanto que *a** e *b** diminuíram, obtendo-se rodelas mais esverdeadas e azuladas. Na secagem, as rodelas de alho sem ou com branqueamento em vapor por 4 minutos, foram levadas a um secador de ar forçado às temperaturas de 50, 60 e 70 °C durante 6 horas. Para a liofilização as rodelas foram previamente congeladas a -80 °C por 24 horas e dispostas em bandejas dentro de um liofilizador empregando pressão de 64 µmHg, onde permaneceram por tempo médio de 48 horas. Ao estudar a cinética de secagem em ar forçado usando os modelos propostos por Henderson-Pabis, Page e Newton constatou-se o aumento da constante da taxa de secagem com o aumento da temperatura e com o uso do branqueamento. Os valores de umidade e de atividade de água no equilíbrio, obtidos a partir da porção assintótica das curvas de secagem em condições dinâmicas, estiveram na faixa de 0,086 a 0,102 g/g de matéria seca e de 0,376 a 0,521 a_w respectivamente, sendo estes menores com o aumento da temperatura e com emprego do branqueamento. Posteriormente as amostras desidratadas por ar quente e liofilização, foram moídas para medição dos parâmetros de cor, tamanho de partículas e determinação dos teores de inulina, glicose e frutose, temperatura de transição vítrea e observação da microestrutura por microscopia eletrônica de varredura. As amostras liofilizadas tiveram valores de *L** significativamente maiores, e parâmetros de *a** e *b** menores que as amostras desidratadas em ar forçado, sendo as amostras liofilizadas mais claras, esverdeadas e azuladas, próxima à cor do alho in natura. Também foi encontrado a

diminuição da concentração de inulina e aumento dos teores de glicose e frutose nas amostras desidratadas, indicando a possível hidrólise da inulina, podendo estar relacionada à atividade residual da enzima inulinase. Também foi observado nas amostras branqueadas menores concentração de inulina, glicose e frutose, que quando não branqueadas, devido à possível lixiviação na água. Nas amostras liofilizadas os diâmetros médios das partículas foram de 79,35 µm e 104,79 µm no alho sem e com branqueamento respectivamente, sendo menores que às desidratadas em ar quente que variaram de 119,28 µm à 302,04 µm, observando-se, por microscopia eletrônica, menores danos na superfície das amostras liofilizadas, pois este processo origina menor contração e conseqüentemente menor rugosidade nas amostras quando comparado com a secagem em ar. As temperaturas de transição vítrea do alho desidratado em pó variaram de 99,39±1,98 °C à 120,15±3,80 °C, sendo maior quanto menor o valor de atividade de água, confirmando o efeito plasticizante da água.

Palavras-chave: alho, inativação enzimática, desidratação, cor, inulina, inulinase.

SUMMARY

Garlic (*Allium sativum L.*) originated in the temperate zones of Central Asia and is a herbaceous plant of the Alliaceae family; its main functional components are allicin and inulin. Allicin is the active component responsible for the characteristic odor and anti-microbial activity, whereas inulin is classified as a prebiotic substance and as a soluble dietary fiber since it is resistant to digestion in the small intestine. The physicochemical properties of garlic *in natura* were studied in the present work, and also the processes of water and steam blanching and dehydration in hot air and by freeze-drying. The garlic cloves were cleaned and selected considering the absence of visual injuries and infections and also uniformity of size and color. The *in natura* garlic presented a moisture content of 64.15±0.09 %, water activity of 0.986±0.001, soluble solids of 36.00±0.85 °Brix and pH of 6.41±0.008. The inulin, glucose and fructose concentrations were 56.62±0.89, 2.37±0.03 and 2.23±0.05 g/100g of dry matter, respectively. The garlic cloves were peeled and cut into slices with diameters of 15±2.40 mm and thicknesses of 1±0.35 mm. The samples were submitted to blanching in water baths previously heated to 80 and 90 ºC and, steam at a temperature of 100 ºC at atmospheric pressure. Times of 1, 2, 4, 6, 8 and 10 minutes were employed for both water and steam blanching. At this stage the effects of blanching time and temperature on the activities of the enzymes peroxidase, polyphenoloxidase and inulinase were determined, and the color parameters of L^*, a^* and b^* were measured, evaluating the kinetics of inactivation of these enzymes and the color changes. The concentrations of inulin, glucose and fructose were determined by high performance liquid chromatography in the samples treated with the best of the blanching conditions. The best blanching conditions were obtained using steam for 4 minutes. Under these conditions no changes in texture were observed, and the enzymatic activities were reduced by 93.53 %, 92.15 % and 81.96 % for peroxidase, polyphenoloxidase and inulinase, respectively. Under these conditions the inulin concentration decreased by 3.72 % and the glucose and fructose concentrations increased by 0.67 % and 0.55 %, respectively, when compared to the *in natura* garlic, due to the residual inulinase activity. The color parameter L^* increased with time during both steam and water blanching, whereas a^* and b^* decreased, obtaining slices which were greener and bluer. For drying, the garlic slices, with or without steam blanching for 4 minutes, were placed in forced air dryers at 50, 60 and 70 ºC for 6 hours. For freeze drying, the slices were previously frozen at -80 ºC for 24 hours and then placed in trays in the freeze drier at a pressure of 64 µmHg, where they remained for a mean time of 48 hours. The models proposed by Henderson-Pabis, Page & Newton were used to study the drying kinetics, and it was shown that the drying rate constant increased with increase in temperature and with the use of blanching. The equilibrium values for moisture content and water activity (a_w), obtained from the asymptotic part of the drying curves under dynamic conditions, were in the range from 0.086 to 0.102 g/g of dry matter and from 0.376 to 0.521 respectively, these values decreasing with increase in temperature and with the use of blanching. After drying the samples using both hot air and freeze-drying, they were ground for subsequent determination of the color parameters, particle size, inulin, glucose and fructose contents and glass transition temperatures, and for observation of the microstructure by scanning electronic microscopy. The freeze-dried samples showed significantly higher values for L^* and lower values for a^* and b^* than the samples dried by forced air, and thus the freeze-dried samples were lighter in color and more greenish or bluish, closer to the color of the *in natura* garlic. All the dehydrated samples showed a decrease in the inulin contents and increase in the glucose and fructose contents, indicating a possible hydrolysis of the

inulin which could be related to the residual activity of the enzyme inulinase. There were also lower inulin, glucose and fructose concentrations in the blanched samples as compared to the non-blanched samples, possibly due to leaching into the water. In the freeze-dried samples the mean particle diameters were 79.35 µm and 104.79 µm for the non-blanched and blanched samples, respectively, smaller than the values obtained in the forced air-dried samples, whose diameters varied from 119.28 µm to 302.04 µm. By way of scanning electronic microscopy, it was observed that the surface of the freeze-dried samples was less damaged than that of the forced air-dried samples, since freeze-drying results in less contraction and consequently less wrinkling than air drying. The glass transition temperatures of the dehydrated garlic powders varied from 99.39±1.98 °C to 120.15±3.80 °C, increasing with decrease in the water activity, confirming the plasticizing effect of water.

Keywords: garlic, enzymatic inactivation, dehydration, color, inulin, inulinase.

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CAPÍTULO 1

INTRODUÇÃO

1. INTRODUÇÃO

1.1 Alho (*Allium sativum L.*)

O alho (*Allium sativum L.*), originário das zonas temperadas da Ásia Central, é uma planta herbácea da família Alliaceae com 40 a 70 cm de altura e com raízes de até 50 cm de profundidade. A planta possui folhas lanceoladas formando um pseudocaule o qual se implanta em um caule pequeno e achatado. Os caules desenvolvem-se formando bulbilhos (dentes), que em conjunto formam o bulbo de característica arredondada e constituído por 5 a 20 bulbilhos envoltos por folhas protetoras (brácteas) de coloração branca ou arroxeada (Figura 1) (Oliveira *et al*., 2003).

Após a colheita os bulbos são secos ao sol por três a quatro dias para evitar o seu umedecimento. O armazenamento pode ser feito em câmaras frias a 0ºC, com umidade relativa de 70 a 75%. Umidades relativas inferiores a 70% causam excessiva perda de água e umidades maiores que 75% favorecem o apodrecimento dos bulbos, mesmo que não ocorra condensação de água na superfície deste produto (Neves, 2007).

Figura 1 – Alho (*Allium Sativum L.*)

Fonte: Galante *et al.* (2008).

As regiões sul e sudeste do Brasil são as mais propícias para o cultivo do alho, onde a faixa de temperatura média varia entre 13 e 24°C, sendo a mais indicada para o bom desenvolvimento das plantas; é necessário que a temperatura de inverno seja inferior a 15°C para a formação do bulbo. Temperaturas entre 20 e 30°C podem prejudicar o seu desenvolvimento e, acima de 30°C o alho produzido tem o seu valor comercial reduzido. O alho é cultivado na época fria do ano, considerada a mais seca na maioria das regiões produtoras (Marouelli *et al*., 2002).

O alho apresenta grande importância sócio-econômica ocupando no Brasil o quarto lugar entre as hortaliças, superado apenas pelas culturas de batata, tomate e cebola (Vieira, 2004). De fato, no ano de 2009 o Brasil produziu cerca de oitenta mil toneladas de alho, plantando aproximadamente nove mil hectares, destes, 2700 no estado do Rio Grande do Sul com produção de cerca de vinte mil toneladas, conforme o Instituto Brasileiro de Geografia e Estatística (2010).

O Brasil é um dos países que mais consome alho no mundo, cerca de 1.100 g por pessoa/ano, sendo a maior parte comercializada na forma in natura, embora o consumo de pastas e outros produtos processados venha crescendo gradativamente (Oliveira *et al*., 2003 e Oliveira *et al*., 2004). O seu consumo é elevado por possuir características acentuadas de aroma e sabor que lhe atribuem propriedades condimentares, além de ser reconhecido pelas suas numerosas propriedades medicinais (Block *et al*., 1993).

1.2 Composição Química

Os constituintes químicos mais importantes do alho são a alicina, inulina, ácido fosfórico e sulfúrico, proteínas e sais minerais. Os teores médios, em 100 g de matéria fresca do alho, em minerais são: 535 mg de potássio, 149 mg de fósforo, 14 mg de cálcio, 21 mg de magnésio, 5 mg de sódio e 0,8 mg de ferro e, para as vitaminas tiamina e piridoxina são de 0,18 e 0,44 mg respectivamente, enquanto que apresenta somente traços de riboflavina (Abib, 2004). A Tabela 1 mostra a composição química dos bulbos de alho por 100 g da matéria seca.

Tabela 1 - Composição Química dos Bulbos de Alho.

Composição por 100 g de matéria seca	
Energia (Kcal)	113
Água	67,5
Carboidratos	23,9
Fibra	4,3
Proteínas	7,0
Lipídeos	0,2
Cinza	1,3

Fonte: Tabela Brasileira de Composição de Alimentos – UNICAMP (2006)

O alho é classificado como alimento energético e possui a alicina como princípio ativo, a qual é considerada um tiosulfinato inativo em bulbos inteiros. Com o esmagamento do alho, a alicina é rapidamente formada pela interação do aminoácido aliina com a enzima aliinase. A alicina é responsável pelo odor característico, pelas atividades antimicrobianas, antiinflamatórias, antitrombóticas, anticâncer, antiaterosclerótica, antibacteriana e efeitos antioxidantes do alho (Lawson, 1998). A alicina é encontrada no alho integral, com teores de 0,3 a 0,4 %, podendo ser estável por um longo período de tempo, no entanto, reage rapidamente com algumas proteínas (Mayeux *et al*., 1988).

Entre os carboidratos, possui a inulina, a qual é um carboidrato de reserva em mais de 30.000 espécies de vegetais, tais como, trigo, cevada, cebola, alho, alho porró, aspargos e banana (Robinson, 1995) (Tabela 2).

Tabela 2 - Conteúdo de inulina em alimentos.[1]

Fonte	Nome Científico	Parte Comestível	Inulina (%)
Alcachofra	*Cynara scolymus*	Folhas centrais	3-10
Alho	*Allium sativum*	Bulbo	9-16
Alho porró	*Allium ampeloprasum*	Bulbo	3-10
Almeirão	*Cichorium intybus*	Raízes	15-20
Aspargo	*Aspargus officinalis*	Talo	1-30
Banana	*Musa acuminata*	Fruto	0,3-0,7
Bardana maior	*Arctium lappa*	Raízes	3,5-4
Cebola	*Allium cepa*	Bulbo	2-6
Centeio	*Secale sativa*	Grãos	0,5-1
Cevada	*Hordeum vulgare*	Grãos	0,5-1,5
Dente de leão	*Taraxacum officinale*	Folhas	12-15
Trigo	*Triticum aestivum*	Grãos	1-4
Tupinambo	*Helianthus tuberosus*	Tubérculos	16-20
Yacon	*Polymnia sonchifolia*	Raízes	3-19

[1] = Valores na amostra em base úmida.
Fonte: Gibson *et al*. (1994).

A inulina pertence ao grupo de polissacarídeos chamados frutanos, composta por uma cadeia principal de unidades de frutose, unidas por ligações $\beta(2\rightarrow1)$, e por uma molécula de glicose na porção inicial da cadeia linear de frutose unida por uma ligação do tipo ($\alpha1 - \beta2$). A inulina apresenta grau de polimerização (GP) que varia de 2 a 60, e a sua hidrólise

enzimática pela inulinase produz frutanos denominados oligofrutoses em que o GP varia de 2 a 7 (Roberfroid, 1993).

Ao sofrer hidrólise, a inulina produz oligômeros lineares estruturalmente designados GF_n (onde G representa a molécula de glicose, F a molécula de frutose e n o número de unidades de frutose) e F_m, constituída apenas por frutose (onde m representa o número de unidades de frutose), sendo que o primeiro tem características não redutoras e o segundo redutoras, contudo os dois mantém suas propriedades prebióticas (Roberfroid, 1993). A estrutura química da inulina pode ser observada na Figura 2.

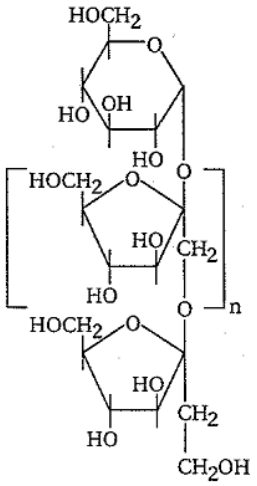

Figura 2 – Estrutura química da inulina.

Fonte: Roberfroid (1993).

A inulina é classificada como prebiótico e como fibra alimentar solúvel, por ser resistente à digestão na parte superior do trato intestinal, alcançando o intestino grosso praticamente intacta, onde é fermentada pelas bactérias (Roberfroid, 1993).

Van Loo (1999) indica que o consumo de frutanos do tipo inulina aumenta a absorção de cálcio, e possivelmente de magnésio e ferro, podendo afetar o metabolismo lipídico humano no sentido de diminuir o colesterol total. A administração de dietas com prebióticos tais como oligofrutose e inulina junto com culturas liofilizadas de *B. longum* inibem a formação de lesões pré-neoplásicas no cólon (Reddy, 1999).

A inulina foi identificada como um ingrediente capaz de substituir a gordura e a oligofrutose o açúcar, usados com sucesso em vários produtos alimentares como bolos,

chocolates, embutidos e produtos lácteos, devido as suas características funcionais e dietéticas (Niness, 1999; Narinder *et al.*, 2002 e Hauly *et al*., 2002).

A oligofrutose é mais solúvel que a sacarose e fornece entre 30 – 50% da doçura desta. Contribui para encorpar produtos lácteos e melhorar a umectância de produtos de panificação, diminuir o ponto de congelamento de sobremesas congeladas e agir como aglutinante em barras nutricionais de granola. Desse modo, ela exerce o mesmo papel que a sacarose, mas tem a vantagem de apresentar menor valor calórico, sendo que a sacarose apresenta 4 Kcal/g enquanto que a oligofrutose tem 1,5 Kcal/g, além de atuar como fibra alimentar. Diferentemente de outras fibras, não tem sabor adicional, permitindo a formulação de alimentos com alto teor de fibras mantendo a aparência e o gosto das formulações padrões (Hauly *et al*., 2002 e Franck, 2002).

Taper e Roberfroid (1999) relatam o uso da inulina na produção de alimentos funcionais para prevenção de câncer de mama. Coxan (2005) apresentou um estudo para a prevenção de osteoporose utilizando dieta rica em frutanos do tipo inulina. O uso de dietas ricas em inulina e oligofrutose reduz a incidência de câncer (Reddy, 1999).

1.3 Inativação Enzimática

O branqueamento é um tratamento térmico essencial antes do processamento de qualquer vegetal por inativar enzimas e destruir microrganismos, além de ajudar na qualidade, principalmente durante a secagem, congelamento, fritura ou armazenamento. Esse tratamento térmico deve ser rápido, de preferência em meio úmido (vapor ou água quente) o qual proporciona aquecimento uniforme e altas taxas de transferência de calor (Cruz *et al*., 2006).

O uso de água quente para a realização do branqueamento é o mais popular processo adotado comercialmente, por ser simples e econômico, porém um branqueamento prolongado resulta em perda considerável de nutrientes, como carboidratos, proteínas, minerais, vitaminas e açúcares solúveis em água, causadas principalmente por difusão ou lixiviação (Lee, 1958). Esses nutrientes são principalmente solúveis em água, resultando em maior perda no branqueamento em água quente do que no vapor. As enzimas peroxidase, polifenoloxidase e inulinase podem ser inativadas com o uso desses tratamentos, através do controle de tempo e temperatura para a melhor inativação (Schweiggert *et al*., 2005).

Quando o alho é descascado, os bulbilhos são expostos ao ambiente e sofrem alterações indesejáveis de qualidade, como o escurecimento rápido (Mayer, 2006). A polifenoloxidase é responsável pelo aparecimento de pigmentação escura provenientes da polimerização oxidativa de quinonas, que podem ser evitadas com métodos tais como uso de tratamento térmico e mudanças de pH (Schweiggert *et al*., 2005).

A enzima peroxidase é considerada indicadora da inativação pelo branqueamento por ser a mais resistente ao calor (Reed, 1975). Uma vez que a inativação completa da peroxidase requer longo tempo, resultando em grande perda de nutrientes durante o branqueamento. Singh e Chen (1980) sugeriram que 90% de inativação da peroxidase seria suficiente para evitar qualquer deterioração no produto.

A enzima inulinase deve sofrer inativação quando se deseja manter a integridade da inulina, a qual não é hidrolisada pelas enzimas digestivas na primeira porção do intestino e como conseqüência disto não aumenta a glicemia e nem os níveis de insulina no sangue, sendo ideal para diabéticos (Leonel *et al*., 2006). A perda de atividade da inulinase é obtida em temperaturas superiores a 70°C (Sharma *et al*., 2006) onde a atividade catalítica é destruída, mantendo desta forma a integridade da inulina (Böhm *et al*., 2005).

Para o estudo cinético de inativação térmica dessas enzimas, geralmente são usados os modelos de ordem zero e primeira ordem, além do modelo bifásico de primeira ordem, o qual consiste na separação de dois grupos diferentes, quanto a sua estabilidade térmica, sendo uma componente lábil e a outra resistente ao calor (Tabela 3).

Tabela 3 – Modelos matemáticos aplicados à cinética de inativação enzimática.

Modelo	Equação	Referência
Ordem zero	$y = y_0 + k_0 t$, (1)	Stamp e Labuza (1983)
Primeira ordem	$y = y_0 \exp k_1 t$, (2)	Stamp e Labuza (1983)
Bifásico	$y = a_L \exp^{-k_L t} + b_R \exp^{-k_R t}$, (3)	Ling e Lund (1978)

y: atividade residual da enzima, y_0 atividade residual enzimática no tempo zero, k_0, k_1, k_L *e* k_R*:* constantes de velocidade da reação, a_L e b_R: parâmetros do modelo e *t:* tempo

1.4 Secagem

A secagem é considerada uma operação importante, podendo ser realizada por ar, vácuo, pulverização e congelamento (Banga e Singh, 1994). A remoção da água é a principal tarefa para a preservação dos alimentos, reduzindo os teores de umidade a um nível que permita o armazenamento seguro por um período de tempo prolongado (Lenart, 1996).

A desidratação por ar é um processo antigo usado na conservação dos alimentos, onde o produto é exposto a uma corrente contínua de ar quente para a remoçao da umidade (Ratti, 2001). As amostras são distribuídas em bandejas cobrindo toda a base, para que a superfície do produto fique exposta ao ar de secagem. O monitoramento da perda de peso da amostra deve ser feito em intervalos de tempo pré-determinados, com a utilização de balança acoplada ao gabinete de secagem (Geankoplis, 1986).

A secagem por liofilização é considerada uma das melhores técnicas para a remoção de água, obtendo-se produtos finais com alta qualidade em comparação a outros métodos de secagem, sendo baseada na desidratação por sublimação de um produto congelado (Genin e René, 1995). Conforme Ratti (2001) ausência de água líquida e as baixas temperaturas do processo de liofilização tornam a maioria das reações microbiológicas e de deterioração mais lentas, obtendo-se produtos de alta qualidade. O estado sólido da água durante a liofilização protege a estrutura primária e a forma dos produtos, com redução mínima de volume. Apesar das vantagens, o processo é considerado caro para a fabricação de produtos desidratados.

Os processos de desidratação melhoram a estabilidade dos alimentos, uma vez que reduzem consideravelmente atividade de água, minimizando as alterações físicas, químicas e microbiológicas durante o armazenamento (Ochoa *et al*., 2002 e Hatamipour *et al*., 2007).

A estabilidade e a atividade de água dos alimentos estão relacionadas com as reações químicas de escurecimento não enzimático, oxidação, atividade enzimática, crescimento microbiano, entre outras. As maiores taxas de reação ocorrem em alimentos com umidade alta e intermediária o que é indesejável, e as velocidades de reações menores se encontram na faixa de atividade de água de 0,2 a 0,4 (Fennema, 1996). A atividade de água máxima tolerada nos produtos desidratados, sem induzir a perda de propriedades desejáveis, oscila entre 0,35 e 0,5 dependendo do produto. No entanto, na faixa de atividade de água entre 0,2 e 0,35 não ocorre mais à proliferação de microrganismos (Fennema, 1993). A Figura 3 mostra que o principal fator na estabilidade de um alimento é a disponibilidade de água para o crescimento microbiano e para as reações químicas.

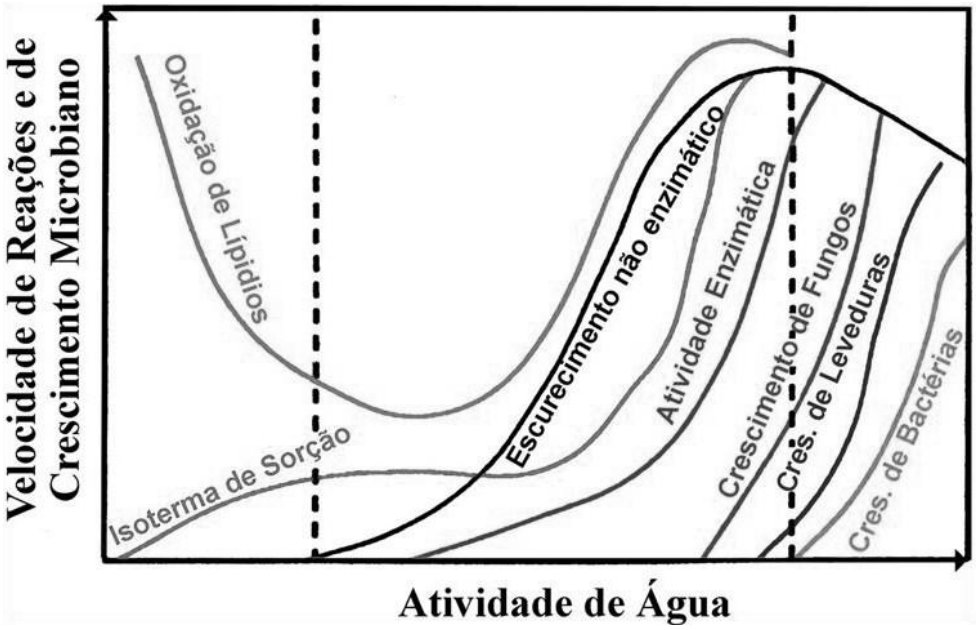

Figura 3 - Velocidade relativa de reações em função da atividade de água.

Fonte: Labuza (1968).

A desidratação dos alimentos torna mais fácil o manuseio do produto devido à diminuição do volume e a perda de peso que os produtos são submetidos durante o processo (Ochoa *et al*., 2002).

As características específicas de cada produto, associadas às propriedades do ar de secagem e ao meio de transferência de calor adotado, determinam diversas condições de secagem, conforme apresentado na Figura 4 (Brod *et al*., 1999), onde a curva (a) representa a diminuição do teor de umidade do produto, em base seca (X), com o tempo de secagem (t); a curva (b) representa a taxa de secagem do produto (dX/dt) em função do tempo (t) e a curva (c) representa a variação da temperatura do produto durante a secagem.

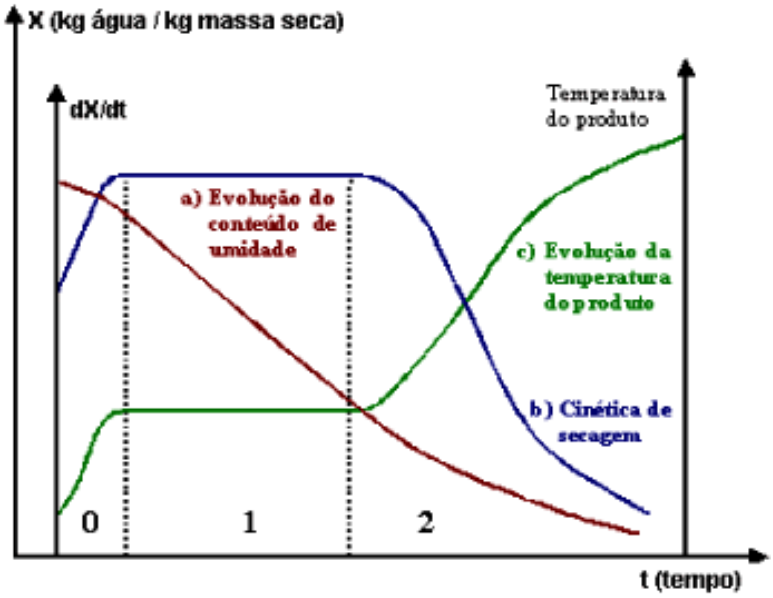

Figura 4 – Curvas típicas de secagem.

Fonte: Brod *et al*. 1999.

Alguns aspectos na desidratação de alimentos devem ser considerados, tais como os fatores físicos (encolhimento, escurecimento) e matemáticos do processo (Van Arsdel, 1973). Diversos modelos matemáticos têm sido utilizados para descrever a cinética de secagem em camada delgada de produtos agrícolas e alimentares (Tabela 4), sendo utilizados para estimar o tempo de secagem de diferentes produtos e na geração das curvas de secagem (Midilli *et al*., 2002).

Tabela 4 – Modelos matemáticos aplicados às curvas de secagem.

Modelo	Equação
Henderson-Pabis	$W = k_1 \exp(-k_2.t)$, (4)
Page	$W = \exp(-k_3.t^{k4})$, (5)
Newton	$W = \exp(-k_5.t)$, (6)

W: umidade adimensional; k_1, k_2, k_3, k_4 e k_5: parâmetros do modelo; *t:* tempo; $W = \frac{X_{wt} - X_{we}}{X_{wo} - X_{we}}$; X_{wo}, X_{we} e X_{wt}: valores da umidade inicial, no equilíbrio e no tempo *t*.

Fonte: Kiranoudis *et al*. (1992)

1.5 Cor

A cor é considerada um atributo importante, pois normalmente é uma das primeiras propriedades observadas pelo consumidor (Saenz, 1993). Muitas reações podem afetar a cor durante o processamento térmico, entre as mais comuns estão a degradação de pigmentos, especialmente carotenóides e clorofila e, reações de escurecimento enzimático e não enzimático (Barreiro *et al*., 1997). Outros fatores que afetam a cor incluem o pH, acidez, tempo e temperatura do processo (Rejano *et al*., 1997). A fim de minimizar a deterioração da cor devem-se utilizar equipamentos de processamento adequados, além de pré-tratamentos como o branqueamento (Weemaes *et al*., 1999).

Uma das técnicas usadas para observar as mudanças de cor nos alimentos é a sua medição através de aparelhos específicos que iluminam a amostra e medem a energia luminosa refletida ou transmitida pela superfície (Little, 1976). Os aparelhos mais utilizados pela sua sensibilidade são os espectrofotômetros e os colorímetros *tristímulos*, os quais são úteis para descrever a deterioração visual da cor além de fornecer informações para o controle de qualidade dos produtos (Sapers e Douglas, 1987).

O sistema tridimensional CIE$L^*a^*b^*$ de cores fornece três coordenadas retangulares (L^*, a^* e b^*) que permitem ao observador determinar com exatidão a cor da amostra. Neste sistema, o eixo x corresponde às cores que variam do verde ($-a^* = -60$) ao vermelho ($+a^* = +60$), o eixo y corresponde às cores que variam do azul ($-b^* = -60$) ao amarelo ($+b^* = +60$) e o eixo z corresponde à luminosidade expressa pela variável (L^*) e assume valor zero para o preto absoluto e 100 para o branco total (Wendt, 2006). A Figura 5 apresenta as coordenadas do sistema CIE$L^*a^*b^*$.

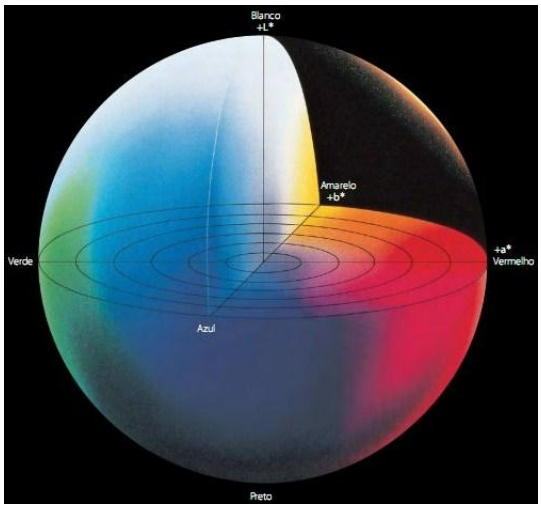

Figura 5 – Coordenadas do sistema CIE$L^*a^*b^*$.

Conforme Palou *et al*. (1999) os parâmetros *L**, *a** e *b** são usados para descrever outros parâmetros de cor, tais como, *Chroma*, ângulo *Hue*, diferença total de cor (ΔE) e índice de escurecimento (*IE*) (Tabela 5). Os parâmetros *Chroma* (saturação de cor) e *Hue* (ângulo de tonalidade) utilizam coordenadas cilíndricas, sendo que o valor de *Chroma* é zero no centro e aumenta conforme se distancia deste, o ângulo *Hue* inicia-se no eixo +*a** e é dado em graus sexagesimal onde, 0° = +*a** (vermelho), 90° = +*b** (amarelo), 180° = -*a** (verde) e 270° = -b* (azul). A diferença total de cor é usada para indicar a mudança de cor nas amostras tratadas em comparação à amostra original. O índice de escurecimento representa a pureza da cor marrom e é considerado um parâmetro importante nos processos que envolvem escurecimento enzimático (Castañón *et al*., 1998).

Tabela 5 – Parâmetros de cor a partir de *L**, *a** e *b**.

Parâmetro	Equação
Chroma	$(a^{*2} + b^{*2})^{1/2}$, (7)
Hue	$\tan^{-1}\left(\frac{b^*}{a^*}\right)$, (8)
ΔE	$\sqrt{(L_o^* - L^*)^2 + (a_o^* - a^*)^2 + (b_o^* - b^*)^2}$, (9)
IE	$\frac{[100(x - 0,31)]}{0,17}$, (10), onde $x = \frac{(a^* + 1,75L^*]}{5,645L^* + a^* - 3,012b^*}$

L_0*, a_0* e b_0* são as leituras de cor na amostra original.
Fonte: Palou *et al*. (1999)

1.6 Microestrutura

A microestrutura de alimentos é definida pela organização dos seus componentes e suas interações. Durante o processamento a microestrutura dos alimentos pode ser destruída e posteriormente reconstituída, resultando na sua reestruturação e reorganização (Aguilera e Stanley, 1990).

O conhecimento da microestrutura de alimentos é um pré-requisito necessário para entender suas propriedades, podendo-se desta forma descrever, prever e controlar o seu comportamento e a organização de seus componentes. Os métodos para o processamento de

alimentos podem ser baseados no conceito de que mudanças na microestrutura afetam as propriedades do produto. Desse modo, técnicas de análise de microestrutura são necessárias para entender as relações estrutura-propriedade (Aguilera, 2005).

A microscopia eletrônica de varredura (MEV) é usada para examinar superfícies onde as amostras podem se apresentar secas (MEV convencional) ou congeladas abaixo de –80°C (cryo-MEV). Uma camada de metal, responsável pela condutividade elétrica, é pulverizada sobre a amostra para que possa ser visualizada. As imagens geradas pela técnica de MEV possuem bom foco e intensidade e são relativamente fáceis de serem entendidas (Danilatos, 1993).

O MEV destina-se basicamente ao exame de superfície das amostras, sendo que as superfícies internas das amostras também podem ser visualizadas desde que sejam previamente fraturadas e expostas. Uma ampla faixa de aumentos pode ser usada (20x-100.000x) e a MEV pode alcançar uma profundidade de campo aproximadamente 500 vezes maior que a microscopia ótica. Este tipo de microscópio é constituído de lentes, circuito de varredura, coletor e ampliador de sinais, tubo de raios catódicos, sistema de vácuo e registro de imagens (James, 2009).

1.7 Transição Vítrea

Em alimentos constituídos de materiais amorfos, a mudança de fase mais comum é a transição vítrea, que consiste em uma transição de fase de segunda ordem de um estado sólido-vítreo para um estado semi-líquido gomoso (Roos, 1995).

A transição vítrea ocorre a certa temperatura denominada temperatura de transição vítrea (T_g), que depende da composição do alimento, principalmente do teor de água. Um dos métodos empregados para a determinação da temperatura de transição vítrea de alimentos é a técnica de calorimetria diferencial de varredura (DSC). Essa metodologia detecta uma mudança típica no calor específico da amostra à temperatura de transição vítrea (Sperling, 1992).

A T_g está diretamente ligada à alteração de textura dos alimentos, que é uma das propriedades mais relevantes do ponto de vista sensorial e microbiológico. Quando materiais amorfos se encontram sob baixas temperaturas (estado vítreo) eles são duros e quebradiços, com características vítreas, similares às observadas em plásticos duros ou biscoitos crocantes.

O aumento da temperatura pode causar uma transformação para o estado gomoso (menos viscoso) à temperatura de transição vítrea (Labuza, 2004 e Sperling, 1992).

A crocância característica de alimentos no estado vítreo é altamente desejada em biscoitos, batatas fritas, cereais matinais e alimentos desidratados, já a maciez associada ao estado gomoso é desejável em alguns produtos desidratados, como damasco e banana, e, também, em produtos industrializados, como é o caso de alguns biscoitos e recheios (Baroni, 2004 e Labuza, 2004). A determinação das propriedades de estado em alimentos, em função da temperatura e da concentração de água, fornece informações valiosas no estabelecimento da formulação, processamento, embalagem e estocagem para que seja evitada a mudança de fase (vítreo-gomoso), mantendo o alimento na forma desejada para o consumo pelo maior tempo possível (Roos *et al*., 1991).

Na Figura 6, pode-se observar um diagrama representativo da transição entre os estados vítreo e gomoso.

Figura 6 - Diagrama representativo da transição entre os estados vítreo e gomoso.

Fonte: Labuza et al. (2004).

1.8 Objetivo Geral

O presente trabalho visou estudar as características físico-químicas do alho (*Allium sativum L.*) sem e com branqueamento e, desidratado por ar quente e liofilização.

1.9 Objetivos Específicos

- Estudar a cinética de inativação das enzimas peroxidase, polifenoloxidase e inulinase e da cor do alho durante o branqueamento em água a 80 e 90 °C e vapor a 100 °C

- determinar as concentrações de inulina, glicose e frutose nas melhores condições de branqueamento em água e vapor

- estudar a cinética de secagem em ar quente nas temperaturas de 50, 60 e 70 °C no alho sem e com branqueamento

- estudar as mudanças de cor L^*, a^* e b^*, concentrações de inulina, glicose e frutose, tamanho de partículas, microestrutura e temperatura de transição vítrea no alho em pó desidratado por ar quente e liofilização, sem e com branqueamento.

CAPÍTULO 2

Study of the enzymatic inactivation and changes in color of garlic (*Allium sativum L.*) treated under different blanching conditions

Artigo a ser submetido para publicação na revista International Journal of Food Science & Technology e formatado de acordo com as normas desta revista.

Study of the enzymatic inactivation and changes in color of garlic (Allium sativum L.) treated under different blanching conditions

Luciane Fante & Caciano Pelayo Zapata Noreña*

Institute of Food Science and Technology, Federal University of Rio Grande do Sul, Porto Alegre, RS, Brazil.

Summary

The inactivation kinetics of the enzymes peroxidase (POD), polyphenoloxidase (PPO) and inulinase and changes in the color parameters *L**, *a** and *b** of garlic, were studied during steam blanching at 100ºC and water blanching at 80 and 90ºC. The garlic cloves were peeled, cut into slices with a diameter of 15±2.40 mm and thickness of 1±0.35 mm, and distributed uniformly in metal baskets. One batch was placed in an autoclave generating steam at a temperature of 100ºC; and the others in water baths at 80 and 90ºC, for 1, 2, 4, 6, 8 and 10 minutes. The best blanching conditions were in steam for 4 minutes, where no changes in texture were observed, and the enzymatic activities were reduced by 93.53%, 92.15% and 81.96% for peroxidase, polyphenoloxidase and inulinase, respectively. Under these conditions the inulin concentration reduced by 3.72%. The color parameter *L** increased with increase in blanching time, the samples becoming lighter in color, and the parameters *a** and *b** decreased, resulting in slices that were greener and bluer. Using the principal components analysis it was shown that the enzymes POD, PPO and inulinase were strongly correlated with the color parameter *b**, the hue and the browning index (R^2>0.93).

Keywords: garlic, enzymatic inactivation, inulinase, color.

* C*orrespondent: Fax: +55-51-3308*7048*; e-mail: czapatan@ufrgs.br*

Introduction

Garlic (*Allium sativum* L.) is classified as an energetic food, the major chemical constituents being allicin, carbohydrates, phosphoric and sulfuric acids, proteins and mineral salts (Rahman *et al.* 2006). Allicin is responsible for the characteristic odor and the antimicrobial, anti-inflammatory, anti-thrombosis, anti-cancer and anti-atherosclerosis activities and also for the antioxidant effect (Rabinkov *et al*., 1998). One of the carbohydrates present is inulin, which belongs to the group of polysaccharides called fructans. Its is composed of a main chain of fructose units connected by β(2→1) bonds and a glucose molecule attached to the initial part of the linear fructose chain by a type (α1 – β2) bond (Roberfroid, 1993).

Inulin is found as a reserve carbohydrate in various vegetables, fruits and cereals, including leeks, onions, garlic, wheat, chicory, artichokes, bananas (Van Loo, 1995) and yacon (Scher *et al*. 2009). It is classified as a prebiotic substance and used in a wide variety of food products since it presents excellent technological attributes, for example as a fat substitute and as dietary fiber (Roberfroid, 1993).

According to Schorr-Galindo & Guiraud (1997), the inulin molecule can undergo partial or complete hydrolysis using the enzyme inulinase, present in the natural form in both plants and microorganisms. When partially hydrolyzed, inulin produces an extensive group of different compounds with an F_n structure such as: inulobiose (F_2), inulotriose (F_3) and inulotetraose (F_4), and GF_n compounds such as: sucrose (GF), kestose (GF_2), nistose (GF_3) and fructofuranosyl nystose (GF_4), which maintain their prebiotic properties (Ronkarta *et al.* 2007). Complete hydrolysis results in the formation of glucose and fructose (Roberfroid, 1999).

These oligosaccharides are not hydrolyzed by the digestive enzymes of the small intestine, and, as a consequence, do not increase glycemia or the blood insulin levels, being ideal for diabetics (Leonel *et al.* 2006). The loss of activity of inulinase is obtained at temperatures above 70ºC (Sharma *et al.* 2006) where the catalytic activity is destroyed, thus maintaining the integrity of the inulin (Böhm *et al*., 2005).

Heat inactivation of the enzymes is fundamental due to the importance of preserving the color of the raw material before any transformation (Ndiaye, 2009). In the case of garlic, after peeling, the cloves are exposed to the environment and suffer undesirable changes in quality, such as rapid browning (Mayer, 2006). Polyphenoloxidase is responsible for the appearance of brown substances due to oxidative polymerization of the quinones, which can be avoided using methods such as heat treatment and pH changes (Schweiggert *et al*., 2005). Since peroxidase is one of the most heat stable enzymes, it is considered as an indicator for inactivation by blanching (Yemenicioglu *et al*., 1999).

According to Lee (1958), hot water blanching is the process adopted commercially since it is simple and economical, although prolonged blanching leads to a series of undesirable alterations in the food including losses of color, flavor and texture and also considerable losses of nutrients such as carbohydrates, proteins, minerals, vitamins and water soluble sugars, mainly by diffusion or leaching. Most of these nutrients are soluble in water, resulting in a greater loss during hot water blanching than during steam blanching (Schweiggert *et al*., 2005).

The objective of the present work to study the inactivation kinetics of the enzymes peroxidase (POD), polyphenoloxidase (PPO) and inulinase, and the variation in color of garlic slices during water blanching at 80 and 90°C and steam blanching at 100°C, for different

times. The concentrations of inulin, glucose and fructose in the garlic with and without blanching was also investigated.

Material and methods

Material

The garlic (*Allium sativum L.*), cultivated in the city of Nova Pádua in the state of Rio Grande do Sul, Brazil, was acquired directly from the producer. The heads were cleaned and selected considering the absence of visual injury and infections and also uniformity of size and color. They were then stored at room temperature (22±2ºC) until used.

Experimental procedure

The garlic cloves were peeled and sliced using a food processor, presenting diameters and thicknesses of 15±2.40 mm and 1±0.35 mm, respectively. The samples were then submitted to a blanching process in which the slices were placed in metal baskets in water baths containing 2 liters of water previously heated to 80 or 90ºC. With respect to steam blanching, the slices were uniformly distributed in metal baskets and placed in an autoclave generating steam at 100ºC and atmospheric pressure. Times of 1, 2, 4, 6, 8 and 10 minutes were used in both cases. After blanching, the samples were cooled in an ice bath for 3 minutes (Agüero *et al.*, 2008). The effects of blanching time and temperature on the activities of POD, PPO and inulinase were evaluated at this stage and the color parameters of L^*, a^* and b^* measured.

The concentrations of inulin, glucose and fructose were determined in the samples prepared under the best blanching conditions in water at 80 or 90ºC and in steam at 100ºC.

Preparation of the enzyme extracts

Following the methodology of Serradell *et al*. (2000) the samples were ground with 0.05 M phosphate buffer (pH 7.0) at a maximum of 4ºC in a proportion of 1:5 in the case of POD and PPO and 1:1 in the case of inulinase. The suspensions were then vacuum filtered through Whatman nº 01 filter paper to remove larger particles and the filtrates centrifuged at 1680 x g for 10 min at 4ºC. The supernatants were again vacuum filtered through Whatman nº 01 filter paper to obtain the enzyme extracts.

Peroxidase activity

POD activity was determined using the method described by Hultin *et al.* (1966), which consisted of mixing 3 mL enzyme extract with 5 mL 0.1 M phosphate buffer (pH 5.0), 0.5 mL 3% (v/v) hydrogen peroxide and 0.5 mL guaiacol. The mixture was incubated at 30ºC for 5 minutes and 1 mL 30% (w/v) sodium bisulfate then added to interrupt the enzymatic reaction and the reading carried out in a UV-visible spectrophotometer (Ultrospec 3100pro) at 470 nm.

One unit of enzyme activity was defined as an increase of 0.001 absorbance units per minute of reaction at 470 nm (Sun & Song, 2003).

Polyphenoloxidase activity

PPO activity was determined by the method described by Teisson (1979) using a UV-visible spectrophotometer (Ultrospec 3100pro). An aliquot of 0.5 mL of enzyme extract was mixed with 1.8 mL 0.05 M phosphate buffer (pH 7.0) and 0.05 mL 10 mM catechol and incubated in a water bath at 30ºC for 30 minutes. An aliquot of 0.8 mL 2 N perchloric acid was then added to interrupt the enzymatic reaction and the absorbance read at 395 nm.

One unit of enzyme activity was defined as an increase of 0.001 absorbance units per minute of reaction at 395 (Sun & Song, 2003).

Inulinase activity

Inulinase activity was determined using the methodology of Sharma *et al*. (2007) with some modifications. The trial consisted of using 0.4 mL enzyme extract, 0.45 mL 2% (w/v) inulin dissolved in 0.1 M citrate-phosphate buffer (pH 6.0) and 0.15 mL 0.1 M citrate-phosphate buffer (pH 6.0). The mixture was incubated at 30 ºC for 1h and the reaction then interrupted by placing in a boiling water bath (100ºC) for 10 minutes. One mL dinitrosalicylic acid was then added and the absorbance read in a UV-visible spectrophotometer (Ultrospec 3100pro) at 570 nm.

One unit of enzyme activity was defined as an increase of 0.001 absorbance units per minute of reaction at 570 nm (Sun & Song, 2003).

Color measurement

The garlic samples were ground and placed in Petri dishes (diameter=5 cm and height=1 cm) and filled to the top (Ancos *et al.* 1999). The color was measured by direct reading in a Minolta colorimeter (CR400/410) using the tridimensional CIE*L*a*b** system, where *L** indicates the luminosity (varying from 9=black to 100=white), *a** is a measurement varying from green (-60) to red (+60) and *b** varies from blue (-60) to yellow (+60). The instrument was calibrated using a white ceramic plate (*L**= 97.47; *a**= 0.08; *b**= 1.76). The parameters *L** *a** *b** were used to describe the *Chroma* (Eq. (1)), *Hue* (Eq. (2)), total color difference (Δ*E*) (Eq.(3)) and the browning index (*BI*) (Eq. (4)) during blanching (Palou *et al*., 1999).

$$Chroma = (a^{*2} + b^{*2})^{1/2}, \quad (1)$$

$$Hue = \tan^{-1}\left(\frac{b^*}{a^*}\right), \quad (2)$$

$$\Delta E = \sqrt{(L_o^* - L^*)^2 + (a_o^* - a^*)^2 + (b_o^* - b^*)^2}, \quad (3)$$

$$IE = \frac{[100(x - 0{,}31)]}{0{,}17}; \quad (4)$$

where,

$$x = \frac{(a^* + 1{,}75L^*)}{(5{,}645L^* + a^* - 3{,}012b^*)}$$

The subscript ‘o’ refers to the reading of the color of the fresh garlic, used as the reference. Δ*E* indicates the change in color of the samples when compared with *in natura* garlic.

Determination of sugars by HPLC

The samples were prepared according to the methodology cited by Scher *et al*. (2009) and adapted according to the method proposed by Toneli *et al.* (2007). Two grams of garlic were ground with 60 mL water at 90ºC. The product was heated in a water bath at 80ºC for 1 hour with constant stirring and the suspension then cooled to room temperature and centrifuged (Centrifuge 5415R) at 1680 x g for 15 minutes at 25°C. The supernatant was filtered through Whatman nº 1 filter paper and the through a 22 µm membrane filter and stored at -18ºC. For the analysis, the samples were pre-heated in a water bath at 80ºC and then placed in an ultrasonic bath for 4 minutes before injection into the HPLC.

The inulin, glucose and fructose contents were determined using an adaptation of the method described by Zuleta & Sambucetti (2001) by direct determination using high performance liquid chromatography (HPLC-RI) with a Perkin Elmer series 200 chromatograph, refractive index detector, water (Milli-Q) as the mobile phase at 0.6mL/min, temperature of 80ºC, a Phenomenex Rezex RHM Monosaccharide column (300 x 7.8 mm) and a total run time of 13 minutes.

Kinetic analysis of enzyme inactivation and browning index

The first order biphasic model proposed by Ling & Lund (1978) was used to describe the kinetics of the heat inactivation of the enzymes POD, PPO and inulinase and the browning index (*BI*). This model consists of the separation into two different groups with respect to their heat stability, one component being heat labile and the other heat resistant:

$$y = a_L \mathrm{e}^{-k_L t} + b_\mathrm{R} \mathrm{e}^{-k_R t} \quad (5)$$

Where y represents the value of the residual enzyme activities or the browning index, k_L and k_R are the velocity constants of the heat labile and heat resistant components, respectively, a_L and b_R are parameters of the model and t is the blanching time.

The velocity constants were obtained from a regression analysis of the values for y of the heat resistant fraction as a function of time, which corresponds to the part of the curve where drastic changes in the residual enzyme activity or browning index did not occur, thus obtaining b_R and k_R. Subsequently the values for y of the heat labile fraction, which correspond to the initial blanching times, were corrected by subtracting the values for b_R. The velocity constant k_L was estimated from the regression analysis of the values corrected as a function of time.

Statistical analysis

ANOVA was used for the statistical analyses and the treatments compared using Tukey's means multiple comparison test. The SAS 6.2 program was used for the statistical tests. The kinetic model parameters (Eq. (5)) were estimated by a non-linear regression analysis using the Sigma Plot 8.0 program. The SAS 6.2 and STATISTICA 7.0 statistical packs were used for the principal components analysis (PCA), which consists of reducing the number of variables and establishing correlations between the data for the possible groups. All these analyses were carried out in triplicate.

Results and discussion

Inactivation of Peroxidase and Polyphenoloxidase

The peroxidase and polyphenoloxidase activities of the fresh garlic were 255.33±1.01 and 158.00±1.06 U g^{-1} fresh sample, respectively. During blanching both in water at 80 and 90ºC and in steam, the activities of peroxidase (Figure 1) and polyphenoloxidase (Figure 2) decreased rapidly in the first minute for both enzymes, and then decreased continuously but slowly up to 10 minutes of blanching.

The activity of the enzyme POD showed no significant difference (p>0.05) after 4 minutes blanching in steam or water at 90ºC and after 6 minutes in water at 80ºC. For the enzyme PPO there was no significant difference (p>0.05) as from 6 minutes blanching in steam and water at 90ºC and after 8 minutes in water at 80ºC. After 10 minutes of blanching, the activity of POD decreased to 11.20±1.06, 23.60±0.69 and 14.13±0.83 U g^{-1} fresh sample in steam, water at 80ºC and water at 90ºC, respectively, corresponding to activity losses of 95.61%, 90.76% and 94.47%. For PPO the values decreased to 9.60±1.20, 33.87±0.83 and 13.60±0.40 U g^{-1} fresh sample after 10 minutes of blanching in steam, water at 80ºC and water at 90ºC, respectively, corresponding to activity losses of 93.92%, 80.24% and 91.39%.

For both enzymes the residual activities were significantly different (p<0.05) for all the temperatures at the same times, with the exception of polyphenoloxidase after two minutes, which showed no significant difference (p<0.05) between blanching in water at 80 and at 90ºC. A decrease in residual activity with increase in temperature was also observed (Figures 1 and 2).

No studies related to the inactivation of these enzymes in garlic were found in the literature, however compared with other sources such as mint (Neves *et al*., 2009), yam and potato (Duangmal *et al*., 1999), grape (Rapeanu *et al*., 2006), peppers (Kavrayan *et al*., 2001), sweet potato (Lourenço *et al*., 1992), hearts of palm (Lourenço *et al*., 1990) and peach (Garro *et al*., 2010), the rates of inactivation of the PPO with increase in temperature were also higher. The same was observed for the enzyme POD in carrot slices, the loss in residual activity at high temperatures being related to the separation of the tertiary structure of the enzyme with formation of the secondary structure (Shivhare, 2009).

Leonardis *et al.* (2010) found that during the inactivation of the enzyme PPO in sunflower seeds by blanching in water at 80ºC for 15 minutes, the enzyme activity only started decreasing after 6 minutes, and after 15 minutes of treatment there was still 17% of residual activity. In the present study the residual PPO activity in the garlic blanched in water at 80ºC for 10 minutes was 19.76% (Figure 2).

In a study of enzyme inactivation carried out by Troiani *et al*. (2003) in three different grape cultivars submitted to heat treatment in water at 60 to 75ºC, they also failed to completely inactivate the enzymes POD and PPO. The authors reported 35 to 60% inactivation for POD and 25 to 50% for PPO. Ismail *et al.* (2006), studying the inactivation of POD in green peppers puree submitted to blanching in water at 90, 95 and 100ºC, found that inactivation was complete at 95ºC after 10 minutes and as from 8 minutes at 100ºC.

In order to obtain vegetables maintaining optimum quality during storage, a reduction of 90% of the POD activity after blanching is recommended (Bahçeci *et al*. 2005). In the present study, losses of 93.53% and 92.15% of the POD and PPO activities, respectively, were obtained after 4 minutes steam blanching.

Inulinase inactivation

The inulinase activity of the fresh garlic was 20.55±0.08 U g^{-1} fresh sample. A loss of activity with time (Figure 3) was observed during blanching in water at 80 and 90ºC and in steam at 100ºC, showing a rapid decrease in activity in the first minute. After this time the activity continued decreasing, but slowly, up to 10 minutes of blanching. This behavior was similar to that observed for the enzymes POD and PPO (Figures 1 and 2).

After 8 minutes of blanching at the three temperatures, the inulinase activity showed no significant difference (p>0.05). After 10 minutes of blanching, the inulinase activity decreased to 2.48±0.06, 4.77±0.06 and 3.32±0.06 U g^{-1} fresh sample in steam and water at 80 and 90ºC, respectively, corresponding to activity losses of 87.91%, 76.76% and 83.85%. The residual activity showed significant differences (p<0.05) for all the temperatures at the same times, decreasing with increase in temperature (Figure 3).

Inulinase is an enzyme of biotechnological interest and thus there are many studies on its production. Sharma *et al.* (2006) produced inulinase using *Streptomyces* sp. and garlic as a natural source of inulin, and studied the effect of heat treatment on the production of inulinase, observing activity losses of 64% at 60°C and 67% at 70°C, after 72 hours. For Sharma *et al.* (2007), the loss of inulinase activity was 80% after heat treatment at 70ºC for 72 hours, and 90% at 80ºC.

Haraguchi *et al*. (2006), studied the thermal stability of inulinase at different temperatures for 30 minutes, in a study on the purification of the enzyme, and found that the enzyme was stable at 60ºC and became inactive at 70ºC. Ishimaru *et al*. (2004), in a study on burdock roots, evaluated the inulinase activity at temperatures of 2, 8 and 20ºC for 20 days of storage, and found that the lowest enzyme activity was at 20ºC on the eighth day of storage.

Cruz *et al.* (1998) produced inulinase from *Aspergillus niger* with dahlia extracts and pure inulin as the carbon sources and found 8 % losses at 65ºC and complete inactivation at 75ºC for 30 minutes. In the present study, losses of inulinase activity of 72.16%, 78.51% and 81.96% were found after 4 minutes blanching in water at 80 and 90°C and steam, respectively.

From the results for the inactivation of the enzymes POD, PPO and inulinase, it was verified that the best conditions for blanching were in steam and water at 90ºC for 4 minutes and in water at 80ºC for 6 minutes. At these temperatures the use of longer blanching times resulted in modifications of the texture due to the thinness of the garlic slices, since during prolonged heating the cells can separate completely resulting in a great loss of textural strength (Kidmose *et al.*, 1999).

Effect of blanching on the sugar concentrations

Table 1 shows the concentrations of inulin, glucose and fructose in the fresh garlic and in the slices submitted to the best times of blanching in steam and in water at 80 and 90ºC. A possible hydrolysis of inulin can be seen in the treatment with steam for 4 minutes due to the decrease in the inulin concentration and significant increase ($p<0.05$) in the reducing sugar (glucose and fructose) concentrations, which could be due to the presence of the residual inulinase activity of 18.04% (Figure 3). For blanching in water, there was a significant decrease ($p<0.05$) in both the inulin and reducing sugar (glucose and fructose) contents as compared to the *in natura* sample and that blanched in steam. One of the possible causes of the significant loss of these sugars in the water-treated samples was leaching.

With respect to the reducing sugar contents of the garlic samples blanched in water at 80 and 90ºC, the higher the temperature the greater the leaching, as also observed by Pedreschi *et al*. (2009) on blanching potatoes in water at 60, 75 and 90°C for 120 minutes. The loss of inulin in water can also be caused by the use of high temperatures, causing greater dissolution. Hoehn *et al.* (1983), studied the extraction of inulin from Jerusalém artichokes using hot water, and reported the importance of using elevated temperatures between 80 and 90ºC to increase the dissolution of inulin, as also to obtain a purer inulin extract.

The significant decrease in concentration of the inulin in garlic blanched in water could also be caused by inulin depolymerization, due to a residual inulinase activity of 21.49% and 25.34% in water at 90°C for 4 minutes and in water at 80°C for 6 minutes, respectively. Hence considering the three blanching conditions examined, steam for 4 minutes was considered to be the best one due to the lowest inulin losses.

Enzyme inactivation kinetics

Table 2 shows the estimations for the kinetic parameters of the enzymes POD, PPO and inulinase. It can be seen that the reaction velocity constants increased with increase in temperature for both the heat resistant and heat labile components, as also observed by Zhu *et al.* (2010) studying the inactivation kinetics of PPO in apple pieces blanched at temperatures of 70 to 80ºC. The velocity constants of the heat resistant component were lower than those of the heat labile component for the three enzymes. These same observations were made by Morales-Blancas *et al.* (2002) on studying the inactivation kinetics of POD in broccolis, asparagus and carrot at temperatures of 70 to 95ºC using a first order biphasic model, who found values for k_R varying from 0.004 to 0.58 min^{-1} and for k_L from 1.50 to 19.18 min^{-1}. For

Ling & Lund (1978), working with the inactivation of pure POD in water, k_R varied from 0.034 to 0.84 min^{-1} and k_L from 1.09 to 4.61 min^{-1} at temperatures from 76.7 to 87.2°C.

The kinetic parameters (k_L and k_R) of the enzyme peroxidase were higher than those obtained for the enzymes polyphenoloxidase and inulinase, as also reported by Galdino *et al.* (2008) on studying the inactivation kinetics of POD and PPO of hearts of palm blanched in water at 65 to 80°C for 10 minutes, who found values for k_L and k_R varying from 0.971 to 1.052 min^{-1} and 0.147 to 0.172 min^{-1}, respectively, for POD, whilst for PPO the values varied from 0.146 to 0.172 min^{-1} for k_L and from 0.093 to 0.153 min^{-1} for k_R.

Color changes

For fresh garlic the rectangular coordinates L_0*, a_0* and b_0* were 62.55±0.81, -3.45±0.06 and 21.61±0.46, respectively. In a study of garlic paste, Shivhare & Ahmed (2001) found values of 69.37, -3.25 and 15.95 for *L**, *a** and *b**, respectively. Rejano *et al*. (1997), studying the color of garlic in a 5% NaCl brine solution, pasteurized at 90 ºC for 8 minutes, observed values for *L** of 80.8, for *a** of -1.9 and for *b** of 21.8.

Table 3 shows the different values obtained for this parameter during blanching in steam and in water at 80 and 90ºC for different times. It can be seen that the parameter *L** increased with blanching time for all the treatments, showing no significant difference as from 4 minutes for all the temperatures. The samples became significantly (p<0.05) lighter in color with the increase in temperature, and after 10 minutes of blanching presented values of 67.07±0.09, 64.24±0.17 and 65.91±0.11 for steam and for water at 80 and 90°C, respectively. According to Rocha & Morais (2001) working with apples, the lighter the color of the sample, the greater the value for *L**.

The parameters *a** and *b** decreased with blanching time for all the temperatures, making the samples greener (-*a**) and bluer (-*b**). There was no significant difference (p>0.05) in the values for *a** as from 6 minutes in steam at 100ºC, and for water at 80ºC there was no significant difference (p>0.05) between the times of 4 to 6 minutes, and for water at 90ºC between the times of 6 and 8 minutes. There was no significant difference (p>0.05) in the values for *b** as from 8 minutes in steam at 100ºC, as from 4 minutes in water at 80ºC or between the times of 4 and 6 minutes in water at 90ºC. The samples became significantly (p<0.05) bluer and greener with increase in temperature, as also observed by Shivhare & Ahmed (2001), who blanched garlic paste in a water bath at 70 to 90ºC.

The value of the cylindrical coordinate *Hue* was 99.09±0.19 in the *in natura* garlic and was found in the second quadrant. The value for *Hue* increased with blanching indicating displacement to an angle of 180º (-*a**, green). In steam at 100ºC and water at 80 ºC, the hue angle showed no significant difference (p>0.05) as from 8 minutes, and in water at 90ºC, there was no significant difference between 4 and 6 minutes. The samples became significantly (p<0.05) greener with increase in temperature, as also observed for the rectangular parameter *a**.

The value for *Chroma* of the *in natura* garlic was 21.89±0.46, and decreased with blanching time. For steam blanching at 100ºC the *Chroma* showed no significant difference (p>0.05) as from 8 minutes and in water at 80ºC as from 4 minutes. For water at 90ºC there was no significant difference (p>0.05) between 4 and 6 minutes. The samples became significantly (p<0.05) bluer with increase in blanching temperature. The same was observed in the evaluation of the rectangular parameter *b**.

The browning index (*BI*) of fresh garlic was 36.54±1.57, decreasing with blanching time, being significantly lower (p<0.05) at higher temperatures. Both for *BI* and ΔE there was

no significant difference (p>0.05) as from 8 minutes in steam and 4 minutes in water at 80ºC, whilst in water at 90ºC there was no significant difference (p>0.05) between 4 and 6 minutes. For the total color variation (ΔE) there was a significant increase (p<0.05) with increase in blanching temperature, indicating that the greatest difference in color from the standard sample (*in natura* garlic) was in steam at 100ºC as from 8 minutes. In a study with peppers puree carried out by Ismail *et al*. (2006), submitted to water blanching at 90 and 100ºC for 10 minutes, the total color variation also increased with increase in temperature.

Kinetics of the browning index

The browning index (*BI*) represents the purity of the brown color and is considered to be an important parameter in processes involving enzymatic browning (Castañon *et al*., 1998) and thus its velocity constants must be determined.

The data did not fit the classical zero and first order models well, obtaining correlation coefficients below 0.5 for the zero order model and below 0.7 for the first order model. Skrede (1985) mentioned that it is not always possible to apply the zero and first order models to describe the kinetics of color changes, since these changes are not always due to the Maillard reaction, but also involve the heat destruction of pigments present in the sample. However a good fit of the data was obtained when the first order biphasic model was employed, with correlation coefficients above 0.93 (Table 4).

Table 4 shows the increase in the velocity constants with increase in temperature, for both the resistant and labile components, the velocity constants of the resistant component being lower than those of the labile component. This same behavior was observed in the kinetic study of the enzymes POD, PPO and inulinase (Table 2). The orders of magnitude of

k_L and k_R were similar to those found in the enzymatic study in this work, and varied from 2.044 to 5.396 min^{-1} and from 0.031 to 0.098 min^{-1}, respectively.

Barreiro *et al*. (1997) also used the first order biphasic kinetic model to describe the total color change (*ΔE*) of a tomato paste concentrate heat treated in a capillary tube from 70 to 100ºC, obtaining a correlation coefficient above 0.94. Ibarz *et al*. (1999) used a combination of the zero and first order models to describe the total color change (*ΔE*) of a pear puree heat treated in water at temperatures from 80 to 98ºC and found correlation coefficients between 0.87 and 0.99. Gonçalves *et al*. (2007), studying kinetic models for the color changes of pumpkin during water blanching at temperatures from 75 to 95ºC, proposed the use of a fractionated conversion kinetic model, obtaining satisfactory correlation coefficients with a mean of 0.92.

Multivariate analysis

The principal component analysis (PCA) was used to calculate correlations between the color changes (*L**, *a**, *b**, *Chroma*, *Hue* and *BI*) and the enzyme activities (POD, PPO and inulinase) for the different blanching treatments employed.

The results obtained from the PCA showed that all the original variables (color and enzyme activity) could be reduced to two principal components that represented 98.73% of the total variability of the results. The first principal component (PC1) represented 91.33% of the total variability of the results and the second principal component (PC2) was responsible for 7.40% of the data variance. These results confirmed that the two principal components were highly significant in classifying the blanching treatments.

PC1 was strongly correlated with POD (R^2= 0.92), PPO (R^2= 0.97), inulinase (R^2= 0.96), *BI* (R^2= 0.99), *Chroma* (R^2= 0.99), *b** (R^2= 0.99), *Hue* (R^2= 0.94), *a** (R^2= 0.92) and *L** (R^2= 0.87), as can be seen in Figure 4 where, according to Kilimann *et al*. (2006), the measurements more distant from zero and smaller angles between the principal component and the variables, correspond to a strong association between them. PC2 showed no strong correlations with any of the variables, with correlation coefficients below 0.45.

Figure 4 also shows the existence of strong correlation between the parameters *L** and *Hue* (R^2>0.94) and between the color parameters *a**, *b**, *Chroma* and *BI* (R^2>0.91). The enzymes POD, PPO and inulinase were correlated with the color parameters *b**, *Chroma* and *BI* (R^2>0.93).

Figure 5 shows that all the blanching treatments differed strongly from the *in natura* garlic, located in the lower left hand side quadrant. In addition the samples submitted to the different blanching treatments could be divided into four different groups: the first showed strong correlation between steam blanching for 4, 6, 8 and 10 minutes and water blanching at 90ºC for 10 minutes, the second between steam blanching for 1 and 2 minutes and water at 90ºC for 4, 6 and 8 minutes, the third group between water blanching at 90ºC for 1 and 2 minutes and water at 80ºC for 8 and 10 minutes and the fourth group showed strong correlation for water blanching at 80ºC for 1, 2, 4 and 6 minutes.

Conclusions

The enzymes peroxidase, polyphenoloxidase and inulinase showed increases in enzyme inactivation and in the velocity constants with increase in temperature for both the labile and resistant components.

The best conditions for blanching at the three temperatures with respect to enzyme inactivation were obtained in steam and in water at 90ºC for 4 minutes, and in water at 80ºC for 6 minutes, where changes in texture were not observed and the enzyme activities were reduced as follows: peroxidase by 93.53%, 91.96% and 89.77%, polyphenoloxidase by 92.15%, 89.79% and 77.40% and inulinase by 81.96%, 78.51% and 74.66%, respectively. Correspondent inulin losses were 3.72%, 24.88% and 25.58% respectively.

The use of blanching caused significant modifications in the parameters L^*, a^* and b^*. The parameter L^* increased with blanching time and with temperature, the samples becoming lighter in color. The parameters a^* and b^* decreased with blanching time for all treatments, indicating greener ($-a^*$) and bluer ($-b^*$) slices. The browning index decreased with increase in time and in blanching temperature. The results for total color difference (ΔE) indicated that the greatest difference in color in relation to the *in natura* sample was for the sample steam blanched as from 8 minutes.

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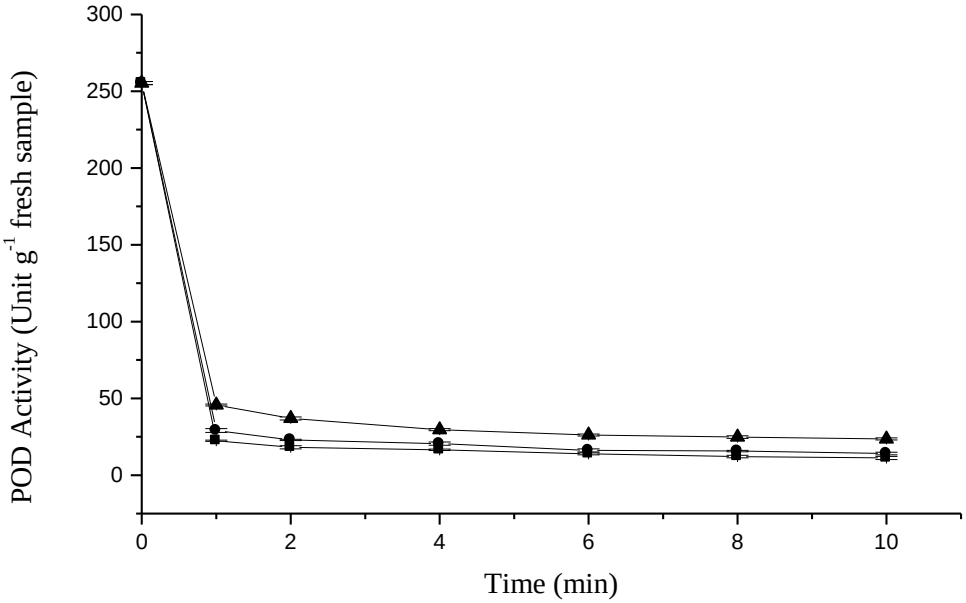

Figure 1 – Loss of activity for the enzyme peroxidase in garlic after different times and blanching conditions (Unit g^{-1} fresh sample). (■) Steam100°C; (●) Water 90°C and (▲) Water 80°C.

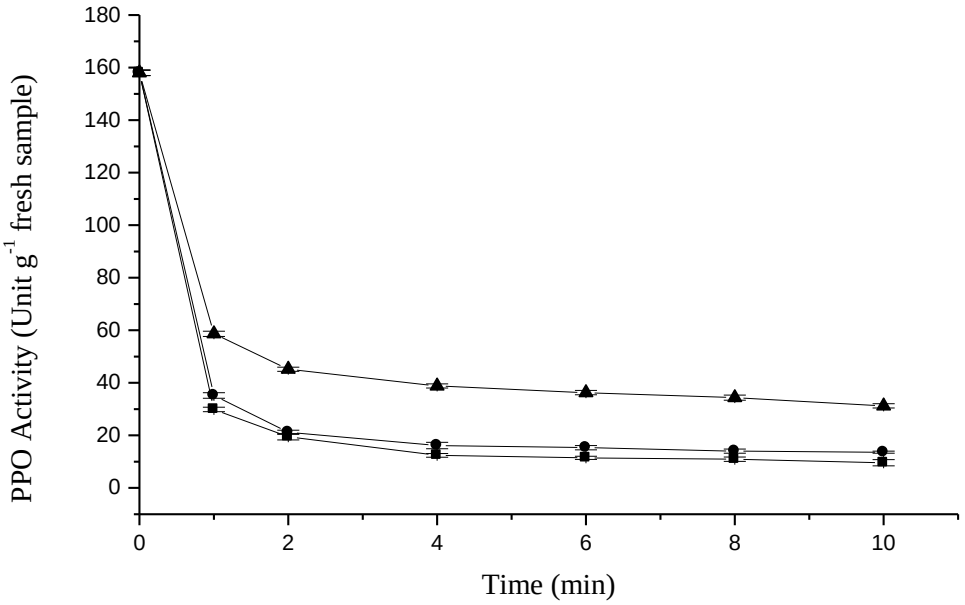

Figure 2 – Loss of activity for the enzyme polyphenoloxidase in garlic after different times and blanching conditions (Unit g^{-1} fresh sample). (■) Steam100°C; (●) Water 90°C and (▲) Water 80°C.

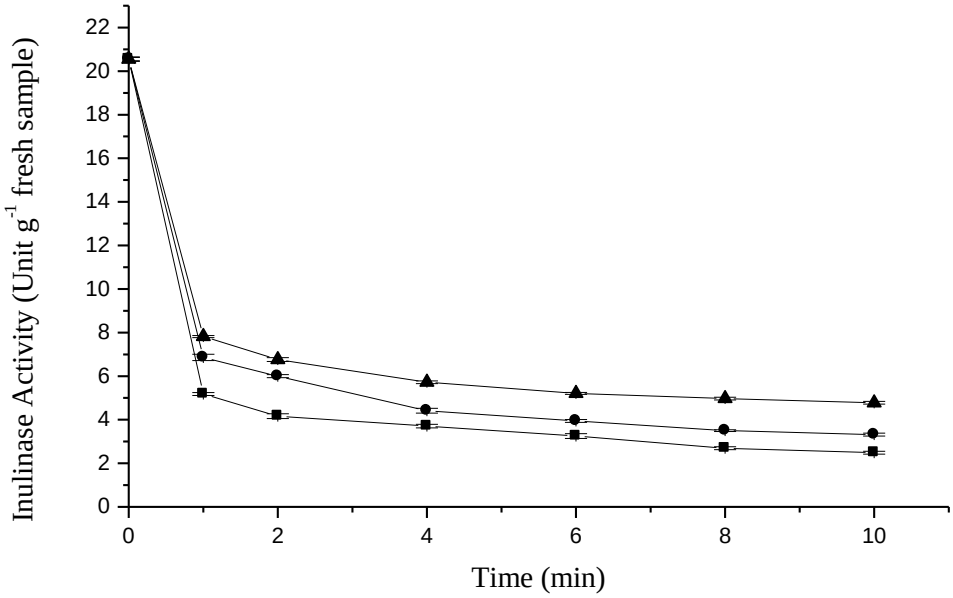

Figure 3 – Loss of activity for the enzyme inulinase in garlic after different times and blanching conditions (Unit g^{-1} fresh sample). (■) Steam100°C; (●) Water 90°C and (▲) Water 80°C.

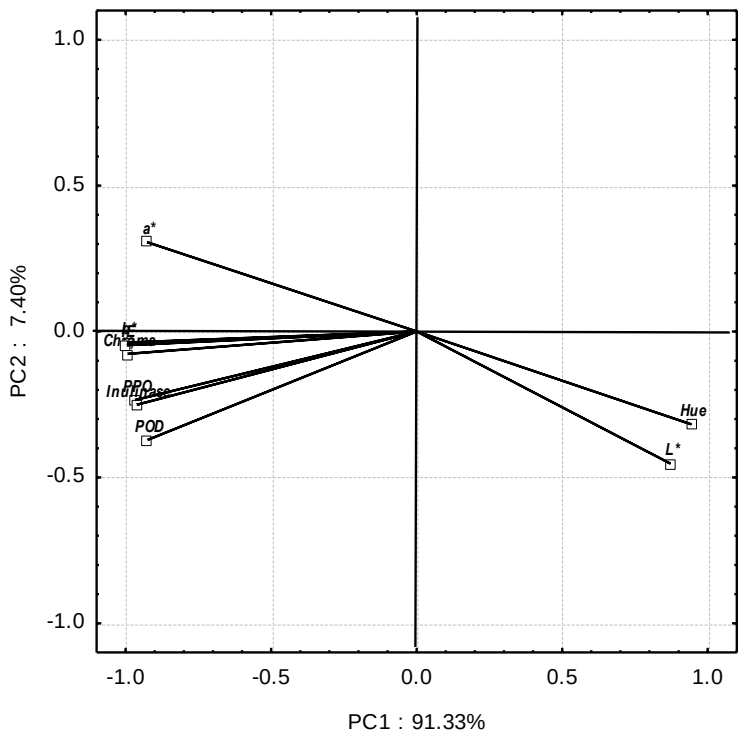

Figure 4 – Principal Components Analysis for the garlic submitted to different blanching treatments: color parameters (*L**, *a**, *b**, *Hue*, *Chroma* and *BI*) and the enzyme parameters (PPO, POD and inulinase).

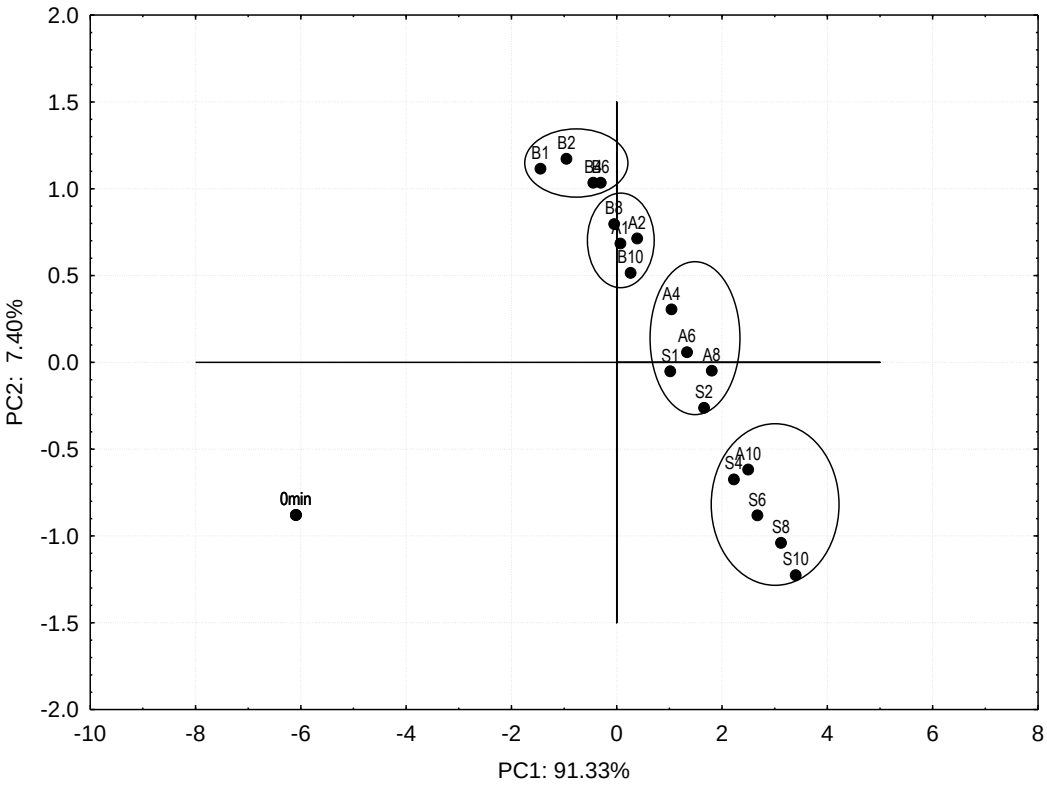

Figure 5 – Principal Components Analysis for the garlic submitted to different blanching treatments. (S1, S2, S4, S6, S8 and S10) = steam at 100°C for 1, 2, 4, 6, 8 and 10 minutes, (A1, A2, A4, A6, A8 and A10) = water at 90°C for 1, 2, 4, 6, 8 and 10 minutes and (B1, B2, B4, B6, B8 and B10) = water at 80°C for 1, 2, 4, 6, 8 and 10 minutes.

Table 1 – Contents of the sugars (dry weight basis) in the fresh garlic and in the samples submitted to different blanching treatments.

	Time (min)	Inulin (%)	Glucose (%)	Fructose (%)
Fresh garlic	0	56.62±0.89 [a]	2.37±0.03 [b]	2.23±0.05 [b]
Steam at 100ºC	4	52.90±1.16 [b]	3.04±0.06 [a]	2.78±0.07 [a]
Water at 90°C	4	31.74±0.78 [c]	1.38±0.01 [d]	1.30±0.01 [d]
Water at 80°C	6	31.04±0.74 [c]	1.61±0.08 [c]	1.54±0.06 [c]

Values expressed as the mean ± standard deviation.
The same letters in the same column indicate no significant difference (p>0.05).

Table 2 – Kinetic parameters for the inactivation of the enzymes peroxidase, polyphenoloxidase and inulinase under different blanching conditions.

	Temperature (°C)	k_L (min^{-1})	R^2	k_R (min^{-1})	R^2
Peroxidase					
Water	80	2.898±0.202	0.99	0.038±0.007	0.94
Water	90	4.116±0.632	0.99	0.061±0.006	0.98
Steam	100	5.396±0.994	0.99	0.068±0.008	0.98
Polyphenoloxidase					
Water	80	2.044±0.032	0.99	0.031±0.002	0.99
Water	90	2.123±0.033	0.99	0.035±0.006	0.97
Steam	100	2.197±0.194	0.99	0.040±0.005	0.96
Inulinase					
Water	80	2.241±0.161	0.99	0.030±0.004	0.95
Water	90	2.244±0.298	0.99	0.050±0.005	0.98
Steam	100	4.110±0.286	0.99	0.067±0.007	0.98

Values expressed as the mean ± standard deviation.

Table 3 – Color parameters for garlic pre-treated by blanching.

Blanching time (min)	*L**	*a**	*b**	*Hue*	*Chroma*	*IE*	Δ*E*
Fresh garlic	62.55±0.81	-3.45±0.06	21.61±0.46	99.09±0.19	21.89±0.46	36.54±1.57	
Steam Blanching (100°C)							
1	64.91±0.13^{bA}	-4.54±0.08^{aC}	11.52±0.20^{aC}	111.46±0.52^{eA}	12.38±0.19^{aC}	13.61±0.38^{aC}	10.67±0.20^{eA}
2	65.38±0.09^{bA}	-4.64±0.02^{bC}	10.64±0.10^{bC}	113.65±0.16^{dA}	11.61±0.10^{bC}	11.83±0.16^{bC}	11.64±0.09^{dA}
4	66.59±0.12^{aA}	-4.69±0.02^{bC}	10.03±0.25^{cC}	115.31±0.63^{cA}	11.07±0.22^{cC}	10.50±0.43^{cC}	12.60±0.20^{cA}
6	66.60±0.20^{aA}	-4.83±0.03^{cC}	9.12±0.45^{dC}	117.64±1.26^{bA}	10.33±0.39^{dC}	8.82±0.78^{dC}	13.47±0.45^{bA}
8	66.78±0.15^{aA}	-4.84±0.03^{cC}	8.16±0.17^{eC}	120.55±0.54^{aA}	9.49±0.15^{eC}	7.21±0.26^{eC}	14.43±0.13^{aA}
10	67.07±0.09^{aA}	-4.91±0.07^{cC}	7.91±0.27^{eC}	121.93±0.67^{aA}	9.31±0.26^{eC}	6.69±0.40^{eC}	14.76±0.27^{aA}
Water Blanching (90°C)							
1	63.10±0.07^{bB}	-4.35±0.08^{aB}	12.40±0.34^{aB}	109.35±0.66^{dB}	13.14±0.31^{aB}	15.93±0.67^{aB}	9.47±0.33^{eB}
2	63.27±0.08^{bB}	-4.39±0.06^{aB}	11.97±0.22^{aB}	110.10±0.38^{dB}	12.75±0.22^{abB}	15.03±0.43^{aB}	9.92±0.23^{dB}
4	64.54±0.15^{aB}	-4.44±0.07^{aB}	11.34±0.21^{bB}	111.27±0.57^{cB}	12.18±0.19^{cB}	13.49±0.39^{bB}	10.75±0.18^{cB}
6	64.85±0.18^{aB}	-4.61±0.18^{bB}	11.36±0.10^{bB}	112.12±0.10^{cB}	12.26±0.12^{bcB}	13.25±0.12^{bB}	10.82±0.09^{cB}
8	65.14±0.21^{aB}	-4.61±0.18^{bB}	10.11±0.07^{cB}	114.50±0.95^{bB}	11.12±0.05^{dB}	11.01±0.30^{cB}	12.09±0.09^{bB}
10	65.91±0.11^{aB}	-4.78±0.04^{cB}	9.13±0.38^{dB}	117.71±0.79^{aB}	10.31±0.35^{eB}	9.00±0.60^{dB}	13.24±0.37^{aB}
Water Blanching (80°C)							
1	62.78±0.14^{bC}	-3.73±0.08^{aA}	14.46±0.21^{aA}	104.62±0.41^{cC}	14.93±0.20^{aA}	20.77±0.44^{aA}	7.36±0.21^{cC}
2	63.01±0.13^{bC}	-3.82±0.02^{aA}	13.80±0.10^{bA}	105.48±0.18^{cC}	14.32±0.10^{bA}	19.29±0.22^{bA}	8.04±0.10^{bC}
4	63.70±0.31^{aC}	-3.89±0.13^{abA}	12.93±0.37^{cA}	106.50±0.95^{bC}	13.50±0.33^{cA}	17.31±0.80^{cA}	9.00±0.36^{aC}
6	63.80±0.10^{aC}	-3.84±0.06^{aA}	12.82±0.32^{cA}	106.64±0.48^{bC}	13.38±0.30^{cA}	17.12±0.59^{cA}	9.12±0.30^{aC}
8	64.05±0.09^{aC}	-4.04±0.04^{bA}	12.80±0.31^{cA}	107.36±0.37^{abC}	13.42±0.30^{cA}	16.77±0.58^{cA}	9.19±0.31^{aC}
10	64.24±0.17^{aC}	-4.22±0.16^{cA}	12.72±0.23^{cA}	108.24±0.80^{aC}	13.40±0.21^{cA}	16.35±0.52^{cA}	9.32±0.23^{aC}

Values expressed as the mean ± standard deviation.
The same small letters in the same column and at the same temperature indicate no significant difference between the times ($p \leq 0.05$).
The same capital letters in the same column for the same time indicate no significant difference between the different temperatures ($p \leq 0.05$).

Table 4 – Kinetic parameters of the Browning Index for the different blanching conditions.

	Temperature (°C)	k_L (min^{-1})	R^2	k_R (min^{-1})	R^2
Water	80	1.903±0.206	0.99	0.010±0.001	0.96
Water	90	3.581±0.213	0.94	0.067±0.015	0.93
Steam	100	5.259±0.161	0.98	0.080±0.005	0.99

Values expressed as the mean ± standard deviation.

CAPÍTULO 3

Study of the hot air drying and freeze-drying of garlic (*Allium Sativum L.*)

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Study of the hot air drying and freeze-drying of garlic (Allium Sativum L.)

Luciane Fante & Caciano Pelayo Zapata Noreña*

Institute of Food Science and Technology, Federal University of Rio Grande do Sul, Porto Alegre, RS, Brazil.

Summary

The kinetics of drying garlic using hot air, with and without prior blanching was studied, also the color changes, inulin, glucose and fructose contents, particle size, microstructure and glass transition temperatures of garlic powders obtained by air drying and by freeze-drying. The garlic cloves were peeled and cut into slices with a diameter of 15±2.40 mm and thickness of 1±0.35 mm. For blanching they were distributed uniformly in metal baskets and placed in an autoclave generating steam at a temperature of 100ºC for 4 minutes. The drying trials were carried out in a forced air dryer at temperatures of 50, 60 and 70ºC for 6 hours. For the freeze-drying process the garlic slices, previously frozen at -80ºC, were freeze-dried at a pressure of 64 µmHg for 48 hours. The kinetics of forced air-drying indicated an increase in the drying constant with increase in temperature and with the use of blanching. For the freeze-dried garlic powder the parameter L^* was greater and the parameters a^* and b^* smaller than for the forced air dried garlic powder, obtaining lighter, greener and bluer samples, closer to the color of *in natura* garlic. A decrease in the inulin content and increase in the glucose and fructose contents was observed in the dehydrated samples as compared to the *in natura* sample, indicating possible hydrolysis of the inulin which could be related to residual activity of the enzyme inulinase. A comparison of the garlic samples dehydrated with and without blanching showed lower concentrations of inulin, glucose and fructose in the pre-blanched samples, due

to possible leaching of the sugars. The mean particle size of the freeze-dried powder was smaller than that of the forced air- dried powder and showed a less wrinkled surface. The glass transition temperature of the powdered garlic samples increased with decrease in water activity, confirming the plasticizing effect of the water.

Keywords: garlic, dehydration, inulin, color.

* C*orrespondent: Fax: +55-51-3308*7048*; e-mail: czapatan@ufrgs.br*

Introduction

Garlic (*Allium sativum L.*) is a herbaceous plant recognized for its numerous medicinal and culinary properties, used in diverse food preparations for its characteristic flavor and odor [1]. Its main chemical constituents are allicin, carbohydrates, phosphoric and sulfuric acids, proteins and mineral salts [2].

Allicin is responsible for the characteristic odor and the antimicrobial, anti-inflammatory, anti-thrombosis, anti-cancer and anti-atherosclerosis activities and also for the antioxidant effect [3]. Amongst the carbohydrates present is inulin, which belongs to the group of polysaccharides called fructans, composed of a main chain of fructose units connected by β(2→1) bonds and by a glucose molecule attached to the initial part of the linear fructose chain by a type (α1 → β2) bond [4].

Inulin is present in vegetable products such as chicory, onions and garlic, being classified as a prebiotic substance and as a soluble dietary fiber since it is resistant to digestion in the small intestine, passing practically intact to the large intestine, where it is fermented by bifidobacteria [4].

The use of specific technological treatments such as enzyme inactivation and drying is necessary in order to preserve the garlic, thus increasing its shelf life and making it available

for processing and consumption throughout the entire year [5]. Dehydrated garlic powder is of high commercial value and is used as a seasoning or standard ingredient in food preparations and formulations [6].

Garlic cloves, when peeled and exposed to the environment undergo undesirable alterations in their quality such as rapid browning [7]. The enzyme polyphenoloxidase is responsible for the appearance of these brown substances as a result of the oxidative polymerization of quinones, but their production can be avoided by the use of heat treatment [8]. Enzyme inactivation improves the color of the products, preventing the discoloration, making the foods more attractive for consumption and increasing their luminosity [9].

The dehydration process improves food stability, since it reduces the water activity considerably and minimizes the physical, chemical and microbiological alterations during storage [10]. The removal of water is the main task in the preservation of foods, reducing the moisture contents to a level that allows for their safe storage for a prolonged period of time [11].

Dehydration by freeze-drying is considered to be one of the best methods for drying heat sensitive foods, using low temperatures during drying and eliminating the water by sublimation [12]. Freeze-dried products are characterized by showing good hydration properties and minimal changes in the flavor and color when compared to products obtained by forced air drying [13]. However, the freeze-drying process involves high production costs and the drying rate is generally low with elevated drying times, which can vary from a few hours to up to three days [12].

The objectives of this work were to study the drying kinetics of sliced garlic, with and without blanching, drying in forced air at 50, 60 and 70ºC, also studying the color changes,

inulin, glucose and fructose concentrations, particle size, microstructure and glass transition temperature of garlic powders obtained by air drying and by freeze-drying.

Material and methods

Material

The garlic (*Allium sativum L.*), cultivated in the city of Nova Pádua in the state of Rio Grande do Sul, Brazil, was acquired directly from the producer. The heads were cleaned and selected considering the absence of visual injury and infections and also uniformity in size and color. They were then stored at room temperature (22±2ºC) until used.

Methods

Experimental procedure

The garlic cloves were peeled and sliced using a food processor, presenting diameters and thicknesses of 15±2.40 mm and 1±0.35 mm, respectively. The samples were then submitted to a steam blanching process where the slices were uniformly distributed in metal baskets and placed in an autoclave generating steam at 100ºC and atmospheric pressure for 4 minutes [14]. After blanching, the samples were cooled in an ice bath for 3 minutes [9]. The garlic slices, with and without blanching, were subsequently distributed uniformly in stainless steel trays and placed in forced air dryers at 50, 60 and 70ºC. During drying, the loss in weight of the material was determined by weighing every 5 minutes during the first hour, every 15

minutes in the second hour, and at 30 minute intervals for the rest of the time until a total time of 6 hours of drying. The water activity (a_w) was also measured at 15 minute intervals during the first two hours of drying and then every 30 minutes up to the end of drying [14]. Garlic samples were also dehydrated by freeze-drying according to Alves [15], which consisted of previously freezing the samples in an ultrafreezer (Liobras, Brazil) at -80ºC for 24 hours. The frozen garlic slices were then arranged in trays inside the freeze dryer (Liobras L101, Brazil) and freeze-dried at a pressure of 64 µmHg, taking a mean time of 48 hours.

Analytical determinations

The following determinations were carried out on the *in natura* sample: pH using a pHmeter (QUIMIS) and A.O.A.C. method n°981.12 [16], moisture content using A.O.A.C. method n°984.25 [17] and soluble solids by refractometry (Atago, NAR T3, Atago Co, Ltd,, Tokyo, Japan) at 20 °C, according to A.O.A.C. method n°932.12 [18]. The water activity (a_w) was measured directly in an electronic equipment (Aqualab 3TE-Decagon, Pullman, USA) at 25ºC according to A.O.A.C. method n°978.18 [19].

Drying kinetics

With the objective of evaluating the variation in moisture content and a_w of the samples dehydrated in hot air, the values for moisture content on a dry weight basis and for a_w were expressed non-dimensionally (W) according to Equation 1 [20];

$$W = \frac{X_{wt} - X_{we}}{X_{wo} - X_{we}} \quad (1)$$

where X_{wo}, X_{we} and X_{wt} are the values for moisture content on a dry weight basis or the initial a_w at equilibrium and at time *t* respectively. The values at equilibrium were estimated as from the drying curves when these became asymptotic to the abscissa axis [21].

The data obtained in the drying trials were fitted to the empirical equations of Henderson-Pabis, Page & Newton [22]:

Henderson-Pabis: $\mathrm{W} = k_1 \exp(-k_2.t)$ (2)

Page: $\mathrm{W} = \exp(-k_3.t^{k4})$ (3)

Newton: $\mathrm{W} = \exp(-k_5.t)$ (4)

where W is the non-dimensional moisture content or a_w, k_1, k_2, k_3, k_4 and k_5 are constants to be estimated and *t* is the drying time.

Color measurement

The dehydrated garlic was ground in a Pulverizzette 14 hammer mill (Fritsch, Idar-Oberstein, Germany) at 1200 rpm, with a Tyler 0.5 mesh sieve, and placed in 5 cm diameter x 1 cm height Petri dishes, and filled to the top [23]. The color was measured by direct reading in a Minolta colorimeter (CR400/410) using the tridimensional CIE*L*a*b** system, where *L** indicates the luminosity (varying from 0 – black to 100 – white), *a** is a measurement varying from green (-60) to red (+60), and *b** varied from blue (-60) to yellow (+60). The instrument was standardized with a white ceramic plate (*L**= 97.47; *a**= 0.08; *b**= 1.76). The parameters *L* a* b** were used to calculate the values for *Chroma*, the *Hue* angle and the browning index (*BI*) as from the equations described by Ndiaye [24].

Determination of the sugars by HPLC

The samples were prepared according to the methodology of Scher [14] and adapted by Toneli [25]. Two grams of dehydrated garlic were ground with 60 mL water at 90ºC and the product then heated in a water bath at 80ºC for one hour with constant stirring. The suspension was then cooled to room temperature and centrifuged at 1680 x g for 15 minutes at 25°C (Centrifuge 5415R). The supernatant was filtered through Whatman nº1 filter paper and then through a 22 μm membrane filter and stored at -18ºC. For the analysis, the samples were pre-heated in a water bath at 80ºC and then placed in an ultrasonic bath for 4 minutes before injection into the HPLC.

The inulin, glucose and fructose contents were determined using an adaptation of the method described by Zuleta & Sambucetti [26] by direct determination using high performance liquid chromatography (HPLC-RI) with a Perkin Elmer series 200 chromatograph, refractive index detector, water (Milli-Q) as the mobile phase at 0.6 mL/min, temperature of 80ºC, a Phenomenex Rezex RHM Monosaccharide column (300 x 7.8 mm) and a total run time of 13 minutes.

Granulometric analysis

The particle size distribution of the powdered dehydrated garlic was measured by laser ray diffraction in the equipment CILAS 1180 L/D. Approximately 1 g of sample was shaken with filtered water at room temperature (22 ± 2°C) in order to disperse the particles according to the methodology employed by Ma [27]. The software Size Expert, provided with the equipment, was used to measure the diameters, calculate the mean diameter and the particle

size distribution according to the range of diameters contained within the accumulated percent fractions of 10, 50 and 90% (D[0.1], D[0.5] and D[0.9]).

Scanning Electronic Microscopy

The microstructures of the dehydrated garlic powders were visualized by scanning electronic microscopy using the method recommended by Toneli [28], which consisted of weighing approximately one gram of sample onto double faced adhesive tape fixed to a metal stub. The samples were sputtered with a fine layer of metal and viewed under the scanning electronic microscope (MEV) (JSM 6060, Tokyo, Japan) operating at 10 kV.

Glass Transition Temperature

The glass transition temperature of the dehydrated garlic powder was determined by differential scanning calorimetry using the equipment DSC Q20, TA Instruments (New Castle, DE, USA) according to the method recommended by Telis & Sobral [29], and ultra-pure nitrogen as the purging gas with a flow rate of 50 mL/min. Approximately 10 mg sample was weighed into hermetically sealed aluminum pans. The initial sample temperature was -10ºC, and it was heated to 215ºC, cooled to -10°C and again heated to 215ºC at a rate of 20ºC/min. The glass transition temperatures (T_g) were determined from the thermograms obtained on the DSC using the Universal Analyses 2000 software.

Statistical analysis

ANOVA was used for the statistical analyses and the treatments compared by way of Tukey's multiple means comparison tests. The statistical software SAS version 6.2 was used to analyze the results and the kinetic drying parameters were estimated using non-linear regression with the Statistica version 7.0 software. All these analyses were carried out in triplicate.

Results and discussion

Raw material

The moisture content of the *in natura* garlic was 64.15±0.09%, close to the value found by Haciseferogullari [30] of 66.32%, and the water activity (a_w) was 0.986±0.001, typical of fresh products which vary from 0.97 to 0.99 [31]. The moisture content of the blanched garlic was 67.35±0.07% and the a_w 0.992±0.001, observing a slight increase due to absorption of water during steam blanching. With respect to the soluble solids content, the *in natura* garlic showed values of 36.00±0.85 °Brix, close to the values reported by Mota [32] which varied from 29.33 to 37.16 °Brix, and by Resende [33] of from 35.75 to 37.25 °Brix. With respect to pH, a value of 6.41±0.01 was found, similar to the values found by Montaño [34] and Haciseferogullari [30].

Forced air drying curves

Figure 1a shows the moisture content of the non-blanched garlic as a function of drying time at the temperatures of 50, 60 and 70ºC. It can be seen that the moisture content decreased

quickly in the first 40 minutes, the drying rate increasing with increase in temperature. The rate of water loss then decreased until reaching constant weight, corresponding to the values for the equilibrium moisture content estimated on the asymptotic part of the drying curve [35]. Under these conditions the moisture contents on a dry weight basis corresponded to 0.1020±0.0007, 0.0980±0.0004 and 0.0920±0.0007 g/g of dry matter at the temperatures of 50, 60 and 70 °C respectively. The equilibrium moisture contents decreased with increase in temperature but the decrease was not significant (p>0.05) (Table 1). On dehydrating various vegetables, Krokida [36] also observed a decrease in the equilibrium moisture contents with increase in temperature, but without significant differences.

On dehydrating 2 mm thick garlic slices at temperatures of 50, 70 and 90ºC, Madamba [37] observed a decrease in moisture content with increase in temperature. Sacilik [38], dehydrating 3 and 5 mm thick garlic slices at temperatures of 40, 50 and 60ºC, also observed an increase in drying rate with increase in temperature, but again the differences were not significant. The increase in drying rate with increase in temperature occurs due to the decrease in external resistance and due to a greater transference of water during the first drying stages [5]. Kaya [39] also indicated that an increase in temperature of the drying air increased the rate of heat transfer and, consequently, reduced the total drying time. During in this period drying the water transfer mechanism occurs by mass diffusion [40].

Figure 1b shows the moisture content of the blanched garlic as a function of drying time at the temperatures of 50, 60 and 70ºC. It can be seen that the moisture content decreased quickly in the first 30 minutes, the drying rate increasing with increase in temperature. The rate of water loss then decreased until reaching constant weight, corresponding to the values for the equilibrium moisture content estimated on the asymptotic part of the drying curve as in the previous case. Under these conditions the moisture contents on a dry weight basis

corresponded to 0.0950±0.0008, 0.0900±0.0007 and 0.0860±0.0003 g/g of dry matter at the temperatures of 50, 60 and 70°C respectively. The equilibrium moisture contents decreased with increase in temperature but the decrease was not significant (p>0.05) (Table 1).

On comparing the drying curves (Figure 1) it can be seen that for the same temperature, the dehydrated samples with enzymatic inactivation showed greater moisture losses than the samples without inactivation. This occurred because blanching contributes to increasing the surface for heat transfer and to the modifications in the initial sample structure, improving the transfer of moisture and resulting in greater drying rates [5, 41].

For each drying temperature, the samples with blanching reached equilibrium conditions in shorter times than the samples dehydrated without prior blanching, since the rates of water transfer increased with this pre-treatment. A similar behavior was reported by Hatamipour [10] for the dehydration of potatoes and by Scher [14] for the dehydration of yacon tubers, both with and without enzymatic inactivation.

Figure 2a shows the values for the water activity (a_w) of the non-blanched garlic as a function of drying time at the temperatures of 50, 60 and 70ºC. It can be seen that the a_w decreased rapidly in the first hour of drying, the a_w becoming lower the higher the temperature. After this period, the a_w continued decreasing, but slowly, until reaching equilibrium. The values for a_w at equilibrium estimated from the asymptotic part of the drying curve were 0.521±0.004, 0.451±0.004 and 0.381±0.005 at the temperatures of 50, 60 and 70°C respectively. The values for water activity at equilibrium decreased significantly (p>0.05) with increase in temperature (Table 1). On dehydrating cassava, Lertworasirikul [42] also observed a significant decrease in the values for a_w at equilibrium with increase in temperature.

Similar behavior was shown in Figure 2b for the blanched garlic slices, but less time was required to reach the equilibrium a_w, since blanching modified the physical properties of the tissues causing heat destruction of the cell membrane [43] and modifying some physical properties such as porosity [44]. The values for a_w at equilibrium estimated from the asymptotic part of the drying curve were 0.518±0.001, 0.428±0.003 and 0.376±0.003 at the temperatures of 50, 60 and 70°C respectively. The values for water activity at equilibrium decreased significantly (p>0.05) with increase in temperature (Table 1).

Table 1 shows the values for moisture content and water activity at equilibrium under dynamic conditions during the drying period, obtained from Figures 1 and 2 for the period where the moisture and a_w curves became asymptotic.

Drying kinetics in forced air

Table 2 shows the estimates for the kinetic parameters of the models proposed for the non-dimensional moisture content as a function of drying time for the garlic slices. It can be seen that the values for R^2 for all the models were above 0.98, indicating a good fit. It can also be seen that for the Henderson-Pabis and Newton models there was an increase in the drying constants with increase in temperature and with the use of blanching, whereas with the Page model, the drying constants only increased with the use of blanching and not with increase in temperature. According to Pezzutti [6], who studied the drying kinetics of garlic dehydrated at temperatures from 45 to 75ºC, they also observed an increase in the drying constants with increase in temperature, using the model proposed by Henderson-Pabis. Doymaz [45] on studying the drying kinetics of leek slices with a thickness of 1 cm, submitted to water blanching at 70ºC for 3 minutes and dehydrated at 50ºC, found values for k of 0.043 and

0.055 min^{-1} for the product without and with blanching, respectively, using the model proposed by Henderson-Pabis.

Figure 3 represents the Henderson-Pabis model, which showed the highest correlation coefficients (R^2=0.99) during the drying of garlic.

On employing the above models to evaluate the behavior of the non-dimensional a_w during drying, Table 3 shows that the models that best predicted the experimental values were the Henderson-Pabis and Page models (R^2=0.98), indicating a good fit with these models. The drying constants also increased with increase in temperature and with the use of blanching for all the models proposed.

Figure 4 represents the Henderson-Pabis model during the drying of the garlic.

Color changes

Table 4 shows the values for the parameters of L^*, a^* and b^* in the garlic powders dehydrated by forced air and by freeze-drying, with and without blanching. It can be seen that the values for L^* were significantly higher (p<0.05) in the freeze-dried garlic, with and without blanching as compared to the samples dehydrated by forced air at all the temperatures used, the freeze-dried samples being lighter in color. Li [46], studying the dehydration of garlic slices, also reported slightly higher values for L^* for the freeze-dried samples in comparison with those dehydrated in a vacuum dryer at 40ºC. When studying the dehydration of green pepper puree, Topuz [47] also found significantly higher values for L^* for samples dehydrated by freeze drying for 8 days as compared to those dehydrated with forced air at 60ºC for 7 hours.

For the air-dried non-blanched garlic samples, there was a significant decrease ($p<0.05$) in the parameter L^* with increase in temperature, making the samples darker. This observation was also reported by Sacilik [38] on dehydrating garlic slices with 10-12 mm in diameter and 3-5 mm thickness, using a forced air dryer at 40 and 60ºC. For the air-dried pre-blanched garlic, there was a significant decrease ($p<0.05$) in the parameter L^* with increase in temperature, from 50 to 60ºC.

With respect to the use of blanching, there was a significant increase ($p<0.05$) in the parameter L^* for the non-blanched samples as compared to the blanched samples, the non-blanched samples being lighter in color.

The parameter a^* was significantly lower ($p<0.05$) for the freeze-dried garlic with and without blanching as compared to the samples dehydrated by forced air, the freeze-dried samples being greener ($-a^*$). With respect to forced air dehydration, there was a significant increase ($p<0.05$) in this parameter at 70ºC for the samples with and without blanching.

Regarding the use of pre-blanching, it can be seen that the parameter a^* was significantly higher ($p<0.05$) for the non-blanched samples, which were redder in color.

The parameter b^* was significantly lower ($p<0.05$) in the freeze-dried garlic, with and without blanching, than in the forced air dried samples, the freeze-dried samples being bluer ($-b^*$). For the forced air dried samples there was a significant increase ($p<0.05$) in this parameter with increase in temperature for the non-blanched garlic, but for the blanched samples there was no significant difference ($p<0.05$) between the temperatures.

For to the use of pre-blanching, with the exception of those dried at 70 ºC, it can be seen that the parameter b^* was significantly higher ($p<0.05$) for the non-blanched samples, which were more yellow in color.

On dehydrating red pepper slices with 4 mm of thickness at temperatures from 50 to 90ºC, Gálvez [48] observed a slight increase in the values for a^* and b^*, and decrease in the values for L^* with increase in drying temperature, becoming darker in color due to a greater influence of the Maillard reaction, especially at the higher temperatures.

Table 5 shows the values obtained for *Chroma*, *Hue* and *BI*. It can be seen that the values for *Chroma* of the freeze-dried garlic, with and without blanching, were significantly lower ($p<0.05$) as compared to the forced-air dried samples, the freeze-dried samples being bluer. With respect to air drying, there was a significant increase ($p<0.05$) in *Chroma* with increase in temperature for the non-blanched samples, which became more yellow, but for the blanched samples, there was no significant difference ($p>0.05$) between the temperatures.

With respect to the effect of pre-blanching, it was observed that the values for *Chroma* were significantly lower ($p<0.05$) when freeze-dried and significantly higher ($p<0.05$) when dehydrated by forced air, with the exception of the use of 70ºC.

The values for the *Hue* angle were found in the second quadrant for all the samples. For the freeze-dried garlic, with and without blanching, the values for *Hue* were significantly higher ($p<0.05$) when compared to the forced air dried samples. With respect to air drying, the values for *Hue* of the non-blanched samples were significantly lower ($p<0.05$) at 70ºC, whereas for the blanched samples, the values for *Hue* decreased significantly ($p<0.05$) with increase in temperature, the samples becoming redder.

With respect to the effect of pre-blanching, it was observed that the values for *Hue* were significantly higher ($p<0.05$) for the dehydrated blanched samples for all the temperatures used.

The browning index (*BI*) was significantly lower ($p<0.05$) in the freeze-dried garlic, with or without blanching, when compared to the samples dehydrated using forced air at all

the temperatures employed, as also observed by Topuz [47] on dehydrating 1 cm thick pepper slices in a freeze-drier at -70ºC and in forced air at 60ºC. In the non-blanched air-dried garlic samples, there was a significant increase ($p<0.05$) in *BI* with increase in temperature, whereas for the blanched samples there was only significant difference ($p<0.05$) between 50 and 70ºC. On dehydrating 3.5 mm thick potato slices in a forced air drier at temperatures of 70, 80 and 90ºC, Leeratanarak [49] also reported an increase in *BI* with increase in temperature, indicating a greater degree of browning due to a greater influence of the Maillard reaction with increase in temperature.

According to Sacilik [38], with respect to the color properties, lower values for a^* and higher values for *Hue* and L^* would be ideal for garlic slices, due to their proximity to the natural color of garlic. In the present study, the lowest value for a^* and the highest values for the angle *Hue* and for L^* were found in the non-blanched, freeze-dried garlic samples.

Effect of drying on the sugar concentrations

Table 6 shows the inulin, glucose and fructose concentrations in the *in natura* and dehydrated garlic samples. It can be seen that the inulin content decreased and the glucose and fructose contents increased significantly ($p<0.05$) after freeze- or forced air drying as compared to the *in natura* garlic. These results could indicate hydrolysis of the inulin to reducing sugars. Graefe [50] reported the hydrolysis of inulin to reducing sugars in yacon roots with the increase in storage temperature. In a study on the formation of inulin gel, the use of high temperatures (above 80 ºC) resulted in hydrolysis of the polysaccharide to simple sugars, the degree of conversion increasing with increase in temperature [51]. The same was observed by Ishiguro [52] when studying the storage of burdock roots at temperatures from 0 to 20ºC. The

increase in the reducing sugar concentration could be related to the residual activity of inulinase in the product.

The freeze-dried samples showed significantly higher ($p<0.05$) inulin contents than the forced-air dried samples, due to the lower drying temperature employed. Saengthongpinit [53], studying the storage of Jerusalem artichoke at different temperatures, also observed higher inulin concentrations with decrease in temperature, obtaining concentrations of 31.67%, 27.48% and 23.64% at temperatures of -18°C, 2°C and 5°C respectively.

In the non-blanched forced air dried samples, the inulin content was significantly lower ($p<0.05$) at 70 ºC and there was a significant increase ($p<0.05$) in glucose from 50 to 70ºC, whereas for fructose there was no significant difference ($p>0.05$) between the temperatures. In the pre-blanched garlic samples the inulin concentration was significantly lower ($p<0.05$) with increase in temperature and for glucose there was a significant increase ($p<0.05$) at 60ºC, whereas the fructose concentration was significantly higher ($p<0.05$) at 70ºC.

It can also be seen that the inulin, glucose and fructose concentrations were higher in the non-blanched samples than in the pre-blanched samples. Bomben [54] showed losses in product during steam blanching due to leaching of the solids by evaporation and condensation of the water.

Particle size

The granulometric distribution of food powders has been considered as one of the most important physical properties, since it directly affects their fluidity and adherence and is related to solubility of the products [55].

Table 7 shows the particle size distribution of the various garlic powders. A considerable variation in particle diameter can be seen, the non-blanched samples showing mean diameters in the range from 79.35 µm to 201.61 µm and the pre-blanched samples from 104.79 µm to 302.04 µm, the particle diameters being larger in all the pre-blanched samples, suggesting the occurrence of cell grouping as reported by Chantaro [56] in a study on carrot peel powders with and without water blanching at 90ºC for 1 minute.

The freeze-dried garlic, with and without pre-blanching, showed smaller-sized particles than the forced-air dehydrated samples, possibly due to the formation of more porous particles obtained by sublimation of the water during freeze drying [12]. The same was observed by Kim [57] on studying the particle size of nano-suspensions dehydrated by freeze-drying and in conventional driers from 40 to 80ºC.

With respect to the particle sizes of the pre-blanched and non-blanched forced air-dried samples, smaller particles were observed at 60 and 70ºC when compared to drying at 50ºC. According to Lee [58], an increase in drying temperature results in greater drying rates with lower final or equilibrium moisture contents, and consequently with smaller particles showing better powder re-dispersion properties.

Figure 5 shows the frequency histogram and particle size distribution in garlic powder samples pre-blanched and dehydrated at 50 ºC and non-blanched and freeze-dried, corresponding to the distributions of the largest and smallest particle sizes, respectively.

Microscopy

Electronic microscopy has been used to observe the particle agglomeration characteristics in powders, including the form and size [59].

Figure 6 shows the microscopic images of garlic powder, with and without pre-blanching. Microstructures with a great variety of sizes and forms can be observed and also isolated particles. In the case of the non-blanched samples dehydrated in air, the particles showed a laminar type structure with wrinkles over the entire surface, as well as deformations that became more accentuated with increase in the air drying temperature (Fig. 6a, b and c), forming more pointed microstructures. The non-blanched freeze-dried samples (Fig. 6d) presented some particles with a tendency to an irregular spherical geometry and others with the aspect of broken glass, typical of freeze-dried products as observed by Chronakis [60] for oat products.

With respect to the blanched samples (Fig. 6e, f, g and h), irregular microstructures with more accentuated wrinkling of the surface were observed when compared with the non-blanched products, caused by the pre-treatment which modifies the structural properties of the tissues and consequent deformation of the particles [43]. Nevertheless the freeze-dried samples showed less surface damage than the forced-air dried samples. According to James [61] freeze-drying results in less contraction and consequently less wrinkling of the samples as compared to air drying, although structural damage can be caused by the formation of ice crystals during the freezing process prior to freeze drying.

Glass Transition Temperature

Figure 7 shows the thermograms (energy flow as a function of temperature) of the freeze-dried and forced air-dried at 50 ºC garlic powders, both non-blanched, representing the samples with the highest and lowest glass transition temperatures (T_g) as obtained by DSC. According to Tonon [62], the thermograms show a typically second order transition,

producing a change in heat flow due to changes in the heat capacity at a phase transition temperature. The glass transition temperature was taken as the mid-point of the glass transition.

Table 8 shows the values for T_g obtained for the different values of water activity for the garlic powder samples. It can be seen that the values for T_g of the freeze-dried garlic powders were higher than those of the forced air-dried powders, due to their lower a_w values, consequence of the fact that the freeze-drying process removed a greater amount of free water according to Bai [63] and Guizani [64]. In the case of the freeze-dried garlic samples, it can be seen that the value for T_g of the pre-blanched product was lower than that of the non-blanched product, due to incorporation of water with the use of this pre-treatment. Deng [65] dehydrated apple slices with forced air at 55ºC and by freeze-drying, and also observed that the values for T_g decreased with increase in the a_w values in the product, obtaining values for T_g of -3.41°C and 2.19°C for a_w values of 0.318 and 0.282, respectively. Slade & Levine [66] suggested that the decrease in moisture content leads to a reduction in the free volume, increase in viscosity and consequent decrease in mobility of the molecules in the amorphous region, resulting in an increase in T_g.

According to Collares [67] water is a well known plasticizer capable of decreasing the glass transition temperature and the mechanical resistance, resulting in a softening effect with an increase in its concentration. The effect of the plasticizing capacity of water in decreasing the value of T_g was shown by Telis & Sobral [68] when studying the T_g of freeze-dried pineapple, obtaining values of -58.2, -36.5 and 46 °C for a_w values of 0.90, 0.65 and 0.11 respectively, and by Tonon [62], studying the T_g of spray-dried acai juice containing Arabic gum, obtaining values of -56.32, 6.11 and 74.37°C for a_w values of 0.843, 0.689 and 0.112 respectively.

Conclusions

The kinetics of forced air drying indicated an increase in the drying constant with increase in temperature and with the use of blanching. The values for the moisture content at equilibrium obtained for the dehydration of garlic with and without blanching were in the range from 0.086 to 0.102 g/g dry matter, whilst the values for the water activity at equilibrium were below 0.521.

The color parameter L^* was significantly greater and the parameters a^* and b^* significantly lower for garlic dehydrated by freeze-drying with and without pre-blanching, when compared with forced air-drying, obtaining lighter, greener and bluer samples, closer to the color of *in natura* garlic. With respect to forced air dehydration, the parameter L^* decreased and the parameters a^* and b^* increased with increase in temperature, making the samples darker, redder and more yellow.

With respect to the sugar contents, decreases in the inulin concentrations and increases in the glucose and fructose contents were observed in the dehydrated garlic samples as compared to the *in natura* sample, indicating hydrolysis of the inulin which could be related to residual inulinase activity in the product. Higher inulin contents were found in the freeze-dried samples as compared to the hot air dehydrated samples due to the lower drying temperature employed in freeze-drying. On comparing the samples dehydrated with and without prior blanching, lower inulin, glucose and fructose concentrations were found in the pre-blanched samples due to possible leaching of the sugars.

The mean particle diameters were smaller in the freeze-dried samples than in the forced air dehydrated samples, and electronic microscopy showed that the former had less wrinkled surfaces. The glass transition temperatures of the dehydrated garlic powders were

higher the lower the value of the water activity of the samples, confirming the plasticizing effect of the water.

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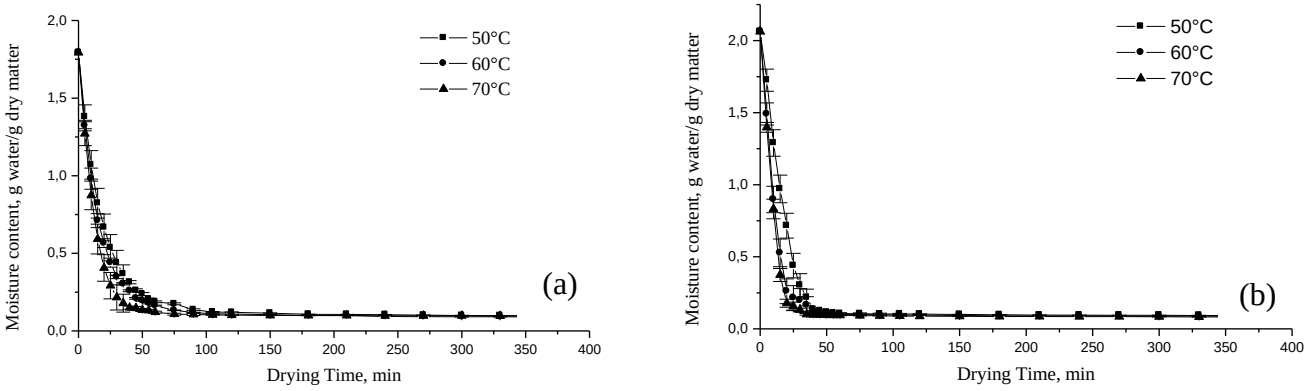

Figure 1 – Moisture content (dry weight basis) as a function of drying time for (a) non-blanched and (b) blanched garlic slices.

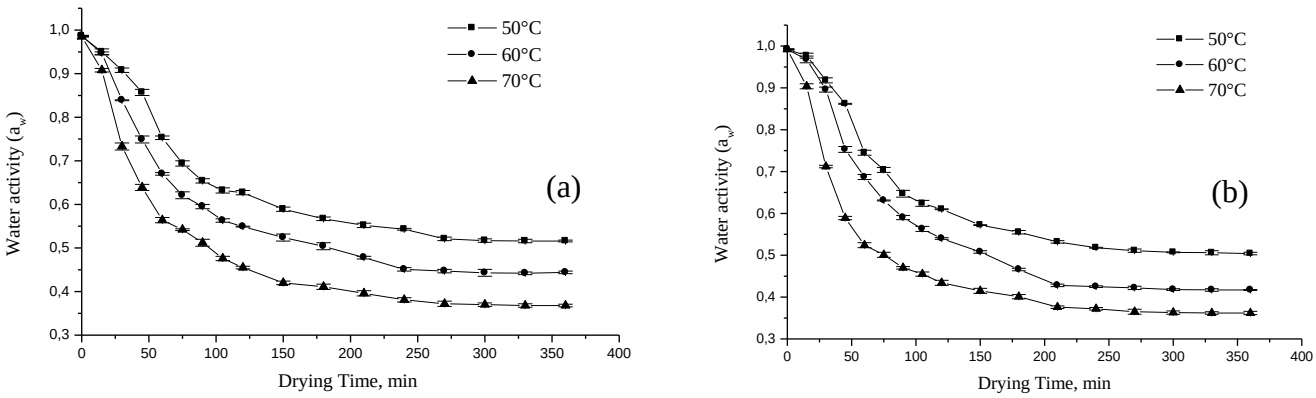

Figure 2 – Water activity as a function of drying time for (a) non-blanched and (b) blanched garlic slices.

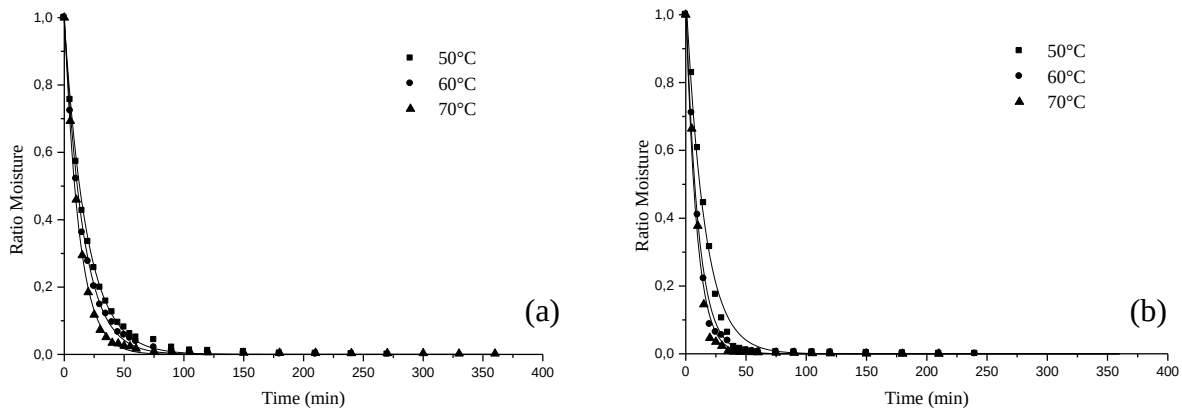

Figure 3 – Drying kinetics for the non-dimensional moisture content as a function of drying time for non-blanched (a) and blanched (b) garlic slices.

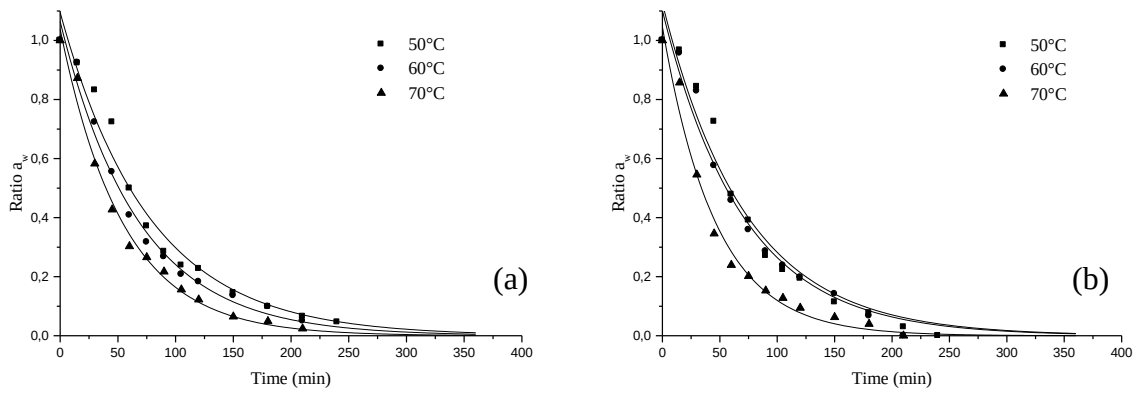

Figure 4 - Drying kinetics for the non-dimensional a_w as a function of drying time for non-blanched (a) and blanched (b) garlic slices.

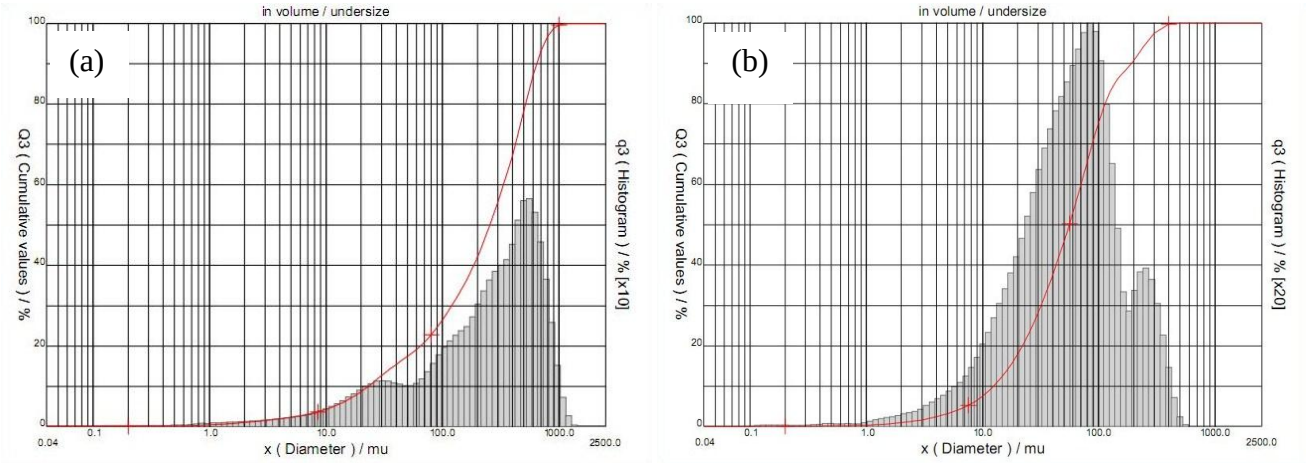

Figure 5 – Frequency histogram and particle size distribution in garlic powder samples (a) pre-blanched and dehydrated at 50ºC and (b) non-blanched and freeze-dried

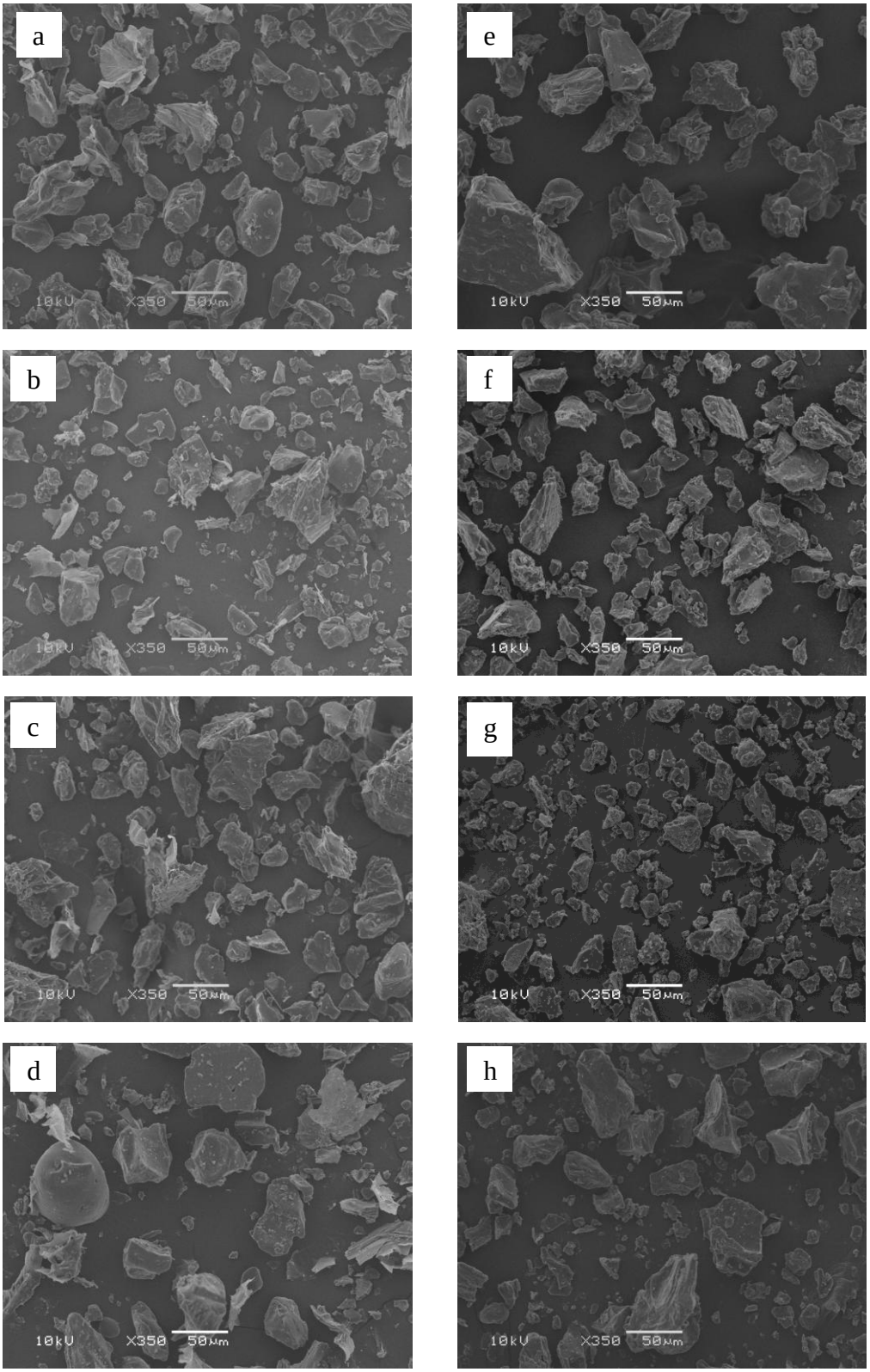

Figure 6 – Microscopic images with magnification of x350 of the garlic powders: non-blanched forced-air dried at (a) 50°C, (b) 60°C and (c) 70°C and (d) freeze-dried; and pre-blanched forced-air dried at (e) 50°C, (f) 60°C and (g) 70°C and (h) freeze-dried.

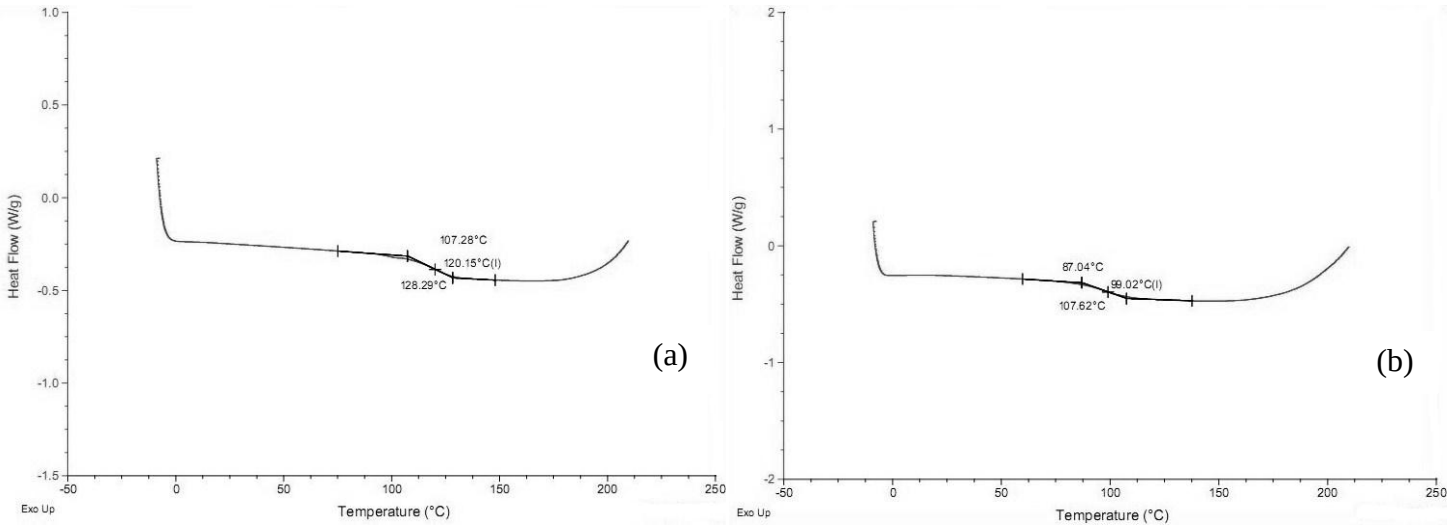

Figure 7 – Thermograms of the glass transition of non-blanched garlic powders (a) freeze-dried and (b) dehydrated in hot air at 50ºC.

Table 1 – Values for moisture content and water activity at equilibrium in the garlic slices with and without blanching

Drying temperature (°C)	Pre-treatment	Time to reach equilibrium (min)	a_w content at equilibrium	Moisture content at equilibrium (DWB)
50	Non-blanched	270	0.521±0.004[a]	0.102±0.0007[a]
	Blanched	240	0.518±0.001[a]	0.095±0.0008[a]
60	Non-blanched	240	0.451±0.004[b]	0.098±0.0004[a]
	Blanched	210	0.428±0.003[b]	0.090±0.0007[a]
70	Non-blanched	240	0.381±0.005[c]	0.092±0.0007[a]
	Blanched	210	0.376±0.003[c]	0.086±0.0003[a]

Values expressed as the mean ± standard deviation.
The same small letters in the same column indicate no significant difference (p>0.05).

Table 2 – Kinetic parameters for the non-dimensional moisture content as a function of time for garlic slices with and without blanching for the Henderson-Pabis, Page and Newton models

Temperature	Pre-treatment	Henderson-Pabis		Page		Newton	
		k_2(min^{-1})	R^2	k_3(min^{-1})	R^2	k_5(min^{-1})	R^2
50°C	Non-blanched	0.053±0.0006	0.99	0.064±0.0022	0.98	0.054±0.0005	0.98
	Blanched	0.065±0.0043	0.99	1.393±0.0010	0.98	0.061±0.0025	0.98
60°C	Non-blanched	0.063±0.0009	0.99	0.076±0.0031	0.98	0.063±0.0007	0.98
	Blanched	0.098±0.0029	0.99	1.304±0.0031	0.98	0.094±0.0031	0.98
70°C	Non-blanched	0.082±0.0008	0.99	0.066±0.0031	0.98	0.080±0.0010	0.98
	Blanched	0.112±0.0048	0.99	1.401±0.0022	0.98	0.108±0.0042	0.98

Values expressed as the mean ± standard deviation.

Table 3 - Kinetic parameters for the non-dimensional a_w as a function of time for garlic slices with and without blanching for the Henderson-Pabis, Page and Newton models

Temperature	Pre-treatment	Henderson-Pabis		Page		Newton	
		k_2(min^{-1})	R^2	k_3(min^{-1})	R^2	k_5(min^{-1})	R^2
50°C	Non-blanched	0.013±0.0008	0.98	0.004±0.0001	0.98	0.036±0.0007	0.94
	Blanched	0.014±0.0010	0.98	0.007±0.0005	0.98	0.039±0.0009	0.96
60°C	Non-blanched	0.015±0.0006	0.98	0.009±0.0002	0.98	0.042±0.0006	0.98
	Blanched	0.016±0.0009	0.98	0.019±0.0012	0.98	0.046±0.0008	0.92
70°C	Non-blanched	0.018±0.0007	0.98	0.037±0.0036	0.98	0.053±0.0006	0.94
	Blanched	0.022±0.0011	0.98	0.040±0.0046	0.98	0.055±0.0010	0.96

Values expressed as the mean ± standard deviation.

Table 4 – Color parameters for garlic powder, blanched or otherwise

Temperature	*L**		*a**		*b**	
	I[1]	*II*[2]	*I*[1]	*II*[2]	*I*[1]	*II*[2]
Freeze-drying	94.97±0.13^{aA}	93.73±0.05^{aB}	-2.10±0.03^{cA}	-2.20±0.06^{cB}	11.85±0.11^{dA}	10.71±0.24^{bB}
Air-drying 50°C	93.58±0.35^{bA}	91.32±0.22^{bB}	-1.26±0.08^{bA}	-1.77±0.05^{bB}	14.04±0.47^{cA}	17.06±0.83^{aB}
Air-drying 60°C	92.31±0.25^{cA}	89.59±0.48^{cB}	-1.29±0.05^{bA}	-1.74±0.05^{bB}	15.39±0.51^{bA}	17.43±0.46^{aB}
Air-drying 70°C	91.28±0.35^{dA}	89.52±0.62^{cB}	-1.16±0.06^{aA}	-1.29±0.06^{aB}	17.37±0.19^{aA}	17.63±0.7^{aA}

[1] Non-blanched, [2] Blanched.
Values expressed as the mean ± standard deviation.
The same small letters in the same column indicate no significant difference (p>0.05).
The same capital letters in the same line for the same parameter indicate no significant difference (p>0.05).

Table 5 – Color parameters and browning index in blanched and non-blanched garlic powder

Temperature	*Chroma*		*Hue*		*IE*	
	***I*[1]**	***II*[2]**	***I*[1]**	***II*[2]**	***I*[1]**	***II*[2]**
Freeze-drying	12.04±0.11^{dA}	10.90±0.25^{bB}	100.08±0.08^{aA}	100.57±0.10$^{a\,B}$	11.23±0.12^{dA}	10.20±0.23^{cB}
Air-drying 50°C	14.09±0.47^{cA}	17.14±0.83^{aB}	95.12±0.33^{bA}	95.94±0.17^{bB}	14.74±0.61^{cA}	18.56±1.02^{bB}
Air-drying 60°C	15.45±0.51^{bA}	17.52±0.46^{aB}	94.78±0.22^{bA}	95.69±0.19^{cB}	16.63±0.67^{bA}	19.49±0.53^{abB}
Air-drying 70°C	17.41±0.19^{aA}	17.68±0.71^{aA}	93.80±0.15^{cA}	94.17±0.13^{dB}	19.48±0.17^{aA}	20.17±1.06^{aA}

[1] Non-blanched, [2] Blanched.
Values expressed as the mean ± standard deviation.
The same small letters in the same column indicate no significant difference (p>0.05).
The same capital letters in the same line for the same parameter indicate no significant difference (p>0.05).

Table 6 – Sugar concentrations (g/100g d.w.b.) of garlic with and without blanching

Temperature	*Inulin*		*Glucose*		*Fructose*	
	***I*[1]**	***II*[2]**	***I*[1]**	***II*[2]**	***I*[1]**	***II*[2]**
Fresh garlic	56.63±0.89^{a}	52.90±1.16^{a}	3.05±0.06^{d}	2.37±0.03^{d}	2.68±0.07^{c}	2.23±0.05^{d}
Freeze-drying	54.33±0.39^{b}	50.44±0.38^{b}	3.13±0.01^{c}	2.56±0.02^{c}	2.76±0.01^{b}	2.45±0.02^{c}
Air-drying 50°C	50.31±0.48^{c}	48.39±0.34^{c}	3.31±0.02^{b}	2.67±0.01^{b}	2.85±0.01^{a}	2.55±0.02^{b}
Air-drying 60°C	50.21±0.72^{c}	45.44±0.45^{d}	3.36±0.03^{ab}	2.92±0.01^{a}	2.89±0.04^{a}	2.62±0.02^{b}
Air-drying 70°C	48.15±0.32^{d}	41.68±0.05^{e}	3.41±0.01^{a}	2.94±0.02^{a}	2.94±0.01^{a}	2.80±0.01^{a}

[1] Non-blanched, [2] Blanched.
Values expressed as the mean ± standard deviation.
The same small letters in the same column indicate no significant difference (p>0.05).
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Table 7 – Particle size distribution (µm) in the garlic powder with and without pre-blanching

Temperature	Mean diameter		D[0,1]		D[0,5]		D[0,9]	
	***I*[1]**	***II*[2]**	***I*[1]**	***II*[2]**	***I*[1]**	***II*[2]**	***I*[1]**	***II*[2]**
Air-drying 50°C	201.61	302.04	21.43	23.05	143.67	255.64	486.88	646.17
Air-drying 60°C	119.28	201.81	13.40	17.93	84.87	186.13	277.17	401.56
Air-drying 70°C	183.23	220.82	18.11	16.13	134.29	159.54	421.86	542.59
Freeze-drying	79.35	104.79	12.38	12.02	55.60	82.31	191.41	237.10

[1] Non-blanched, [2] Blanched.

Table 8 – Glass transition temperatures of the garlic powders, with and without blanching

Temperature	a_w		T_g(°C)	
	***I*[1]**	***II*[2]**	***I*[1]**	***II*[2]**
Air-drying 50°C	0.521±0.004	0.518±0.001	99.02±1.98	99.39±1.98
Air-drying 60°C	0.451±0.004	0.428±0.003	99.65±1.99	99.82±1.99
Air-drying 70°C	0.381±0.005	0.376±0.003	109.35±2.28	109.85±2.19
Freeze-drying	0.126±0.001	0.136±0.001	120.15±3.80	112.53±3.12

[1] Non-blanched, [2] Blanched.

CAPÍTULO 4

DISCUSSÃO GERAL

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Para a execução e cumprimento dos objetivos gerais e específicos deste trabalho de dissertação, foram apresentados os resultados obtidos na forma de dois artigos científicos:

1) Estudo da inativação enzimática e das mudanças de cor do alho (*Allium sativum L.*) em diferentes condições de branqueamento.
2) Estudo da secagem do alho (*Allium sativum L.*) por ar quente e liofilização.

Inicialmente foi estudada a composição físico-química do alho in natura (Tabela 1). O teor de umidade do alho foi 64,15±0,09% e o pH 6,41±0,008 próximos aos encontrados por Haciseferogullari *et al*. (2005), de 66,32% e 6,05 respectivamente. A atividade de água (a_w) foi de 0,986±0,001 típica de produtos frescos que variam de 0,97 a 0,99 (Chirife *et al*., 1995). Em relação ao teor de sólidos solúveis, o alho in natura apresentou valores de 36,00±0,85°Brix, próximos aos relatados por Resende *et al*. (2003) de 35,75 a 37,25°Brix. Os teores de inulina, glicose e frutose foram de 56,62±0,89, 2,37±0,03 e 2,23±0,05g/100g b.s similares aos encontrados por Dalonso *et al*. (2009) de 51g/100g b.s e por Moreira *et al*. (2009) de 2,03g/100g b.s e 2,99g/100g b.s respectivamente.

Tabela 1 – Composição físico-química do alho in natura.

Umidade (%)	64,15±0,09
Atividade de água	0,986±0,001
Sólidos solúveis (°Brix)	36,00±0,85
pH	6,41±0,008
Inulina (g/100g b.s)	56,62±0,89
Glicose (g/100g b.s)	2,37±0,03
Frutose (g/100g b.s)	2,23±0,05

Observou-se no alho que depois de descascado, os bulbilhos sofrem escurecimento rápido devido à presença das enzimas peroxidase e polifenoloxidase (Mayer, 2006), assim como hidrólise parcial ou completa da inulina devido à presença da enzima inulinase (Schorr-Galindo e Guiraud, 1997). A inativação das enzimas utilizando calor é fundamental, devido à importância da preservação da cor da matéria prima antes de qualquer transformação (Ndiaye, 2009), assim como para a manutenção da integridade da inulina, a qual não é hidrolisada pelas enzimas digestivas na primeira porção do intestino e como conseqüência disto não aumenta a

glicemia e nem os níveis de insulina no sangue, sendo ideal para diabéticos (Leonel *et al*., 2006). Sharma *et al.* (2006) mencionam que a perda de atividade da inulinase é obtida em temperaturas superiores a 70°C.

No branqueamento em água a 80 e 90°C e em vapor a 100°C nos tempos de 1, 2, 4, 6, 8 e 10 minutos para a inativação destas enzimas, observou-se a diminuição da atividade enzimática da peroxidase, polifenoloxidase e inulinase com o aumento da temperatura. Com respeito ao tempo, a atividade dessas enzimas diminuiu rapidamente no primeiro minuto e depois continuou decrescendo, porém lentamente até permanecer constante (Figuras 1, 2 e 3 do Capítulo 2).

Para avaliar a cinética de inativação das enzimas peroxidase, polifenoloxidase e inulinase foi empregado o modelo bifásico de primeira ordem (Equação 3 do Capítulo 1) proposto por Ling e Lund (1978), que consiste na separação de dois grupos enzimáticos diferentes quanto a sua estabilidade térmica, sendo uma componente lábil e a outra resistente ao calor. Observou-se o aumento das constantes de velocidade da reação com a temperatura, tanto para o componente termo resistente, que variou de 0,030±0,004 à 0,068±0,008min^{-1}, quanto para o componente termo lábil que foi de 2,044±0,032 à 5,396±0,994min^{-1} (Tabela 2 do Capítulo 2). Observações similares foram feitas por Zhu *et al.* (2010) ao estudarem a cinética de inativação da enzima polifenoloxidase em pedaços de maça branqueadas nas temperaturas de 70 a 80°C.

Nos parâmetros de cor *L*, a* e b** estudados durante o branqueamento em água e em vapor, foi possível observar aumento do parâmetro *L** e diminuição dos parâmetros *a** e *b** com o aumento do tempo e da temperatura, resultando em amostras mais claras, esverdeadas e azuladas. Quanto ao índice de escurecimento (*IE*) ocorreu diminuição com o aumento do tempo e da temperatura de branqueamento e a maior diferença total de cor (ΔE) em relação à amostra in natura foi obtida no branqueamento em vapor a partir dos 8 minutos (Tabela 3 do Capítulo 2). Em estudos com purê de pimentão branqueados em água de 90 a 100°C, Ismail *et al*. (2006) também encontraram maior variação total da cor com o aumento da temperatura.

Para o estudo da cinética do índice de escurecimento foram empregados os modelos de ordem zero (Equação 1 do Capítulo 1) e de primeira ordem (Equação 2 do Capítulo 1), onde se observou que os dados não se ajustaram bem a esses modelos, obtendo-se coeficientes de correlação menores que 0,5 e 0,7 para os modelos de ordem zero e de primeira ordem respectivamente, razão pela qual se optou por empregar o modelo bifásico de primeira ordem

(Equação 3 do Capítulo 1) usado para o estudo da cinética de inativação enzimática. Constatou-se bom ajuste dos dados nesse modelo, com coeficientes de correlação superiores a 0,93 (Tabela 4 do Capítulo 2). Como resultado do uso desse modelo observou-se que as constantes de velocidade da reação aumentaram com a temperatura, tanto para o componente resistente e termo-lábil. Esse mesmo comportamento foi observado no estudo cinético realizado nas enzimas peroxidase, polifenoloxidase e inulinase (Tabela 2 do Capítulo 2).

A partir da avaliação dos resultados da inativação enzimática e perda de cor foi verificado que a melhor condição de branqueamento foi obtida no vapor por 4 minutos, sendo que o emprego de tempos maiores de branqueamento resultou em modificações na textura, devido à espessura fina das rodelas do alho, pois durante aquecimento prolongado as células podem se separar completamente resultando em grande perda da força textural (Kidmose *et al.*, 1999).

A Tabela 2 apresenta as atividades enzimáticas residuais da peroxidase, polifenoloxidase e inulinase, os teores de inulina, glicose e frutose e, os parâmetros de cor no alho branqueado em vapor a 100°C por 4 minutos. Nela se observa a diminuição da inulina e o aumento dos açúcares redutores (glicose e frutose) em relação à amostra in natura (Tabela 1), que pode ser devido à presença de atividade residual da inulinase que foi de 18,04%.

Tabela 2 – Atividades enzimáticas residuais, teores de açúcares e parâmetros de cor durante branqueamento em vapor a 100°C por 4 minutos.

Atividades enzimáticas residuais						
Peroxidase (%)		Polifenoloxidase (%)			Inulinase (%)	
6,47		7,85			18,04	
Teores de açúcares (b.s)						
Inulina (%)		Glicose (%)			Frutose (%)	
52,90±1,16		3,04±0,06			2,78±0,07	
Parâmetros de cor						
*L**	*a**	*b**	*Hue*	*Chroma*	*IE*	Δ*E*
66,59±0,12	-4,69±0,02	10,03±0,25	115,31±0,63	11,07±0,22	10,50±0,43	12,60±0,20

Para a enzima peroxidase a atividade residual foi de 6,47%. Conforme Bahçeci *et al.* (2005) para a obtenção de vegetais com ótima qualidade durante o armazenamento,

recomenda-se uma atividade residual máxima de 10% para a enzima peroxidase, após tratamento com branqueamento.

No Capítulo 3, se mostra o estudo realizado durante a secagem das rodelas de alho por ar forçado a 50, 60 e 70°C e por liofilização à pressão de 64µmHg, ambos sem e com branqueamento em vapor a 100°C por 4 minutos. A partir das curvas de secagem (Figuras 1 e 2 do Capítulo 3) observou-se que os valores de umidade de equilíbrio das amostras sem e com branqueamento, obtidas na porção assintótica da curva, diminuíram com o aumento da temperatura, porém essa diminuição não foi significativa ($p>0,05$) (Tabela 1 do Capítulo 3), sendo o mesmo observado por Krokida *et al.* (2003) ao desidratarem diferentes vegetais como tomate, alho, cebola, pimenta vermelha e abóbora. Para os valores de atividades de água de equilíbrio observou-se que diminuíram significativamente ($p>0,05$) com o aumento da temperatura (Tabela 1 do Capítulo 3), sendo o mesmo relatado por Lertworasirikul *et al.* (2008) ao desidratarem mandioca.

Para o estudo da cinética de secagem em ar forçado, tanto para a umidade e atividade de água em função do tempo de secagem, empregaram-se os modelos propostos por Henderson-Pabis, Page e Newton (Equações 4, 5 e 6 do Capítulo 1). No caso da umidade, expressa de forma adimensional, os valores de R^2 para os três modelos foram superiores a 0,98, indicando bom ajuste deles, no entanto, o modelo de Henderson-Pabis apresentou maiores coeficientes de correlação (R^2=0,99). Neste modelo as constantes de taxa de secagem aumentaram com a temperatura e com o emprego do branqueamento (Tabela 2 do Capítulo 3) variando de 0,053±0,0006 à 0,112±0,0048min^{-1}. Pezzutti *et al*. (1997) ao estudarem a cinética de secagem do alho desidratado nas temperaturas de 45 a 75°C, também observaram o aumento dessas constantes de secagem com o aumento da temperatura, usando o modelo proposto por Henderson-Pabis.

Da mesma forma, ao empregar os modelos anteriores para avaliar o comportamento da a_w adimensional durante a secagem, observou-se que os modelos que melhor predisseram os valores experimentais foram os modelos de Henderson-Pabis e Page (R^2=0,98) indicando um bom ajuste. Em ambos os modelos também se observaram o aumento das constantes da taxa de secagem com a temperatura e com o uso do branqueamento, variando de 0,013±0,0008 à 0,040±0,0046min^{-1} (Tabela 3 do Capítulo 3).

Nas amostras em pó desidratadas por ar quente e liofilização, quando estudados os parâmetros de cor L^*, a^* e b^* observou-se que o parâmetro L^* foi significativamente maior,

enquanto que os parâmetros *a** e *b** foram significativamente menores no alho em pó desidratado por liofilização quando comparado à desidratação em ar forçado (Tabela 4 do Capítulo 3), obtendo-se amostras mais claras, esverdeadas e azuladas. Li *et al*. (2007) ao estudarem a desidratação de fatias de alho, também relataram valores de *L** ligeiramente maiores na desidratação por liofilização em comparação a desidratação à vácuo na temperatura de 40°C.

Na desidratação em ar forçado observou-se diminuição do parâmetro *L** e aumento dos parâmetros *a** e *b** com o aumento da temperatura, tornando as amostras mais escuras, avermelhadas e amareladas (Tabela 4 do Capítulo 3). Essa observação também foi reportada por Sacilik *et al.* (2005) ao desidratarem rodelas de alho, com tamanhos na faixa de 10 a 12mm de diâmetro e de 3 a 5mm de espessura, em secador de ar forçado nas temperaturas de 40 a 60°C.

Quanto ao índice de escurecimento, observou-se que as amostras liofilizadas sem ou com branqueamento foram significativamente menores ($p<0,05$) em comparação as amostras secas em ar forçado (Tabela 5 do Capítulo 3). O mesmo foi relatado por Topuz *et al.* (2009) ao desidratarem pimentão com 1cm de espessura por liofilização e ar forçado a 60°C. Segundo Sacilik *et al.* (2005) em termos de propriedades de cor desejadas para fatias de alho, os menores valores de *a** e os maiores valores de *L** seriam os ideais, devido a sua proximidade com a cor do alho in natura. No nosso trabalho o menor valor de *a** e os maiores valores de *L** foram encontrados no alho liofilizado sem branqueamento (Tabela 4 do Capítulo 3).

Quanto ao efeito da temperatura e do branqueamento sobre os teores de açúcares após a secagem do alho, observou-se a diminuição da inulina e aumento de glicose e frutose tanto nas amostras sem branqueamento quanto nas amostras com branqueamento em comparação à amostra in natura (Tabela 6 do Capítulo 3). Graefe *et al*. (2004) também reportaram a hidrólise da inulina em açúcares redutores para raízes de yacon com o aumento da temperatura de armazenamento e por Ishiguro *et al*. (2010) ao analisarem o armazenamento de raízes de bardana nas temperaturas de 0 a 20°C. O aumento da concentração de açúcares redutores encontrado no nosso trabalho pode estar relacionado com a atividade residual da enzima inulinase no alho, que foi de 18,04% após branqueamento em vapor por 4 minutos (Tabela 2).

Também observaram-se maiores teores de inulina nas amostras liofilizadas em comparação às amostras desidratadas em ar quente. Isso é devido à menor temperatura de secagem empregada durante a liofilização (Tabela 6 do Capítulo 3).

Ao comparar as amostras desidratadas sem e com branqueamento observaram-se menores concentração de inulina, glicose e frutose nas amostras branqueadas (Tabela 6 do Capítulo 3) devido à possível lixiviação dos açúcares. Bomben *et al*. (1973) encontraram perdas de produto no branqueamento a vapor decorrente da lixiviação dos sólidos pela evaporação e condensação da água.

Quanto aos diâmetros médios das partículas de alho em pó sem e com branqueamento observou-se que as amostras liofilizadas foram menores em comparação às desidratas em ar forçado (Tabela 7 do Capítulo 3), possivelmente pela formação de partículas mais porosas, obtidas pela sublimação da água durante a liofilização (Krokida *et al*., 1998). Observou-se também que os diâmetros das partículas são maiores em todas as amostras pré-tratadas com branqueamento, sugerindo agrupamento de células, conforme relatado por Chantaro *et al.* (2008) ao estudarem cascas de cenoura em pó sem e com branqueamento.

A Figura 1 apresenta os histogramas de freqüência e distribuição do tamanho de partículas das amostras de alho em pó desidratadas em ar a 50°C sem e com branqueamento, onde se confirma o maior tamanho de partículas na amostra branqueada, por apresentar maior pico em torno de 600µm enquanto que na amostra sem branqueamento o maior pico foi próximo a 400µm.

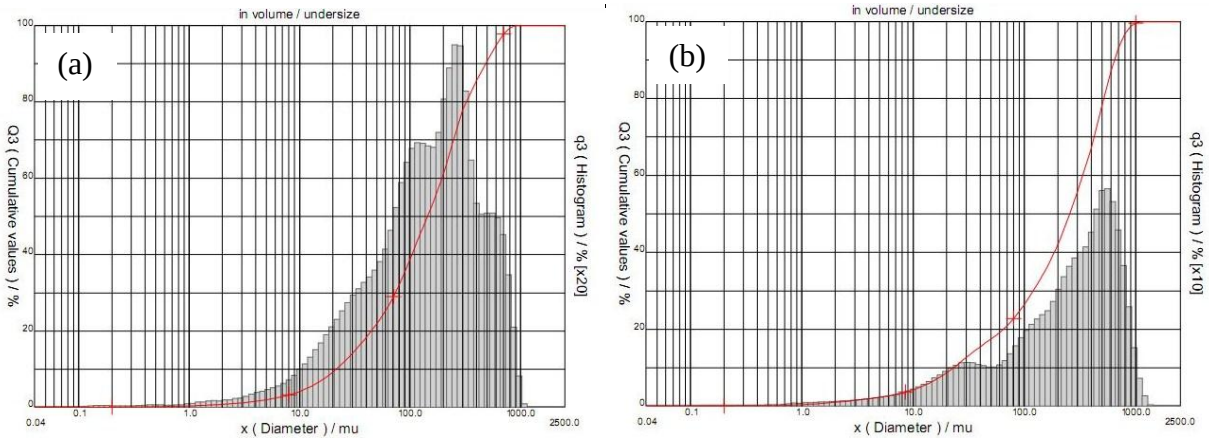

Figura 1 – Histograma de freqüência e distribuição do tamanho de partículas do alho em pó desidratadas em ar a 50°C (a) sem branqueamento e (b) com branqueamento.

Ao utilizar a microscopia eletrônica de varredura, observaram-se menores danos na superfície das amostras sem e com branqueamento liofilizadas quando comparadas com as amostras desidratadas em ar forçado (Figura 6 do Capítulo 3). Isto pode ser verificado na Figura 2 a qual apresenta as imagens microscópicas do alho em pó liofilizado e desidratado em ar forçado à 60°C ambos sem branqueamento. Conforme James (2009) a liofilização origina menor contração e conseqüentemente menor rugosidade nas amostras do que na secagem em ar, porém podem-se produzir danos estruturais devido à formação de cristais de gelo durante o congelamento prévio à liofilização.

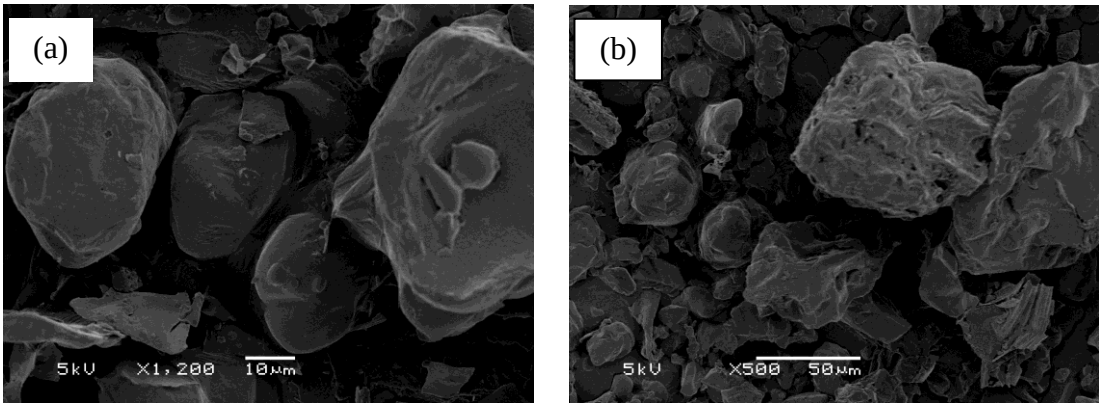

Figura 2 – Imagens microscópicas do alho em pó sem branqueamento: liofilizado com aumento de 1.200x (a) e desidratado em ar forçado à 60°C com aumento de 500x (b).

A Figura 3 mostra os termogramas (fluxo de energia em função da temperatura) do alho em pó liofilizado sem e com branqueamento, obtidos através de análise em DSC. Nela se observa uma transição típica de segunda ordem que produz uma mudança no fluxo de calor devido as mudanças na capacidade de calor a uma temperatura de transição de fase. A temperatura de transição vítrea foi tomada como o ponto médio da transição vítrea (Tonon *et al*., 2009).

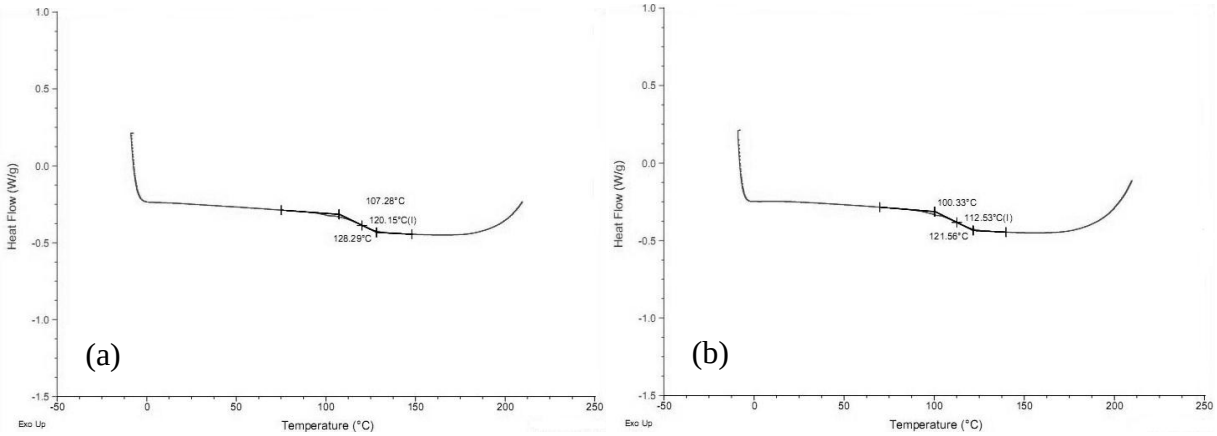

Figura 3. Termogramas de transição vítrea do alho em pó liofilizado sem branqueamento (a) e com branqueamento (b).

As temperaturas de transição vítrea (T_g) do alho desidratado em pó variou de 99,02±1,98 à 120,15±3,80°C, para atividades de água na faixa de 0,126±0,001 à 0,521±0,004, observando-se que não foram afetadas pelo branqueamento. No entanto, houve aumento da T_g com a diminuição da atividade de água (Tabela 8 do Capítulo 3), confirmando o efeito plasticizante da água. O efeito da plasticização na diminuiçao da T_g foi demostrado por Telis e Sobral (1999) ao estudarem a T_g do abacaxi liofilizado obtendo-se valores de -58,2, -36,5 e 46°C para a_w de 0,90, 0,65 e 0,11 respectivamente.

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