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Drying Characteristics of Apple Slabs after Pretreatment with Supercritical CO_2

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Abstract

Supercritical CO_2 pretreatment before dehydration leads to a faster dehydration rate. The best supercritical CO_2 pretreatment conditions for the most effective dehydration were 45°C, 25 MPa and 55°C, 25 MPa. Increasing pressure of the supercritical CO_2 pretreatment system tended to accelerate the dehydration rate more than increasing temperature did. Samples pretreated at higher temperatures and pressures showed greater shrinking and pore distribution on scanning electron microscopy. Control samples maintained their cell walls, whereas samples pretreated at higher temperatures and pressures showed more cell disruption, and more pores were observed. Pore sizes of control and pretreated samples were about 100 and 70~80 μm, respectively. Samples pretreated at higher temperatures and pressures had smaller pores and a denser distribution.

Key words: dehydration, pretreatment, rehydration, supercritical carbon dioxide

INTRODUCTION

Today's consumers demand safer and healthier processed foods, as well as products with natural qualities. Therefore, food processing techniques have been developed to meet these demands, and cost-, energy-, and time-effective procedures have been implemented. One of the most popular food processing methods for keeping food safe is dehydration. Drying is a process that partially or totally removes the water content from foodstuff. Depending on the way the drying is practiced, a number of mass transport mechanism(s) take place to achieve this. Evaporation and sublimation are the main mechanisms in air drying and freeze drying, respectively. Drying is a unit operation with a phase transition and mass transfer of water. Dehydration protects food from decomposition by microorganisms, and it reduces product mass and volume, resulting in cost-effective packaging and delivery (1).

Very often, vegetable dehydration is accomplished with hot air drying, which, in comparison to other drying methods (e.g. freeze drying), is less expensive per kg of dried material produced. But hot air drying can result in shrinkage, surface hardening, and low recovery after rehydration. Additionally, color, texture, taste, and nutrition may change (2). To minimize these changes, several pretreatments for dehydration have been suggested: blanching, steaming (3), microwaving (4), or dipping the food into chemicals such as organic acids (ascorbic or citric acids), antioxidants, alkali solution or ethyl oleate (5). The blanching method, which inactivates enzymes, often results in pronounced leaching of water soluble nutrients, such as minerals and vitamins, due to the thermally-induced loss of cell membrane functionality that regulates the permeability of those compounds. In certain cases, prolonged blanching can also disrupt cell walls, resulting in vegetable tissue undergoing a very pronounced loss of texture upon rehydration (6-10). Due to the disadvantages of blanching (heat-induced changes), chemical treatment (chemical residues), and osmotic dehydration (taste changes), the food products do not maintain their natural flavors and textures.

Studies about supercritical fluids have been widely conducted in many areas. In particular, supercritical carbon dioxide (CO_2) has been studied for the extraction and separation of mixtures. Using supercritical CO_2 pretreatment could be an ideal method of preparing foods prior to dehydration because supercritical CO_2 possess both gas-like and liquid-like properties, which means superior mass transfer properties with a higher diffusion coefficient and lower viscosity than liquid solvents. Also, the absence of surface tension allows the rapid penetration into the pores of matrices of foodstuffs. The pretreatment of foods with supercritical CO_2 could potentially reduce energy costs and save time by enhancing drying velocity, as well as maintain food qualities through prompt enzyme inactivation and low heat-induced change, plus eliminate toxic residues (11,12).

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Although the potential for using supercritical CO_2 as a pretreatment for food dehydration exists, limited information on this process is available. In this study, the drying characteristics of an apple slab (drying velocity, water uptake by re-hydration and surface observation by scanning electron microscopy) were investigated to determine whether supercritical CO_2 can be applied as a pretreatment method.

MATERIALS AND METHODS

System and preparation for experiments

Apples (*Malus domestica* Borkh) were purchased September at local market and stored at 4°C. The apples were peeled, the core was removed, and the fruit was sliced into $3 \times 3 \times 0.5$ cm pieces. The supercritical fluid pretreatment system was self-manufactured (Fig. 1). System temperature and pressure were set by controlling the electro-currency of the heating jacket and the back-pressure regulator valve of the extraction vessel, respectively. Sliced apples (50 g) were put into the extraction vessel, and three different temperatures (35, 45, and 55°C) and pressures (15, 20, and 25 MPa) were used to test the effects of both factors on the drying characteristics of an apple slab. The system took 10 min to reach the desired pressure and an additional 10 min for the pressure to be reduced to atmosphere pressure by vent. Every sample was pretreated for 20 min in each of the supercritical states. Three times were run for replication at each treatment.

Determination of drying temperature and pretreatment time

Before entering into the actual experiments, it was necessary to determine which temperatures were proper for drying and how long the apple samples needed to be pre-treated. First, the weight changes of the apple slab were investigated every 30 min for 4 hours at 55, 70, and 85°C. To determine the proper pretreatment time, the mildest condition among the experimental designs was selected (35°C and 15 MPa), and apples were treated for 10, 30, and 60 min. A convection oven (EDO-L, Gae-Rim Instrument LTD, Tokyo, Japan) was used for drying.

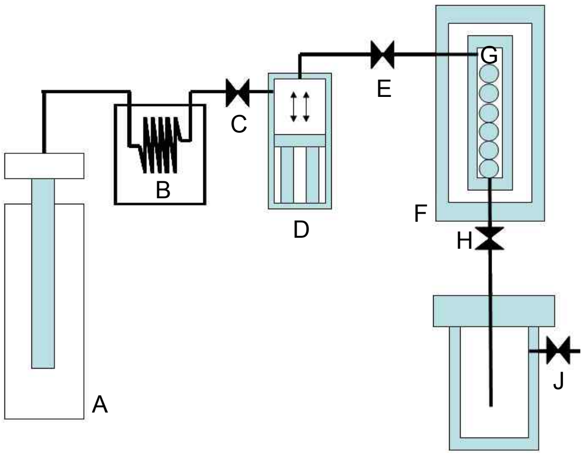


Fig. 1. Schematic flow diagram of supercritical fluid system for the drying pretreatment. A, liquid carbon flow dioxide cylinder; B, cooling circulator; C, E, H, and J, stop valve; D, CO_2 pump with cooling jacket; F, heating jacket; G, extraction vessel; I, reservoir.

Weight changes in the apple slab during drying

After identifying the best drying condition, apple slabs were pretreated at the supercritical CO_2 conditions of 35, 45, 55°C and 15, 20, 25 MPa and then dried for 6 hours at 70°C. Samples were taken every 30 min for 6 hours to analyze weight changes. The weights were measured three times and averaged. The weight losses during drying were considered to be the result of decreasing water content.

Moisture gain during rehydration

Dried apple samples were rehydrated in a water bath at 40°C, and their weights were measured every 20 minutes for 140 minutes.

Surface observation of the dried apple

The sample surface was observed using scanning electron microscopy (SEM, S-4300, Hitachi, Tokyo, Japan) after gold ion coating.

RESULTS AND DISCUSSION

Determination of drying temperature and pretreatment time

The results showed that the fastest dehydration occurred at 85°C, followed by 70°C and 55°C; water content reached an equilibrium moisture content after 120 minutes (Fig. 2). Due to the caramelization of sugar, the samples changed color to brown and smelled burnt at 85°C. The dehydration rate at 55°C was slowest, with no difference in color and texture compared to the 70°C

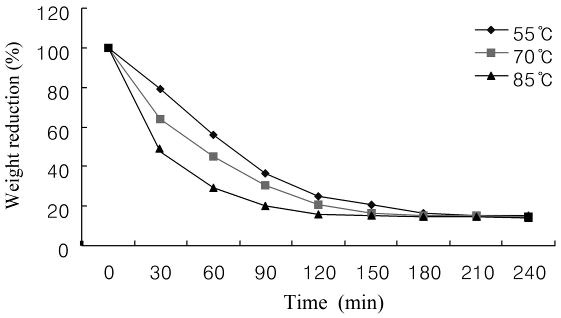


Fig. 2. Weight reduction of the apple slab at different drying temperatures.

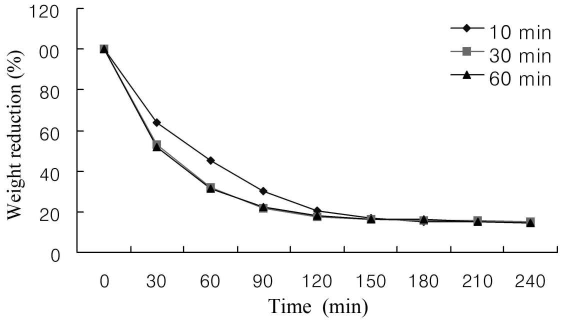


Fig. 3. Weight reduction of the apple slabs with supercritical CO_2 pretreatment time at 35°C and 15 MPa.

treatment; therefore, 70°C was chosen for the drying temperature after supercritical CO_2 pretreatment. Enough drying time (210 min) was provided for the samples to reach equilibrium moisture content. Fig. 3 shows the changes in sample weight for those samples treated with the mildest experimental condition (35°C, 15 MPa) while drying at 70°C. The sample weight decreased more abruptly at 30 and 60 min than at 10 min of treatment. No difference in appearance was detected with the naked eye between samples treated for 30 or for 60 min, so a pretreatment time of 30 min was chosen to save experimental time.

Weight changes in the apple slab during drying

Water content of the dry apple samples over time is shown in Fig. 4. Control samples (water content: 84.3%) that received no supercritical CO_2 pretreatment rapidly dehydrated to 30% within 60 min, and subsequently showed a slow dehydration rate. Water content decreased to a low of 4.2% after 210 min. Samples pretreated at 35°C and 15 MPa dehydrated to 17.5% with a constant water reduction rate during 60 min. The final water content was 3.6% after 120 min. Samples treated with 35°C and 20 MPa (water content, 13.8% after 60 min; final water content, 3.1%) or 25 MPa (water content, 6.5% after 30 min; final water content, 2.5%), showed a trend similar to that for samples pretreated at 15 MPa.

At 45°C and 15, 20, or 25 MPa, water content after 60 min was 18.9, 18.0, and 9.6%, respectively, and the final water content was 3.6, 2.4, and 1.5%, respectively. At 55°C and 15, 20, or 25 MPa, water content after 60 min was 17.1, 16, and 10.9%, respectively, and the final water content was 2.4, 2.3, and 1.5%, respectively.

Supercritical CO_2 pretreatment before dehydration led to a faster dehydration rate. The best supercritical CO_2 pretreatment conditions for the most effective dehydration were at 45°C, 25 MPa and 55°C, 25 MPa. Increasing the pressure of the supercritical CO_2 pretreatment system appeared to accelerate the dehydration rate more than increasing the temperature.

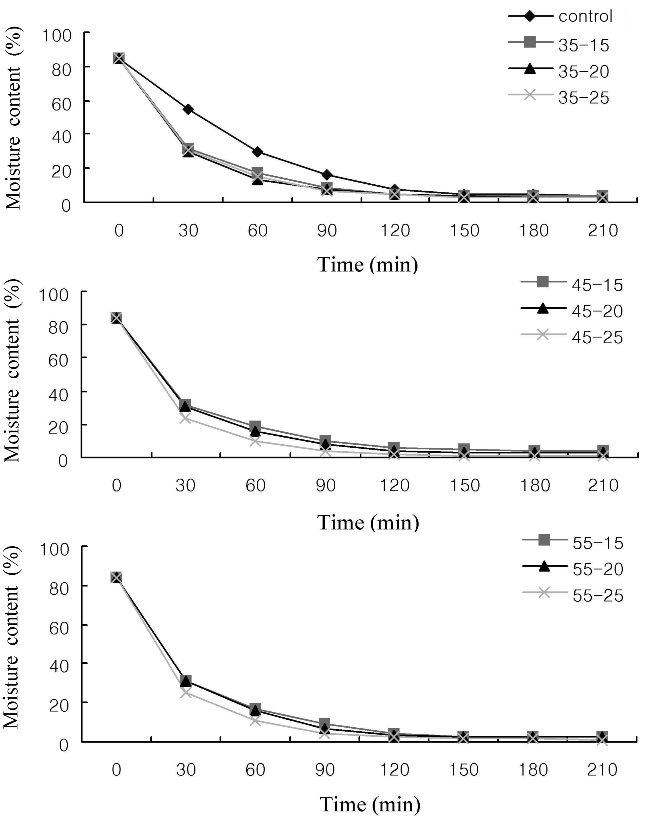


Fig. 4. Moisture content changes in apple slabs after pretreatment at each of the supercritical CO_2 conditions. Control, untreated; 35-15, 35°C, 15 MPa; 35-20, 35°C, 20 MPa; 35-25, 35°C, 25 MPa; 45-15, 45°C, 15 MPa; 45-20, 45°C, 20 MPa; 45-25, 45°C, 25 MPa; 55-15, 55°C, 15 MPa; 55-20, 55°C, 20 MPa; 55-25: 55°C, 25 MPa.

These observations are the result of the low viscosity and high diffusion rate of the supercritical fluid and the near-absence of surface tension. These properties allow the supercritical fluid to easily penetrate into the micropores and cells of the apple slices. Furthermore, new pores or channels could have been made by the supercritical fluid. Thus, newly made channels may increase the surface area that contacts the hot air during the dehydration process, resulting in faster dehydration. The dehydration rate depends on exposed surface area, the difference in moisture content between air and target foods, and the air flow rate (13,14). After the fast dehydration within the 60-min constant-dehydration rate period, the dehydration rate might be controlled by capillary movement and molecular diffusion, and supercritical CO_2 pretreatment may affect these phenomena, resulting in faster dehydration and less water content than the control. Quenzer & Burns (15) also reported that cell disruption by pretreatment increases water movement.

Moisture gain during rehydration

Moisture gains during rehydration are shown in Fig. 5. Control samples absorbed 2.45 g/g water during 40

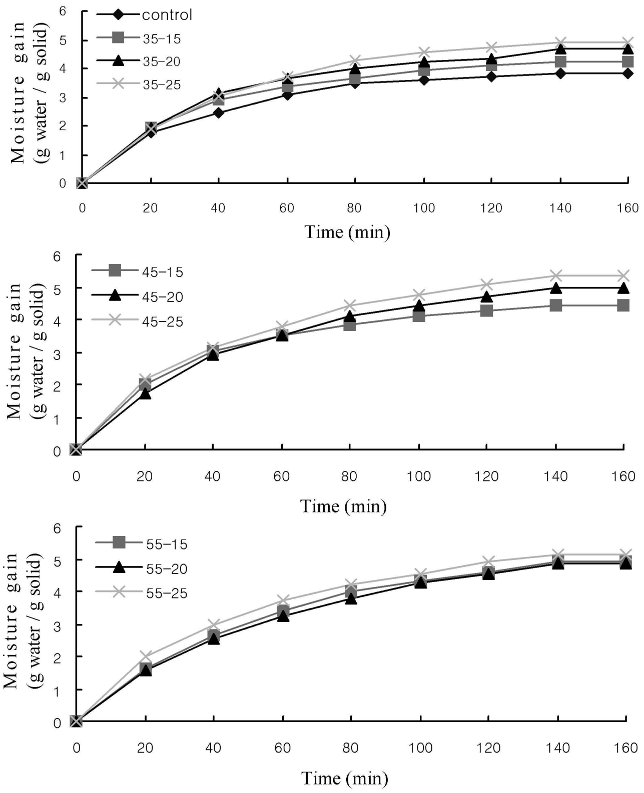


Fig. 5. Moisture gains in apple slabs after pretreatments and drying. Samples: See Fig. 4.

min, followed by slow rehydration. After 120 min, no further water absorption occurred, and 3.82 g/g water was absorbed. Samples pretreated with supercritical CO_2 at 35°C and 15 MPa absorbed 2.91 g/g water during 40 min, but water absorption tended to slow, resulting in 4.24 g/g of water being absorbed by the end of the experiment. Samples pretreated at 35°C and 20 and 25 MPa absorbed 3.15 and 3.04 g/g water, respectively, during the first 40 min, but a big increase in water absorption after 60 min was not observed (final absorption, 4.65 and 4.93 g/g for 20 and 25 MPa, respectively). Samples pretreated at 45°C showed a similar trend (water absorption 3.01, 2.92, and 3.10 g/g during the first 40 min; 4.43, 4.93, and 5.33 g after 240 min for 15, 20 and 25 MPa, respectively).

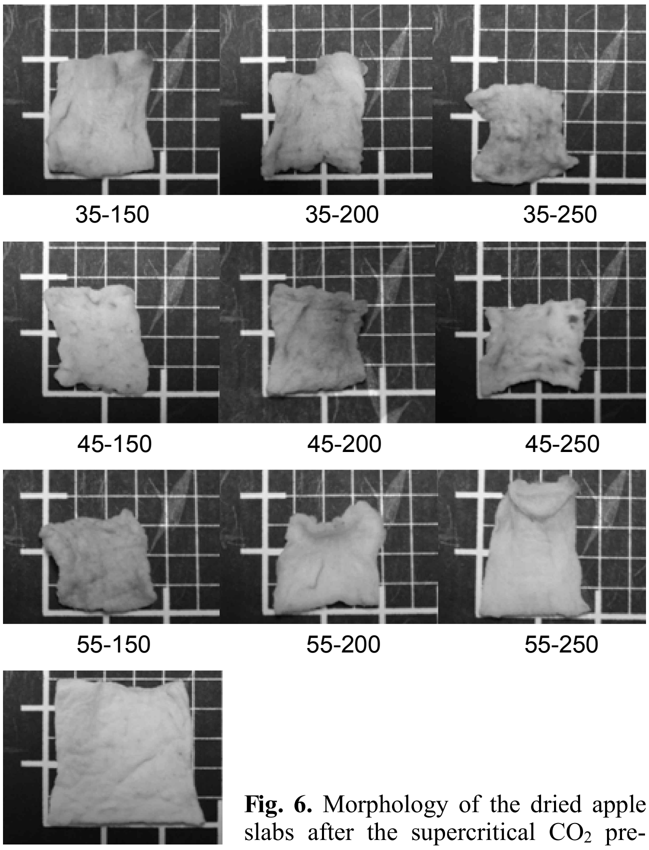
During the first 40 min, water absorption by the samples pretreated at 55°C was 2.66, 2.53, and 2.97 g/g for 15, 20, and 25 MPa, respectively. The final water absorptions were 4.90, 4.88, and 5.13 g/g for 15, 20, and 25 MPa, respectively. After 60 min, a difference in water absorption was observed between samples pretreated at different temperatures. Water absorption rates were similar between 15 and 20 MPa; however, samples under the 25 MPa condition showed the fastest water absorption as soon as rehydration was initiated. Samples pretreated at 45°C and 25 MPa showed the greatest rehydration rate, followed by those at 55°C and 25 MPa, 45°C and 20 MPa, and the control.

Thus, the cell disruption in the apple samples caused by supercritical pressure resulted in an increase in water absorption. The faster rehydration rate in samples pretreated at 55°C than those pretreated at 45°C may have been caused by heat damage to the cells, resulting in a decrease in the rehydration rate. Therefore, additional study concerning cell damage may be needed.

Surface observation

Fresh apples contain relatively high moisture and have a soft texture (16), but surface shrinking occurs easily when they are dried with hot air (17). The shrinking shapes are shown in Fig. 6. Pretreated samples showed more deformation than did controls, but no cracks were observed.

Kilpartrick et al. (18) reported that shrinking volume is close to the volume of water lost. A study of hot-air dehydration of radishes and sweet potatoes by Cho et al. (19) showed that shrinkage depended on sample thickness, air temperature, moisture, and flow rate. The greater deformation and shrinkage found in pretreated samples may have been caused by cell disruption that resulted in an increase in the dehydration rate and capillary movement compared to controls.


35-150 35-200 35-250

45-150 45-200 45-250

55-150 55-200 55-250

control

Fig. 6. Morphology of the dried apple slabs after the supercritical CO_2 pretreatments. Samples: See Fig. 4.

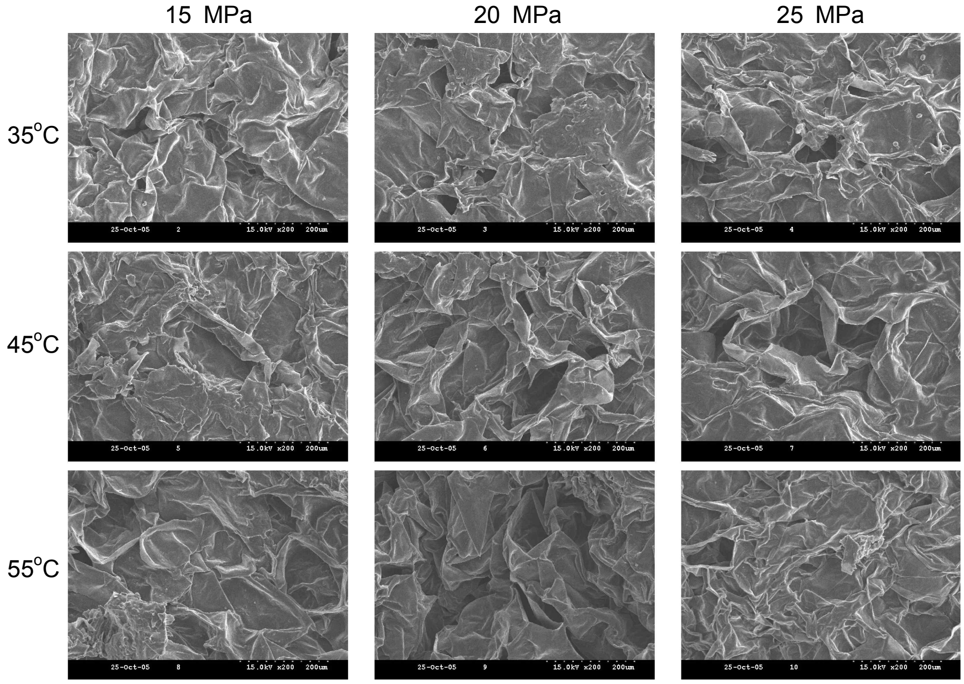


Fig. 7. Scanning electron micrographs of dried apple after supercritical CO_2 pretreatments.

The surfaces of the pretreated dry apple samples as visualized by SEM are shown in Fig. 7. More shrinking occurred in the pretreated samples, and they had a greater degree of pore distribution than did controls. Samples pretreated at higher temperatures and pressures showed greater shrinking and pore distribution. Control samples maintained their cell walls, whereas pretreated samples at higher temperatures and pressures showed more cell disruption and more pores. The pore sizes of control and pretreated samples were about 100 and 70～80 μm, respectively. Samples pretreated at higher temperatures and pressures had smaller pores and a denser distribution. Therefore, the pretreated samples might have had more cell disruption caused by the loss of cell moisture. Saurel et al. (20) showed a similar result in that osmotic treatment leads to cell wall shrinking by dehydration.

REFERENCES

1. Derossi A, Pilli DT, Severini C, McCarthy MJ. 2008. Mass transfer during osmotic dehydration of apples. *J Food Eng* 86: 519-528.
2. Raltti C. 2001. Hot air and freeze-drying of high-value foods: a review. *J Food Eng* 49: 311-319.
3. Moreno J, Chiralt A, Escriche I, Serra JA. 2000. Effect of blanching/osmotic dehydration combined methods on quality and stability of minimally processed strawberries. *Food Res Int* 33: 609-616.
4. Ramaswamy H, Van de Voort FR. 1990. Microwave application in food processing. *Can Inst Food Sci Technol J* 23: 17-23.
5. Jazini MH, Hatamipour MS. 2010. A new physical pretreatment of plum for drying. *Food Bioprod Process* 88: 133-137.
6. Castro SM, Saraiva JA, Lopes-da-Silva JA, Delagadillo I. 2008. Effect of thermal blanching and high pressure treatments on sweet green and red bell pepper fruits. *Food Chem* 107: 1436-1449.
7. Aguilera JM, Chiralt A, Fito P. 2003. Food dehydration and product structure. *Trends Food Sci Technol* 14: 432-437.
8. Contreras C, Martin-Esparza ME, Chiralt A, Martínez-Navarrete N. 2008. Influence of microwave application on convective drying: Effects on drying kinetics and optical and mechanical properties of apple and strawberry. *J Food Eng* 88: 55-64.
9. Kulkarni SG, Vijayanand P, Aksha, M, Reena P, Ramana KVR. 2008. Effect of dehydration on the quality and storage stability of immature dates (*Pheonix dactylifera*). *LWT-Food Sci Technol* 41: 278-283.
10. Tedjo W, Taiwo KA, Eshtiaghi MN, Knorr D. 2002. Comparison of pretreatment methods on water and solid diffusion kinetics of osmotically dehydrated mangos. *J Food Eng* 53: 133-142.
11. Hu Q, Pan B, Xu J, Sheng J, Shi Y. 2007. Effects of supercritical carbon dioxide extraction conditions on yield and antioxidant activity of *Chlorella pyrenoidosa* extracts. *J Food Eng* 80: 997-1001.
12. Mitra P, Ramaswamy HS, Chang KS. 2009. Pumpkin (*Cucurbita maxima*) seed oil extraction using supercritical carbon dioxide and physicochemical properties of the oil. *J Food Eng* 95: 208-213.
13. Severini C, Baiano A, Pilli TD, Carbone BF, Derossi CA. 2005. Combined treatments of blanching and dehydration: study on potato cubes. *J Food Eng* 68: 289-296.
14. Gagan DS, Rajiv S, Bawa AS, Sanxena DC. 2008. Drying and rehydration characteristics of water chestnut (*Trapa natans*) as a function of drying air temperature. *J Food Eng* 87: 213-221.
15. Quenzer NM, Burns EE. 1981. Effects of microwave, steam and water blanching on freeze-dried spinach. *J Food Sci* 46: 410-415.

16. Sacilik FK, Elicin AK. 2006. The thin-layer drying characteristics of organic apple slices. *J Food Eng* 73: 281-289.
17. Mayor L, Sereno AM. 2004. Modeling shrinkage during convective drying of food materials: a review. *J Food Eng* 61: 373-386.
18. Kilpartrick PW, Lowe E, Van A. 1955. Tunnel dehydrators for fruits and vegetables. *Adv Food Res* 6: 359-375.
19. Cho DJ, Hur JW, Kim HY. 1989. Influencing factors in drying and shrinking characteristics of root vegetables. *Korean J Food Sci Technol* 21: 203-211.
20. Saurel R, Raoult-Wack AL, Rios G, Guilbert S. 1994. Mass transfer phenomena during osmotic dehydration of apple. I. Fresh plant tissue. *Inter J Food Sci Technol* 29: 531-542.

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