

MICROWAVE - OSMOTIC DEHYDRATION OF APPLES (*RED GALA*) UNDER CONTINUOUS FLOW MEDIUM SPRAY CONDITIONS (MWODS) FOR IMPROVING MOISTURE TRANSPORT RATE AND PRODUCT QUALITY

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SUGGESTED SHORT TITLE

**MICROWAVE - OSMOTIC DEHYDRATION UNDER
CONTINUOUS FLOW MEDIUM SPRAY CONDITIONS (MWODS)**

This thesis is dedicated to my beloved husband (Bahram)

ABSTRACT

Microwave osmotic dehydration (MWOD) is a novel technique with a good potential for more efficient osmotic drying of fruits and vegetables. It combines the microwave heating with osmotic dehydration for enhancing moisture transfer rate in the osmotic dehydration process and product quality. Previous studies have found the MWOD process under continuous flow medium immersion heating conditions to significantly improve moisture loss rate and product quality, and to lower the solids gain. This research focuses on a system combining microwave and osmotic dehydration under continuous flow medium spray (MWODS) conditions.

Preliminary studies were carried out to compare the osmotic dehydration kinetics of apple (*Red Gala*) cylinders in microwave osmotic dehydration under continuous flow medium spray conditions (MWODS) with microwave osmotic dehydration under continuous flow medium immersion conditions (MWODI), as well as conventional osmotic drying (COD) in immersion (CODI) and spray (CODS) modes. Eight different test conditions (two temperatures (40°C-50°C), solute concentration (40°B-50°B); and four treatment times (30, 60, 90 and 120 min) with three replicates were employed with each method. The process was monitored for moisture loss, weight reduction and solids gain. The results showed that the MWODS process considerably enhanced the moisture transfer rate from the fruit, and limited the solids gain at the same time. In the second part, the two-parameter Azuara model and the conventional diffusion model were evaluated for describing the mass transfer kinetics of apple (*Red Gala*) cylinders during MWODS, MWODI, CODS and CODI. The results showed that both models adequately described the transient mass transfer kinetics during the OD process; however the Azuara model was superior.

MWODS was further studied to evaluate the effect of various process variables (sucrose concentration, medium temperature, flow rate and contact time) by using response surface methodology and a central composite rotatable design. Predictive models were developed relate the response variables to process parameters. Finally optimization studies were carried out to elucidate optimal processing conditions under

MWODS. The study demonstrated that moisture loss (ML), solids gain (SG) and weight reduction (WR) were predictably higher at higher sucrose concentrations, higher medium temperatures, longer contact times and higher flow rates. With ML and WR, all process variables except flow rate had interactions, while with SG, only the contact time – flow rate interactions were significant. A set of optimum conditions were established to provide higher moisture transfer and weight reduction rates with moderate levels of solids gain.

Since OD only results in partial dehydration, a second stage drying was evaluated employing conventional air drying and compared with freeze drying to identify cost effective systems for preserving the quality of the osmotically dehydrated shelf-stable fruits. The effect of MWODS pretreatment on air-drying kinetics and quality parameters (color, texture, and rehydration characteristics) of apple (*Red Gala*) cylinders was evaluated. The results revealed that drying time decreased with increasing concentrations and medium temperature of the MWODS treatment. Compared with untreated control samples, MWODS air-dried samples had higher coefficient of moisture diffusivity (D_m). In terms of quality parameters, the MWODS air-drying combination process resulted in a product with lower color change and a more chewy structure. The air dried product without MWODS had the least desirable quality characteristics. While the color was better preserved in the freeze dried product, it was much more brittle than MWODS – air-dried product. The rehydration capacity of MWODS air-dried products was lower than freeze-dried products and higher than air-dried.

Overall, the thesis research contributes to a better understanding of the moisture transfer behavior during microwave osmotic dehydration under continuous flow medium spray processing conditions. Together with a simple second stage air-drying it can produce high quality dehydrated apple products.

RÉSUMÉ

La déshydratation osmotique à l'aide des micro-ondes (MWOD) est une nouvelle technique avec un bon potentiel pour plus d'efficacité de séchage osmotique des fruits et légumes. Il combine le chauffage micro-onde avec la déshydratation osmotique pour améliorer le taux de transfert de l'humidité dans le procédé de déshydratation osmotique et la qualité du produit. Des études antérieures ont trouvé que le processus MWOD sous flux continu d'immersion avec de chauffage améliore sensiblement le taux de perte d'humidité et la qualité des produits et abaisse le gain des solides. Cette recherche porte sur un système combinant micro-ondes et déshydratation osmotique sous conditions de flux continu de pulvérisation (MWODS).

Des études préliminaires ont été effectuées pour comparer la cinétique de déshydratation osmotique des cylindres de pommes (Rouge Gala) à déshydratation osmotique sous conditions de flux continu de pulvérisation (MWODS) avec déshydratation osmotique à l'aide des micro-ondes sous flux continu d'immersion (MWODI), ainsi que séchage osmotique le conventionnel (COD) en mode d'immersion (CODI) et en mode de pulvérisation (CODS). Huit conditions d'essai différentes (deux températures - combinaisons de concentrations du soluté, 50°C - 50°Brix et 40°C - 40°B et quatre temps de traitement 30, 60, 90 et 120 min) avec trois répétitions ont été utilisées à chaque méthode. Le processus a été suivi pour la perte d'humidité, la perte de poids et le gain des solides. Les résultats ont montré que le processus de MWODS a considérablement amélioré le taux de transfert d'humidité à partir du fruit et a limité le gain des solides en même temps. Dans la deuxième partie, le modèle d'Azuara à deux paramètres et le modèle de diffusion classique ont été évalués pour décrire la cinétique de transfert de masse des cylindres de la pomme (*Rouge Gala*) pendant MWODS, MWODI, COD et CODI. Les résultats ont montré que les deux modèles décrivent adéquatement la cinétique transitoire de transfert de masse pendant le processus d'OD, mais le modèle Azuara était supérieur. MWODS a aussi été étudié pour évaluer l'effet des différentes variables de processus (concentration en saccharose, la température du moyen, le taux de flux et le temps de contact) en utilisant la méthodologie de surface de réponse et la décomposition rotative du programme global. Les modèles prédictifs ont été développés

pour relier les variables de réponse aux paramètres du procédé. Enfin, des études d'optimisation ont été menées pour élucider les conditions optimales de transformation sous MWODS. L'étude a démontré que la perte d'humidité (ML), les gains de solides (SG) et les pertes de poids (WR) ont été plus prévisibles à des concentrations de saccharose, températures de moyen, temps de contact et taux de flux plus élevés. Avec ML et WR, toutes les variables du processus, sauf le taux de flux, ont été prolifératives, tandis que pour SG, seul le temps de contact et son interaction avec le taux de flux ont été significatifs. Un ensemble de conditions optimales a été établi pour assurer un transfert de l'eau et un taux de réduction de poids plus élevés avec des niveaux modérés de gain des solides.

Une deuxième étape de séchage a été évaluée employant l'air de séchage conventionnel par rapport à la lyophilisation pour identifier les systèmes efficaces pouvant préserver la qualité des fruits osmotiquement déshydratés. L'effet du prétraitement du MWODS sur la cinétique du séchage à l'air et l'effet des paramètres de qualité (couleur, texture, et caractéristiques de réhydratation) des cylindres de pomme (*Rouge Gala*) ont été évalués. Les résultats ont révélé que le temps de séchage diminue avec l'augmentation des concentrations et de la température du moyen du traitement MWODS. Par rapport aux échantillons témoins non traités, les MWODS échantillons séchés à l'air ont un coefficient de diffusivité de l'humidité (D_m) plus élevée. En termes de paramètres de qualité, le processus de MWODS avec séchage à l'air arrive à un produit avec un changement de couleur inférieur et une structure plus tendre. Le produit séché à l'air sans MWODS avait les caractéristiques de qualité le moins souhaitable. Bien que la couleur a été mieux préservée dans le produit lyophilisé, le produit était beaucoup plus fragile que le produit MWODS avec séchage à l'air. La capacité de réhydratation des produits de MWODS séchés à l'air est supérieure de celle des produits séchés à l'air et celle des produits lyophilisés.

En général, cette recherche contribue à une meilleure compréhension du comportement de transfert d'humidité à micro-ondes pendant la déshydratation osmotique sous flux continu du moyen avec de la pulvérisation. Avec une deuxième simple étape du séchage à l'air, le processus peut produire des produits de pomme avec une haute qualité.

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CONTRIBUTIONS OF AUTHORS

Part of the thesis research has been used to prepare several presentations at conferences as well as prepare manuscript for publications. This research has been entirely supervised by Dr. Ramaswamy.

The author of this thesis was responsible for the design of experiments, experimental work, and manuscripts preparation under the guidance of Dr. Ramaswamy who helped in defining the problem and providing direct advisory input as the research work progressed. Dr. Ramaswamy is the co-author of all manuscripts that have been published and prepared for his special role in advising and editing of the manuscripts. Dr. Azad was a coauthor in a review paper for her contribution to the gathering of literature. Details of papers presented, published and prepared are provided below:

List of publications and scientific presentations

A: Part of this thesis has been published or submitted as follows;

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Azarpazhooh, E and Ramaswamy, HS. Microwave-Osmotic Dehydration of Apples under Continuous Flow Medium Spray Conditions: Comparison with Other Methods. Drying Technology, 28(1): 49-55, 2010.

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Azarpazhooh, E and Ramaswamy, HS. 2009. Recent Technologies for the Enhancement of Osmotic Dehydration. Agricultural and Biosystems Engineering Technology Conference. March 25, Saint-Hyacinthe, Canada. (Oral presentation)

Azarpazhooh, E and Ramaswamy, HS. 2008. Microwave –assisted osmotic dehydration of apple under continuous spray mode treatment. Annual meeting of Institute of Food Technologists, June 28- July 2, New Orleans, USA. (Poster)

CONTRIBUTIONS TO KNOWLEDGE

The present work contributes to expansion of the scientific knowledgebase in the general area of microwave osmotic dehydration and its influence on the mass transfer kinetics and quality attributes of apples. The specific contributions to knowledge of this thesis work are described below:

1. Microwave osmotic dehydration under continuous flow medium spray (MWODS) processing conditions is a new concept developed in this study.
2. Compared with conventional counterparts – conventional continuous flow osmotic dehydration in both immersion and spray modes (CODI, CODS) as well as MW OD under immersion mode (MWODI), MWODS offers distinct advantages of moisture loss (ML), higher weight reduction (WR), lower solids gain (SG), and higher ML/SG ratio. Therefore, MWODS has a distinct advantage over the other systems and offers great potential as a novel osmotic drying pre-treatment method.
3. It was recognized before that the microwave mode is superior to the conventional mode due to the unique heating of the food through microwaves which is rapid and direct. MW osmotic drying under medium immersion heating conditions was also documented earlier providing significant enhancement of mass transfer over conventional OD. MWODS with a spray mechanism is shown in this study to improved the MWODI performance. This is mainly because of the more efficient exposure of the fruit to MW field as compared to MWODI where it is mostly the syrup that is exposed to MW field. In addition, applying spray can also overcome one of the problems with osmotic dehydration- the floating of the fruit in the solution. These are highlighted for the first time.
4. The study demonstrated that the two-parameter Azuara model adequately describes the transient mass transfer kinetics in the osmotic dehydration process of apple cylinder. The study also demonstrated the Azuara model to be useful in predicting the equilibrium point for the moisture loss and solids gain based on the short duration osmotic treatments, when real long time equilibration data is not available. The study showed that in order to successfully use the conventional diffusion model for modeling the transient mass

transfer, it is necessary to add the intercept parameter making it also a two-parameter model like the Azuara model.

5. Response surface methodology was used for the first time to gather transient kinetic data on mass transfer during osmotic dehydration process and to evaluate the effects of process variables on the mass transfer kinetics of MWODS process and the conventional graphical and desirability function methods were used to identify a range of optimum processing conditions based on user selected optimization criteria. Normally, osmotic dehydration relies on experiments carried out using factorial designs which involve many more experiments than the CCRD models.

6. The effect of MWODS pretreatments on the subsequent air-drying behavior and quality parameters of the final product were also investigated. Compared with control samples (without any treatment), osmotically treated samples had higher moisture diffusivity during subsequent air drying process. Drying rate of MWODS samples were varied depending on pretreated conditions variation. Dehydrated product with lower color change and a more rigid and softer structure was obtained by the MWODS air-dried apples.

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NOMENCLATURE

ε_0	Absolute permittivity of vacuum ($8.854188 \times 10^{-12} \text{ F m}^{-1}$)
ε''	Loss factor
ε'	Dielectric constant
ε''	Loss factor
ε_0	Absolute permittivity of vacuum ($8.854188 \times 10^{-12} \text{ F m}^{-1}$)
j	$\sqrt{-1}$
ΔE	Total color difference
$ E $	Electric field strength (V m^{-1}).
a	Significant dimension, such as the radius of a cylinder
$a \ a_0$	Chromaticity coordinates (red to green) of sample and standard
$b \ b_0$	Chromaticity coordinates (yellow to blue) of sample and standard,
C	Mass concentration
D	Diffusion coefficient (m^2/s)
D_m, D_s	Diffusion coefficients of water and soluble solids, respectively (m^2/s)
f	Frequency Hz
hardness	N
I_m	Coefficient of moisture infusion
J_x	Flux ($\text{g H}_2\text{O}/\text{m}^2 \text{ s}$)
$L \ L_0$	Lightness of sample and standard, respectively (dimensionless)
M_0	Sample mass (kg) at time 0
MG_0, MG_t, MG_e	The fraction moisture gain at initial time, time t, and equilibrium, respectively
$ML-30$	Moisture loss (kg/kg wet base) at time 30
ML_0, ML_t, ML_e	The fraction moisture loss at initial time, time t, and equilibrium, respectively.
M_{mfc}	Unsteady mass concentration in a finite cylinder at final
M_{mfcw}, M_{mfcs}	Moisture loss ratio and solids gain ratio at final, respectively.

M_0, M_t, M_e	Sample mass at initial time, time t, and equilibrium, respectively (kg)
MR	Moisture ratio (dimensionless)
M_t	Sample mass (kg) time t
P	Energy $W\ m^{-3}$
RC	Rehydration capacity
Rigidity	N/mm
S_1	Constant related to the rate of water diffusion out from the product($\min^{-1}$)
S_2	Constant related to the rate of solid diffusion in the product($\min^{-1}$)
SG-30	Solids gain (kg/kg wet base) at time 30
SG_0, SG_t, SG	The fraction of solids gain at initial time, time t, and equilibrium
S_0	Solids fractions (kg/kg wet base) at time 0
s_0, s_t, s_e	Sample solids fraction at initial time, time t, and equilibrium, respectively (kg/kg wet base)
S_t	Solids fractions (kg/kg wet base) at time t
t	Time (s)
$\tan \delta$	Loss tangent
T_m	Time to get the sample moisture loss to a 25% (h)
T_s	Time to get the sample solids gain 3% (h)
T_w	Time to get the sample weight reduction a 20% (h)
V	Volume (m^3)
W	Moisture content ($g\ H_2O/m^3$)
Energy	J
W_r	Weight after rehydration (kg)
W_d	Weight of dried material(kg)
x	Space coordinates measured normal to the section (m)
X	Moisture content at any time t (kg/kg dry solid)
X_e	Moisture fractions (kg/kg dry solid) time equilibrium
X_0	Initial moisture content (kg/kg dry solid)
X_t	Moisture fractions (kg/kg wet base) time t
Y	Response variables
Z, Z_m, Z_s	The apparent half drying time with the superscripts m and s indicating water and soluble solids, respectively.
d_p	Power penetration depth

List of abbreviation

AD	Air- Dried
Adj-R ²	Adjusted- coefficients of determination
ANOVA	Analysis of variance
CCRD	Central Composite Rotatable Design
COD	Conventional osmotic drying
CODI	Conventional osmotic dehydration under continuous flow medium immersion
CODS	Conventional osmotic dehydration under continuous flow medium spray
CV	Coefficient of variation
DISP	Dewatering and Impregnation Soaking Process
FD	Freeze -dried
HDM	Hydrodynamic mechanism
HELP	High-intensity electric field pulses
HHP	High hydrostatic pressure
LOX	Lipoxygenase
ML	Moisture loss
MW	Microwave
MWOD	Microwave osmotic dehydration
MWODI	Microwave osmotic dehydration under continuous flow medium immersion
MWODS	Microwave osmotic dehydration under continuous flow medium spray
OD	Osmotic dehydration
PEF	Pulsed Electric Field
POD	Peroxidase
PVOD	Pulsed vacuum osmotic dehydration
R ²	Coefficients of determination
RSM	Response Surface Methodology
SG	Solids gain
VI	Vacuum impregnation
VOD	Vacuum osmotic dehydration

CHAPTER 1. GENERAL INTRODUCTION

Microwave osmotic dehydration (MWOD) under continuous medium flow is a new process with a good potential for quality optimization. It combines microwave process with osmotic dehydration and improves the mass transfer rate of osmotic dehydration process and product quality. Li and Ramaswamy (2006c) designed this process and found that the MWOD process under continuous immersion medium flow resulted in significant improvement in moisture loss and quality characteristics with limited solid gain. Obviously, the microwave heating causing a positive out flux of moisture from the product not only resulted in greater moisture loss, but also countered the solid gain. This research is an extension of Li's work capitalizing on the same principle. Immersion of the fruits in syrup limits the direct exposure of fruits to MW field as compared to those subjected to a spray. It is hypothesized that microwave combined with osmotic dehydration in the spray mode (MWODS) is more efficient and much easier to adapt under commercial conditions. Osmotic dehydration is widely used for the partial removal of water from plant tissues by immersion in a hypertonic (osmotic) solution. The driving force for the diffusion of water from the tissue into the solution is due to the higher osmotic pressure of the hypertonic solution. The diffusion of water is accompanied by simultaneous counter diffusion of solutes from the osmotic solution into the tissue. Since the membrane responsible for osmotic transport is not perfectly selective, other solutes present in the cells can also be leached into the osmotic solution (Lerici et al., 1985; Dixon and Jen, 1977). The rate of diffusion of water from any material made up of such tissues depends upon factors such as: temperature and concentration of the osmotic solution, the size and geometry of the material, the solution-to-material mass ratio and the level of agitation of the solution. A number of recent publications have described the influence of these variables on mass transfer rates during osmotic dehydration (Torreggiani, 1993; Raoult-Wack, 1994; Rastogi and Raghavarao, 1997). Certain complexities and defects are associated with osmotic dehydration processes that have yet to be studied and resolved to improve their efficiency. These include soluble solid leaching, and extensive solids uptake. Solute uptake and leaching of precious food constituents leads to a negative impact on sensory

characteristics and nutritional profile resulting in substantial modification of the original product (Lazarides and Mavroudis, 1995). The large solute uptake causes additional resistance to the mass transfer of water and leads to a lower dehydration rate during complementary drying (Wang and Sastry, 2000).

Since mass transfer rate is slow and the pretreatment is time-consuming (Rastogi et al., 2000a), a number of techniques such as blanching, freezing/thawing, partial vacuum (Simal et al., 1998), ultrasound (Farr, 1990), high-intensity electrical field pulses (Rastogi et al., 1999), high hydrostatic pressure (Rastogi and Niranjana, 1998) and microwave drying (Tulasidas et al., 1997; Funebo and Ohlsson, 1998; Maskan, 2000; Torreggiani and Bertolo, 2001; Nindo et al., 2003) have been used to improve the mass transfer rate. Microwave heating is based on a physical phenomenon generated by the interaction between electromagnetic waves and foods. Dipole rotation and ionic conduction are the two most important phenomena occurring during the MW heating. With dipole rotation, when polar molecules such as water are exposed to a MW field, rapid change in the direction of the electric field causes the water molecules to align in the direction of the electric field. As the molecules agitate, heat is generated. In ionic conduction, heat is produced because of the increased mobility of the ions caused by their exposure to a MW field (Feng and Tang, 1998). The most important characteristic of microwave heating is volumetric heating (Datta et al., 2005). Volumetric heating, caused by microwave power, drives moisture from the product's interior towards the surface, where it is removed by the heated up air in the surrounding. In microwave heating, heat is generated throughout the material, leading to faster heating rates, compared to conventional heating; where heat is usually transferred from the surface to the interior (Gowen et al., 2006). Interestingly, the use of microwaves to improve osmosis under spray system has never been reported. There is no work reported so far on the microwave osmotic dehydration under spray medium flow. The principal goals of this research project are thus to present an overview of the recent research progress in microwave heating and osmotic dehydration of fruits and vegetables, and to address the specific factors in these methods.

To accomplish these objectives, studies will be conducted on following aspects:

1. To develop the process of microwave osmotic dehydration under spray mode (MWODS) for apples
2. To compare the effect of sucrose concentration, temperature, flow rate and contact time on osmotic dehydration kinetic of apple (*Red Gala*) cylinders on osmotic dehydration under different conditions: a) Microwave osmotic dehydration under spray mode (MWODS), b) Microwave osmotic dehydration under immersion mode (MWODI), c) Conventional osmotic drying in immersion (CODI) and d) Conventional osmotic drying in spray (CODS) mode
3. To evaluate diffusion and Azuara models for predicting mass transfer kinetics during MWODS, MWODI, CODS, and CODI.
4. To evaluate factors influencing microwave osmotic dehydration under spray mode of apple cylinder
5. To optimize microwave osmotic dehydration under spray mode of apple cylinder using response surface methodology.
6. To investigate MWODS pre-treatment effects on convective air drying behavior of apples
7. To investigate MWODS pre-treatment effects on product quality parameters after second stage air drying.

CHAPTER 2. LITERATURE REVIEW

Microwave osmotic dehydration under continuous flow medium spray conditions (MWODS) is an emerging technology which has the potential for enhancing the mass transfer rate of the dehydration process as well as product quality. The goal of this review chapter is to address the available scientific information in literature related to this thesis research; mainly osmotic dehydration, microwave, air drying, and modeling aspects of drying and applications.

2.1 Introduction

Dehydration is a versatile, widespread technique in the food industry; it is the oldest and most frequently used method of food preservation. The main objective of drying is the removal of moisture so as to reduce the water activity and hence the associated microbial and enzymatic activity and product quality deterioration. Drying methods have been applied to extend the shelf life of the product; however, they often affect the quality of final products. The most common quality defects associated with dehydrated products are poor reconstitution, loss in texture, loss in the nutritive and sensory properties such as flavor and color. These are mainly due to the exposure of the product to high temperatures and long drying times mostly in the presence of air (Lenart, 1996; Lin et al., 1998). A new interest has recently arisen in finding new ways to improve the quality of dried food products. Many alternatives have been recognized such as the use of vacuum so that lower temperatures could be used, use of freeze drying which is done under conditions below the triple point of water facilitating sublimation; thereby protecting the product texture and other quality factors, use of rapid drying techniques which would reduce the drying time, use of novel heating sources like microwave and radio frequency heating (significant reduction in drying time), use of various treatments which promote better mass transport phenomena, etc. Osmotic dehydration has been lately recognized as a good pretreatment prior to regular drying to promote better quality and reduce energy needs (Torreggiani, 1993; Sereno et al., 2001b). Osmotic dehydration has the potential to remove water at low temperatures;

in addition, it is an energy efficient method, as water does not go through a phase change (Bolin et al., 1983).

Osmotic dehydration is gaining popularity as a complementary processing step in the chain of integrated food processing in the food industry due to its quality and energy related advantages. It has been shown that osmotic pre-treatment improves the quality of dried products including: reduced discoloration of the fruit from enzymatic browning (Ponting et al., 1966; Contreras and Smyrl, 1981), reduced heat damage to texture and color (Torreggiani, 1993), increased retention of volatiles (Flink, 1975; Dixon and Jen, 1977), increased sugar to acid ratio which improves the textural quality (Raoult-Wack, 1994) and low operating costs (Bolin et al., 1983). Osmotic dehydration is acknowledged to be an excellent energy saving method as moisture is efficiently removed from a food product without a phase change (Bolin et al., 1983; Uddin et al., 2004). In addition, the product is processed in the liquid phase, generally giving good heat- and mass-transfer coefficients (Raoult-Wack, 1994). The cost of shipping, packing and storing is also reduced due to the lower moisture content of the product (Rao, 1977; Biswal and Bozorgmehr, 1992). Since the water activity of the product is decreased, microbial growth is largely inhibited. However, the product is not shelf-stable since relatively large proportion of moisture still exists (up to 50%). Additionally, complementary treatments such as freezing (Tregunno and Goff, 1996; Maestrelli et al., 2001), freeze drying (Donsi et al., 2001), vacuum drying (Rahman and Mujumdar, 2007), air drying, osmo-convective drying (Islam and Flink, 1982; Corzo et al., 2008) and microwave drying (Orsat et al., 2007) are necessary in order to provide shelf stability to the product. Osmotic dehydration is a time-consuming process; therefore, supplementary methods are needed to increase the mass transfer without affecting the product quality (Rastogi et al., 2002). One of the distinctive aspects of osmotic dehydration in comparison with other dehydration methods is the incorporation of solute into the food system, to a certain extent, which can change the functional properties of the product; it is possible to achieve specific formulation properties without modifying its integrity (Torreggiani, 1993). Research on osmotic dehydration of foods was pioneered by Ponting et al. (1966), and since then a steady stream of

publications about it has appeared. These in general have dealt with various parameters, such as the mechanism of osmotic dehydration, the effect of operating variables on osmotic dehydration, modeling of water loss and solids gain, and enhancement of mass transfer (Torreggiani, 1993, Raoult-Wack, 1994; Khin et al., 2005; Mastrocola et al., 2005; Li and Ramaswamy, 2006a,b,c; Zhang et al., 2006; Falade and Igbeka, 2007; Vadivambal and Jayas, 2007). Since osmotic dehydration is an inherently slow process, several researchers have tried to increase the rates of osmotic mass transfer. These researches exclusively deal with the concept of osmotic dehydration, fundamental factors that affect osmotic dehydration, modeling of mass transfer, recent methods developed to enhance mass transfer rates, their industrial applications as well as future prospects.

2.2 Basic conception of osmotic dehydration

Osmotic dehydration can be defined as a ‘dewatering and impregnation soaking process’ (DISP) (Torreggiani, 1993; Raoult-Wack, 1994), a combination of dehydration and impregnation processes which can modify the functional properties of food materials, thereby creating new products. Osmotic dehydration can be defined as a simultaneous counter-current mass transfer process in which biological materials (such as fruits and vegetables) are immersed in a hypertonic aqueous solution for a selected period. The driving force for the diffusion of water from the tissue into the solution is the higher osmotic pressure of osmotic solution and its lower water activity that results in the transfer of water from the product across the cell wall. The diffusion of water is associated with the simultaneous counter diffusion of solutes from the osmotic solution into the tissue. This contributes to a net opposite flux of water and solutes that allow the tissue to become concentrated with a determined ratio of solute gain/water loss (SG/WL) depending on process conditions (Chiralt and Fito, 2003). Since the membrane responsible for osmotic transport is not perfectly selective, other solutes (sugar, organic acids, minerals, vitamins) present in the cells can also leach into the osmotic solution (Lenart and Flink, 1984a; Torreggiani, 1993) in amounts that are quantitatively negligible compared with the other transfer; however, they are important

in terms of final product quality (Dixon and Jen, 1977). During osmotic dehydration, there are different variables that affect the rate of water diffusion from any materials; therefore, it is difficult to establish general rules about them. However, osmotic pressure, plant tissue structure and mass transport relationships, are the most important ones (Islam and Flink, 1982; Lerici et al., 1985).

2.2.1 Osmotic pressure

Water as the main constituent of most foods affects food stability. During osmotic dehydration, water in solution is in interaction with solute. This interaction is characterized by the thermodynamic state of water. Energetic state of each substance can be defined as its internal energy which is called chemical potential. Chemical potential is a function of concentration, temperature, and pressure, however under isothermal conditions; it is just determined by concentration and pressure. The chemical potential can be defined according to the following relationship:

$$\mu_w = \mu_{0w} + RT \ln a_w \quad (2.1)$$

μ_w – chemical potential of water

μ_{0w} – chemical potential in a standard state

T – absolute temperature

R - gas constant

a_w – water activity coefficient

The energy is exchanged during the interaction of two systems with different energy state until reaching the equilibrium state. Under isothermal conditions, chemical potentials of two systems are the same, and can be achieved by the change of either concentration or pressure. Osmotic pressure is the excess pressure that pushes the system to reach the state of equilibrium between pure solvent and a solution and is expressed by the formula:

$$\Pi = -\frac{RT}{V} \ln a_w \quad (2.2)$$

where Π is the osmotic pressure and V is the molar volume of water.

2.2.2 The plant tissue structure

Plant tissue, as a living material, plays an important role during osmotic dehydration (Marcotte and LeMaguer, 1991). Although different parts of a plant such as roots, stems, shoots, leaves, flowers, fruits and seeds can be used during osmotic dehydration, all of them consist of these cells that are highly specialized. These tissues may consist of epidermal tissue which forms the outermost layer of cells which are thick and covered with a waxy substance known as cutin; parenchymatous tissue, is another tissue in main parts of organ, which has the ability to produce and store nutritional substances; and vascular tissue which can carry the solution of minerals and nutritional substances in a plant (Rahman and Perera, 1999).

A fresh plant tissue is composed of cells connected to each other by the middle lamella, and the protoplast (Mayor et al., 2008). The cell wall consists of three independent materials; cellulose microfibrils, hemicelluloses and pectin substance (Carpita, 1996). Hemicelluloses with branched polymers (xyloglucans, glucomannans) link with cellulose and pectin by hydrogen bonds. Generally, the rigidity of a dried product comes from the cellulose whereas plasticity comes from pectin and hemicelluloses (Lewicki and Pawlak, 2003). Middle lamella has two thin semi-permeable membranes: the tonoplast and the plasmalemma. Protoplast is separated by the plasmalemma from the cell wall, and cytosol solution. The osmotic phenomenon is largely controlled by the plasmalemma (Nobel, 1999). The cytosol is the major component of protoplast which contains different organelles such as the chloroplasts, mitochondria, peroxisomes, ribosomes, and proteins. These macromolecules and structures can affect the thermodynamic properties of water. The vacuole is a large central space inside the protoplast filled with water and surrounded by tonoplast (Mauro et al., 2003). A vacuole has an osmotic pressure that pushes protoplasm and

plasmalemma toward the cell wall. This osmotic pressure is called turgor pressure which is the difference between the pressure in the cell and its surroundings. When the cell and the surroundings have the same pressure, the turgor pressure is zero and the system is in equilibrium. If the osmotic pressure of the surroundings is higher than the cell, the water transfers into the cell and the cell swells. During osmotic dehydration, the plant cell is placed in a hypertonic solution with the osmotic pressure higher than that of the cell; as a result, the cell loses its water and decreases its volume. This process is called plasmolysis.

A mass transfer phenomenon is a complex mechanism occurring in plant tissue during osmotic dehydration. Water is transferred from the inner tissue to the outside, through the porous tissue structure, and then through the outside boundary layers. There are three important pathways during osmotic dehydration; symplastic (the transport within the intracellular volume), free-space transport (the transport within the extracellular volume) and apoplastic (water passing through plasma membranes) (Shi and LeMaguer, 2002). The transport of water between cells along the symplastic route is mediated by plasmodesmata, whereas in the transcellular path water has to cross plasma membranes. Furthermore, water moves across a tissue by crossing two membranes per cell layer and the apoplast (Steudle and Frensch, 1996). The removal of water during the osmotic process is mainly by diffusion and capillary flow, whereas solute uptake or leaching is only by diffusion.

2.2.3 Osmotic dehydration mass transport phenomena

In fruits or vegetables, the cell wall membranes are living biological units which can stretch and expand under the influence of growth and turgor pressure generated inside the cells. The semi-permeable membranes present in biological materials are the dominant resistance to mass transfer during osmotic dehydration. The cell membrane can change from being partially to totally permeable, leading to significant changes in tissue architecture (Rastogi et al., 2002). When plant cells are placed in a hypertonic solution, water removal starts from the surface that is in contact with the osmotic

solution, resulting in cell disintegration (Rastogi et al., 2000b). It is reported that sugars penetrate to a depth of 2-3 mm into the plant tissue while changes in water content are observed up to 5 mm (Bolin et al., 1983; Lenart and Flink, 1984b; Salvatori et al., 1999). Water leaves the cell surface by osmosis; therefore, the vacuole and the rest of the protoplasm will shrink, and plasmolysis occurs. However, the interior surface of the material can remain in full turgor pressure. A turgor pressure gradient results in the detaching of plasma membrane and the middle lamella due to the degradation or denaturation of the components of the middle lamella. Consequently, the mechanical properties of the product will change and the structure will deform. Lewicki and Porzecka-Pawlak (2005) reported cell debonding during osmotic dehydration of apple. Consequently, the cell is damaged and reduces in size by the loss of water and contact between the outer cell membrane and the cell wall (Rastogi et al., 2000b; Rastogi et al., 2002). Extensive uptake of osmoactive substance results in the development of a concentrated solids surface layer posing an additional resistance to mass transfer (Lenart and Lewicki, 1987; Lenart, 1994).

Consequently, porosity of the product will increase (Mayor et al., 2008), and the tissue shrinks because the amount of water flowing out is generally greater than the solutes diffusing in. Therefore, the weight of the foods will decrease, as will the water activity. It is reported that up to a 50% reduction in the fresh weight of fruits or vegetables may be brought about by osmosis (Rastogi et al., 1997; Kar and Gupta, 2001; Uddin et al., 2004). All these mass exchanges may have an effect on the organoleptic and/or nutritional quality of the dehydrated product (Sablani et al., 2002). As a consequence of this exchange, the product loses weight and shrinks. Cellular shrinkage during dehydration has been observed during osmotic dehydration of apple (Lewicki and Porzecka-Pawlak, 2005).

2.3 Factors affecting osmotic dehydration

The rate of diffusion of water from any material during osmotic dehydration is dependent upon factors such as type of osmotic agent, concentration of the osmotic solution, temperature, the size and geometry of the material, the solution-to-material

mass ratio and the level of agitation of the solution. There are several publications which describe the influence of these variables on mass transfer rate (Lerici et al., 1985; Raoult-Wack, et al., 1989; Raoult-Wack, 1994; Rastogi et al., 1997; Rastogi and Niranjana, 1998; Rastogi et al., 1999; Corzo and Gomez, 2004). However, the variables mentioned above can be manipulated over a limited range; outside of these ranges, the quality was adversely affected even though mass transfer rates may be enhanced (Rastogi et al., 2002). There are also some techniques which can be combined with osmotic dehydration, and have the ability to alter membranes in order to enhance mass transfer rate. They include: ultrasound (Rodrigues and Fernandes, 2007b) high-intensity electric field (Rastogi et al., 1999), high hydrostatic pressure (Akyol et al., 2006) and microwave (Li and Ramaswamy, 2006c; Azarpazhooh and Ramaswamy, 2010a,b). The choice of process conditions depends on the expected water loss, soluble solids gain, and the sensory properties of the food products.

2.3.1 Influence of size and shape on the mass transfer

Some research has been done on the influence of size and shape on the mass transfer kinetics. The surface area to volume ratio has been shown to be the influencing factor with higher ratios favoring better osmotic dehydration rates. Islam and Flink (1982) reported that the size and geometry of the food has some influence on the extent of final solute concentration, especially during short dehydration times; at such times, dehydration was primarily a transport phenomenon related to surface area. Lerici et al. (1985) compared osmotic drying of apple slices of four different shapes of (i.e slice, stick, ring and cube) and reported that the solids content increased with a decreasing surface area/volume ratio, but that the moisture loss was optimal for the ring shape. Van Nieuwenhuijzen et al. (2001) reported that moisture loss and solids gain increase as particle size is decreased under same processing conditions.

2.3.2 Osmotic solution composition and concentration

One of the predominant factors which affects the driving force and mass exchange is composition and concentration of the osmotic solution. These factors

should be continually controlled and regulated, as these characteristic changes during osmotic dehydration; therefore, Solution management is a major issue which must be taken into consideration precisely. Different solutes can be used in hypertonic solutions which can influence the taste and price of the final product. The solution in osmotic dehydration is generally sucrose for fruits and Sodium chloride for vegetable (Ade-Omowaye et al., 2002). Osmotic agent is solubility increases the driving force and mass transfer. The solution composition is based on the effectiveness, convenience and flavor of final product. Sugar and salt are the best osmotic solution, however their penetration inside the products are different. Sucrose is penetrated as a thin subsurface layer and results in creating a barrier for mass transport, while salt penetrates deeper into the osmosis tissue (Lenart and Flink, 1984b). It is reported that Sugar in osmotic solution can prevent polyphenoloxidase activity and inhibit losing of volatile compound in food (Zhang et al., 2006). Salt causes to increase mass transfer during osmotic dehydration (Rodrigues and Fernandes, 2007b). In most published literature, sucrose is used for fruits and sodium chlorides for vegetables, fish and meat.

The molecular size of the osmotic solution is another important parameter during osmotic dehydration and has also a significant effect on the water loss and solid gain. Smaller molecules obtain higher depth and extent of solute penetration (Hawkes and Flink, 1978; Lenart and Lewicki, 1987; Lerici et al., 1985), whereas increasing the molecular weight results in increasing the moisture loss. Saurel et al. (1994) found that adding ethylene glycol and polyethyl glycole increased the rate of moisture loss.

Although increasing the concentration of solute brought about increasing the moisture loss and solids gain (Hawkes and Flink, 1978; Conway et al., 1983), higher sugar concentrations (above 65%) did not affect the moisture loss (Ponting et al., 1966). Bolin et al. (1983) applied sucrose (disaccharide) and fructose (monosaccharide) corn syrup (HFCS) for osmotic dehydration of apple and found out that apple immersed in HFCS absorbed more solid than the apple in the sucrose solution and water removal in HFCS solution was a little bit more than that in sucrose solution. A binary or ternary system can be used during osmotic dehydration In ternary system (water/sugar/salt)

adding ionized molecule such as salt in osmotic solution increased the moisture loss in potatoes and apples because the salt molecule enters the product easily.(Islam and Flink, 1982; Biswal et al., 1991).

Heredia and Andras (2008) reported that the use of ternary solutions in osmotic dehydrations of tomatoes could be more appropriate than the use of binary solutions with the aim of maximizing water loss and minimizing solutes gain. The low molecular weight of sugars such as glucose is more effective in the transfer of water than the higher molecular weight due to limiting solids uptake of food material. Invert sugar has twice as many molecules per unit volume, and is more effective than sucrose. During osmotic dehydration, leaching the acid from the fruit into the syrup leads to accelerated hydrolysis of sucrose to glucose and fructose, resulting in increasing water removal (Bolin et al., 1983). It is recommended using osmotic dehydration less than 50% weight reduction due to the decrease in the osmosis rate with time (Torreggiani, 1993). It is reported that water loss mainly occurs during the first two hrs and the maximum solids gain within 30 min (Conway et al., 1983). Lazarides et al. (1995) showed that under the same osmotic process conditions, using corn syrups as osmotic agents result in lowering sugar uptake.

2.3.3 Contact time

The contact time of food with the osmotic solution is an important variable during osmotic dehydration (Ade-Omowaye et al., 2003). During osmotic dehydration, increasing the time of the osmotic treatment results in decreasing the rate of mass transfer while weight loss in food so treated is increased (Fasina et al., 2002). In terms of the contact time, the rate of both moisture loss and solids gain is the highest within the first hour of osmosis followed by progressively lower rates for the rest of the time. On average, moisture loss rates drop to about 20% of the initial rate during the first hour of dehydration and nearly level off at about 10% of the initial rate within three hrs. Solids gain rates show a similar decrease trend. Rapid loss of water in the beginning is

due to the large osmotic driving force between the dilute sap of the fresh fruit and the surrounding hypertonic solution.

2.3.4 Temperature of the solution

The mass transfer rate constants increased with increasing the temperature and sucrose syrup concentration (Magee et al. 1983 and Biswal et al. 1991). The temperature of the osmotic treatment is the most significant factor that influences the process of osmotic dehydration. The positive effect of temperature on the removal of water from the food during osmotic treatment has been shown by several researchers (Raoult-Wack et al., 1994; Rastogi and Raghavarao, 1994; Lazarides and Mavroudis, 1996). The main effect of high temperature is faster water diffusion and solid diffusion within the product which is accounted for by decreasing the viscosity of solution (Lazarides et al., 1995). Although increasing the temperature gives result in higher water removal, in fruit and vegetable the temperature above 60°C is not recommended due to a negative impact on the final product (Ponting et al., 1966). (Li and Ramaswamy, 2006a) illustrated that osmotic diffusion is a temperature-dependent phenomenon. Higher process temperature favored faster moisture loss. In osmotic dehydration of apple the solid gain and moisture loss increased with temperature but the rate of water removal was higher than the rate of solid gain. They also reported that the temperature above 60°C damaged the cell membrane of apple.

2.3.5 Agitation and food/ solution ratio

Agitation of the osmotic solution is an important aspect of the osmotic treatment. The agitation ensures that the concentrated solutions are restored around the particle surface and that a concentration difference favorable to mass transfer is recreated. The ratio of osmotic solution to fruit is an important consideration and often influences the production logistics, since it dictates the mass transfer momentum and the equilibrium concentrations. High solution/fruit ratios maintain constant solution concentration, and prevent dilution. On an industrial scale, the ratio needs to be as low as possible to restrict plant size and costs of solution regeneration. On the other hand,

use of a low ratio leads to significant transient changes in the solution composition. Most development studies are carried out with a large excess of osmotic solution to ensure minimal changes in solution concentration during test runs. The weight ratio of solution to product most often used is between 4 and 10.

2.4 Enhancement of osmotic dehydration

Osmotic dehydration is relatively slow so acceleration of mass transfer would be advantageous. There are various methods to increase the mass transfer, such as application of ultrasound, high hydrostatic pressure, high electrical field pulses, vacuum and centrifugal force and microwave.

2.4.1 Application of ultrasound during osmotic dehydration

Ultrasound in the food industry is relatively new and it has not been explored in-depth until recently (De Gennaro et al., 1999). Ultrasound has been applied in the food industry to determine certain food properties using low-frequency and high-energy ultrasound. A phenomenon known as acoustic cavitation is generated during the application as ultrasonic waves can generate minute vapor-filled bubbles that collapse rapidly or generate voids in liquids. Consequently, rapid pressure fluctuations are induced within the wet material by the ultrasonic waves. Ultrasound can be carried out at ambient temperature as no heating is required thus reducing thermal degradation (Rodrigues and Fernandes, 2007b). It can influence mass transfer through structural changes brought by the “sponge effect” (Stojanovic and Silva, 2007), and microscopic channels (Carcel et al., 2007) developed during cavitation. Applying ultrasound during osmotic treatment has a significant effect on the kinetics of water loss, sugar gain, and firmness loss, as well as on the microstructure of osmotically dehydrated different products and processes in liquid–solid system, such as osmotic dehydration of apples (Carcel et al., 2007); The use of ultrasound has been known to improve mass transfer for various products and processes in liquid-solid systems, such as osmotic dehydration (Stojanovic and Silva, 2007; Deng and Zhao, 2008). Water effective diffusivity increases with the use of ultrasound and decreases the amount of sugar in the fruit to

produce a dried low-sugar fruit (Rodrigues and Fernandes, 2007b). Gallego-Juarez et al. (1999) used high-intensity ultrasound to accelerate the osmotic dehydration rate of apples. Duan et al. (2008) used ultrasound pretreatment to improve the freeze-drying rate.

2.4.2 Application of blanching as a pretreatment

Hot water or steam blanching is a pretreatment before osmotic dehydration with the purpose of enzyme inactivation, and to promote gas removal from surfaces and intercellular spaces; oxidation, discoloration, and off-flavor development and microbial growth are thereby prevented (Rahman and Perera, 1999). Blanching has been applied prior to drying of fruits and vegetables such as bananas (Dandamrongrak et al., 2002), red paprika (Ade-Omowaye et al., 2001b), figs (Piga et al., 2004), potatoes (Eshtiaghi and Knorr, 1993), strawberries (Alvarez et al., 1995). However, blanching has some drawbacks such as causing changes in the chemical and physical state of nutrients and vitamins as well as having an adverse environmental impact from the large water and energy usage (Rahman and Perera, 1999). Water blanching (85–100 °C) usually results in loss of nutrients such as minerals and vitamins (Akyol et al., 2006).

2.4.3 Application of high hydrostatic pressure as a pretreatment

High-pressure (HP) treatments have been tested for their effectiveness as an alternate to thermal blanching (Eshtiaghi and Knorr, 1993) because they can be applied to liquid and solid foods, with or without packaging, at pressures between 100 and 800 MPa (Eshtiaghi et al., 1994). Akyol et al. (2006) showed that high hydrostatic pressure (HHP) with the combination of mild heat treatment can be used for blanching purposes to inactivate peroxidase (POD) and lipoxygenase (LOX) in carrots, green beans, and green peas. In addition, high pressures cause permeabilization of the cell structure (Farr, 1990; Eshtiaghi et al. 1994) leading to the enhancement of mass transfer rates during osmotic dehydration. Rastogi and Niranjana (1998) reported that the application of HP on pineapples damaged cell wall structure, leaving the cells more permeable with

a reduction in intercellular material. Taiwo et al. (2001) reported that high pressure may be considered during osmotic dehydration when sugar uptake in the product is desired.

2.4.4 Application of vacuum during osmotic dehydration

Application of vacuum impregnation (VI) simultaneously with osmotic treatment for a short period of time has been widely studied (Fito, 1994). Vacuum impregnation is widely used simultaneously with osmotic treatments to enhance mass transfer and promote more homogeneous concentration profiles in the fruits (Fito et al., 2001). The total transport of water and solute during vacuum pulse osmotic dehydration is caused by two mechanisms: the hydrodynamic mechanism (HDM) and pseudo-fictions mechanism. HDM is promoted by pressure gradients and penetration into the pores of plants over a short time period and the pseudo-fiction mechanism is driven by activity gradients over longer time frames (Fito, 1994). During vacuum impregnation especially in porous products, the action of hydrodynamic mechanisms (HDM) is combined by diffusional phenomena to promote mass transfer (Fito et al., 2001). When a vacuum pulse is applied in the system, the gas and liquids in the internal pores of the product are replaced by the external liquid, and the impregnation process is completed practically by the external solution, resulting in change in the mass transfer behavior in the product due to its porosity reduction (Fito, 1994). When VI is applied, the mass loss is reduced as compared with the process carried out at atmospheric pressure. Moreover, the process yield is increased due to lower mass loss in comparison with atmospheric pressure. In addition, the products are enriched with nutrients, vitamins, minerals, and other additives; in many cases, the sensorial properties of the product are improved (Chiralt et al., 2001a). Vacuum impregnation has a great influence on product characteristics such as the internal ratio, water loss and solids gain (Barat et al., 2001b; Chafer et al., 2001).

Deng and Zhao (2008) reported the significant effect of pulsed-vacuum on depressing a_w , titratable acidity, and in improving color L value of osmo-dehydrated apples. Vacuum osmotic dehydration (VOD) and pulsed-vacuum osmotic dehydration

(PVOD) reduced process time and energy costs (Paes et al., 2007; Deng and Zhao, 2008). Laurindo et al. (2007) developed a device for measuring the dynamics of the vacuum impregnation (VI) process. The device can measure the net force emitted by a food and transfer it to the VI process by a load cell. Determination of water in this system during the VI process is not required which increases the accuracy of the results. The experimental device can satisfactorily quantify the influence of the vacuum level, something that is very important for food process design. Vacuum impregnation (VI) processes reduced the process time (approximately 85%) and the weight loss (approximately 48%), thus increasing yield (Larrazabal-Fuentes et al., 2009). Furthermore, it is a minimally processed method in which the organoleptic characteristics of products and their shelf life are enhanced (Fito et al., 2001; Moreno et al., 2004; Correa et al., 2010). Pulsed vacuum osmotic dehydration (PVOD) is a new method which is applied for a short (normally 5 min) vacuum treatment to a fruit dipped in an osmotic solution, and after that the osmotic dehydration is done at atmospheric pressure. The benefit of this method is that it reduces energy costs (Panadés et al., 2006). Castelló et al. (2010) investigated the effect of osmotic dehydration on the mechanical and optical properties of strawberry halves by applying (PVOD) and adding calcium. They reported that calcium addition and PVOD treatments had beneficial effects on the maintenance of the sample texture during storage. In addition, the sample porosity was greater due to the treatment (vacuum impregnation) which resulted in modifying the color of strawberries. According to different researches (Fito et al., 2001; Barat et al., 2001a; Chafer et al., 2003; Giraldo et al., 2003) higher effective diffusivity values are obtained with the vacuum pulse pre-treatment and with a decrease in the osmotic solution concentration during the osmotic treatment. Correa et al. (2010) reported higher weight loss of osmotically dehydrated guavas when higher sucrose solution concentrations and vacuum pulses are employed. They reported that solid uptake was favored by vacuum application. Increasing the osmotic solution concentration induces an increase in the mass transfer (Barat et al., 2001a; Giraldo et al., 2003; Panadés et al., 2006; Ito et al., 2007).

2.4.5 Application of pulsed electrical field during osmotic dehydration

The pulsed electric field (PEF) as a non-thermal method has been reported to increase permeability of plant cells with positive influence on mass transfer in further processes. The potential of PEF during osmotic dehydration for the first time was demonstrated by Rastogi et al. (1999). This finding has created more research looking into the ability of PEF as a pre-treatment during osmotic dehydration of plant foods. The Pulsed Electric Field as a non-thermal method can increase the cell permeability in a short time (μ s to ms range) while keeping the product matrix unaltered, thereby positively accelerating mass transfer during osmotic dehydration (Ade-Omowaye et al., 2001a). Taiwo et al. (2001) studied the effect of high-intensity electric field pulses (HELP) pretreatment on the diffusion kinetics of apple slices. They reported that HELP has a very minimal effect on solids gain; and application of HP is advantageous when moisture reduction and minimal alteration in product taste are desired. Moreover, firmer texture, brighter color, and better retention of vitamin C are the advantages of applying HELP with osmotic dehydration. Lazarides and Mavroudis (1996), Ade-Omowaye et al. (2001b) and Taiwo et al. (2001) reported that PEF pre-treatment might be a better alternative than processing at high temperatures.

2.4.6 Application of microwave during osmotic dehydration

Microwave-osmotic dehydration is a novel technique with a good potential for more efficient osmotic drying of fruits and vegetables. Carrying out osmotic drying in a microwave environment enhances moisture removal when moist food is immersed in a concentrated solution of an osmotic agent (Li and Ramaswamy, 2006c). The osmotic concentration gradient effect existing between the solution and food, which is the driving force for the removal of moisture from the food into the osmotic medium, is enhanced under the microwave field. This is due to selective absorption of microwave energy by the water molecules in food resulting in increased moisture out-flux, which also has the tendency to limit the simultaneous transfer of solute from the solution into the food. Li and Ramaswamy (2006c) investigated the mass transport coefficients under microwave-osmotic dehydration (MWOD, immersion medium) and compared it with the conventional osmotic dehydration process (COD). They reported that MWOD

significantly increased the rate of moisture loss and decreased the rate of solids gain. They also found that the osmotic dehydration under microwave heating made it possible to obtain a higher diffusion rate of moisture transfer at lower solution temperatures. In their experiments, they immersed the apple slices in the osmotic solution placed within the microwave field. In such an immersion medium, because the sample is surrounded by a large volume of the solution, the absorption of microwaves by the sample itself will be limited, thus reducing the moisture out-flux effectiveness of the microwaves. This finding has helped to stimulate new research employed in this study. Microwave osmotic dehydration under continuous flow medium spray conditions was developed and shown to provide a means of effecting moisture loss and limiting solids gain that was far superior to three other techniques under similar continuous-flow conditions (Azarpazhooh and Ramaswamy, 2010a). It was clearly demonstrated that spray mode heating enhanced the efficiency of the system. This is likely due to the direct and more efficient exposure of the sample to the microwave field. As opposed to the large volume of solution that surrounds the sample in the MWOD immersion system, the spray mode only uses a thin layer of osmotic solution that is continuously flushed down due to the rapidly flowing medium and gravity. The spray mode also eliminates the problem of sample floating, which can restrict the application of immersion mode (Azarpazhooh and Ramaswamy, 2010a). Microwave heating has the specific advantage of rapid and uniform heating due to the penetration of microwaves into the body of the product (Bilbao-Sainz, et al., 2006; Alibas, 2007). The most important characteristic of microwave heating is volumetric heating, which refers to the material absorbing microwave energy directly and internally and converting it into heat. Heat is generated throughout the material, leading to faster heating rates (compared to conventional heating, where heat is usually transferred from the surface to the interior) and producing rapid and uniform heating (Gowen et al., 2006). Microwave heating, causing a positive outflux of moisture from the product, not only results in greater moisture loss but also restricts a higher solids gain.

2.4.6.1 Mechanisms of microwave heating and drying

Microwaves are high frequency electromagnetic waves that can be reflected, transmitted or absorbed, depending on material properties. Microwave energy is electromagnetic radiation in the frequency range between 300MHz to 300GHz (Decareau and Peterson, 1986). The electromagnetic spectrum of *microwaves* overlaps with those used for telecommunications and hence are regulated. Only two microwave frequencies 915 and 2450 MHz are reserved for heating (Orsat et al., 2005). Microwave heating involves the conversion of electromagnetic energy into heat by selective absorption and dissipation. Dipole rotation and ionic polarization are the two major mechanisms that govern microwave heating of dielectric materials. In microwave heating, when molecules carrying dipolar electrical charges such as water are exposed to a microwave radiation, they attempt to align their dipoles with the rapidly changing electric field. Consequently, heat is generated due to the friction of the molecules which can then transfer to the next molecule (Dibben, 2002). In ionic conduction, ions are accelerated by electric fields causing them to move towards the direction opposite to their own polarity. The movement of the ions such as (Na^+ , Cl^- , Ca^{++}) in solution initiates collisions between the molecules of the material; consequently, heat is generated throughout the food. If the solution has more ions, more collisions will happen and ultimately the temperature will rise (Dibben, 2002). The energy conversion from electrical energy to stored potential energy in the material results in the storing of random kinetic or thermal, energy in the material. Generally, the average power density P (volumetric absorption of microwave energy, W/m^3) produced in a material when exposed to microwave energy is defined by the following equation (Venkatesh and Raghavan, 2004):

$$P = 2\pi f \epsilon_0 \epsilon'' |E|^2 \quad (2.3)$$

where P is the energy developed per unit volume (W m^{-3}); f is the frequency (Hz); ϵ_0 is the absolute permittivity of vacuum ($8.854188 \times 10^{-12} \text{ F m}^{-1}$); ϵ'' is the loss factor; and $|E|$ is the electric field strength inside the load (V m^{-1}). Microwave heating has the ability to heat evenly thick material, and areas with high liquid content. During

microwave heating, the internal temperature of the heated sample may reach the boiling point of water in which free moisture is evaporated inside the product; consequently, a vapor pressure gradient that expels moisture from the sample is created (Bouraoui et al., 1993; Orsat et al., 2007).

2.4.6.2 Dielectric properties and their effects on microwave heating

The dielectric properties of materials are important factors when applying microwave electromagnetic energy. The absorption of microwave energy and the consequent heating behavior of food materials in microwave heating and processing applications can be determined by dielectric properties along with thermal and other physical properties, and the characteristics of the microwave electromagnetic fields (Dibben 2002). The dielectric constant ε' and the loss factor ε'' , which are the physical parameters that govern the microwave-matter interaction, vary with different frequencies, and are also dependent on the temperature, moisture content, composition and particle density of the material. The dielectric constant ε' is the ability of a material to couple with microwave energy, and the dielectric loss factor ε'' of the material is the ability of a material to dissipate electric energy (Nelson 1973; Venkatesh and Raghavan, 2004). These are represented by:

$$\varepsilon = \varepsilon' - j\varepsilon'' \quad (2.4)$$

$$\tan \sigma = \frac{\varepsilon''}{\varepsilon'} \quad (2.5)$$

where $\tan \sigma$ is the loss tangent, $j = \sqrt{-1}$ which indicates a phase shift between the real ε' and imaginary ε'' parts of the dielectric constant.

The dielectric properties of most materials are affected by many different factors, but the amount of water is generally the dominant factor. They also depend on the frequency of the electric field applied, the temperature of the material and the material density, structure and chemical composition, especially on the presence of

mobile ions. An increase in ions concentration in water has a significant effect on its dielectric properties and structure that can increase the loss factor, reducing its permittivity by a few percent (Meredith, 1998). McMinn and Magee 1999 pointed out that this increase of the loss factor affects the microwave penetration depth (dp) which represents the distance into a sample where the microwave power has dropped to 1/e or 36.8% of its transmitted value (Datta et al., 2005).

$$d_p = \frac{\lambda_0 \sqrt{\epsilon'}}{2\pi\epsilon''} \quad (2.6)$$

where λ_0 is the free space microwave wavelength (for 2.45 Hz, $\lambda_0=12.2$ cm). The most common food products have $\epsilon'' < 25$, which implies a dp of 0.6- 1.0 cm.

2.5 Modeling of osmotic dehydration

Although considerable efforts have been made to improve the understanding of mass transfer in osmotic dehydration, fundamental knowledge about predicting mass transport is still a gray area (Raoult-Wack et al., 1991). Modeling of the osmotic dehydration process is necessary for optimizing the osmotic dehydration and subsequent drying processes, in order to achieve the highest possible quality at minimum energy costs (Saguy et al., 2005). The unusual features come from the interaction between the solution and materials of biological origin. Mass transfer in osmotic dehydration of cellular plant foods, such as fruit and vegetables, involves several physical effects due to the complex morphology of plant tissues. These can be described, in an ideal way, as osmosis, diffusion and hydrodynamic mechanism (HDM) penetration (Fito and Pastor, 1994). Two basic approaches can be used to model osmotic processes (Ramaswamy et al., 1982; Salvatori et al., 1998). The first one, the macroscopic approach, assumes that the tissue is homogeneous and the modeling is carried out on the cumulated properties of cell walls, cell membranes and cell vacuoles. The second one, the microscopic approach, identifies the heterogeneous properties of the tissue and is based on cell microstructure (Fito et al., 1996).

2.5.1 Macroscopic approach

Macroscopic analysis has been carried out on pseudo-diffusion, square root of time, irreversible thermodynamic and other approaches (Fito et al., 1996). Very little work has been developed from the microscopic point of view (LeMaguer, 1996). The analysis of the concentration profiles developed throughout mass transfer processes, using a macroscopic approach, can be useful to clarify the mass transfer mechanisms and their coupling, especially if data are correlated with micro-structural features (shape, size and geometry changes in cell and intercellular spaces, cell wall deformation and relaxation changes, etc.) observed by a microscopic technique (Alzamora et al., 1996). However, concentration profiles allow us to calculate mass transfer kinetics (Lenart and Flink, 1984c). Mathematical modeling may provide a useful insight into the underlying mechanisms and several mathematical models have been proposed based on a cellular structure approach that assumes water transport as a trans-membrane movement or Fick's second law with estimation of diffusion coefficients for both water loss and sugar gain (Azuara et al., 1992; Fito et al., 1996; Yao and Le Maguer, 1996) also including hydrodynamic mechanisms (Fito et al., 1996; Salvatori et al., 1998). In addition, empirical and semi-empirical models are often applied (Panagiotou et al., 1999; Barat et al., 2001b).

A number of investigators have used Fick's unsteady state law of diffusion to estimate the water or solute diffusivity, simulating the experiments with boundary conditions to overcome the assumptions involved in Fick's law (Barat et al., 2001b; Ade-Omowaye et al., 2002; Fasina et al., 2002; Telis et al., 2004). There are two parameters required in Fick's law; these are sample dimensions and the effective diffusion coefficient. The effective diffusion coefficient can be obtained by finding numerical or analytical solutions to experimental data (Nguyen et al., 2006), calculating the relation between the slope of theoretical diffusion curve and the slope of experimental mass transfer ratio (Rastogi et al., 2000a; Ade-Omowaye et al., 2002;

Rastogi et al., 2002), and applying linear and nonlinear regressions (Akpinar, 2006). Much of the literature considers any finite food geometry as an infinite flat plate configuration, neglecting the diffusion in the other directions. Of these studies, only a few have considered unsteady state mass transfer during osmotic dehydration (Escriche et al., 2000; Roberts et al., 2002; Ade-Omowaye et al., 2002; Kayacier and Singh 2004; Rastogi and Raghavarao 2004).

Modeling of diffusion is a combination of physical and empirical approach. Mass transfer studies in food rehydration are typically founded on Fick's 1st and 2nd laws:

$$J_x = -D \frac{dW}{dx} \quad (2.7)$$

$$V \frac{\partial W}{\partial t} = D \frac{\partial^2 W}{\partial x^2} \quad (2.8)$$

where: J_x , flux ($\text{g H}_2\text{O}/\text{m}^2 \text{ s}$); W , moisture content ($\text{g H}_2\text{O}/\text{m}^3$); x , spatial coordinate (m); t , time (s); D , diffusion coefficient (m^2/s); V , volume (m^3).

The second unsteady Fick's law allows the estimation of the diffusion coefficients for both water loss and solids gain individually or simultaneously. The mass transfer is assumed to be unidirectional and the interactions of the other components on the diffusion of the solute are negligible. Analytical solutions of the equation are available for idealized geometries, i.e. spheres, infinite cylinders, infinite slabs, and semi-infinite medium. For these analytical solutions of the unsteady state diffusion model to exactly apply, it is necessary either to keep the external solution concentration constant or to have a fixed volume of solution. The resistance at the surface of the solids is assumed to be negligible compared to the internal diffusion resistance in the solids. Biswal et al. (1991) and Ramaswamy and van Nieuwenhuijzen (2002) used a rate parameter to model osmotic dehydration of green beans as a function of solution concentration and process temperature. The parameter was calculated from

the slope of the straight line obtained from bean moisture loss and solids gain vs. the square root of time (Biswal et al., 1991).

Azuara et al. (1992) developed a model based on mass balances of water and sugar to predict the kinetics of water loss and solids gain during osmotic dehydration. The model is related to Fick's second law of unsteady state one-dimensional diffusion through a thin slab in order to calculate the apparent diffusion coefficients for each condition. Correlative models have been proposed, either to compute the time required for a given weight reduction as function of the processing temperature and of the solution concentration or to estimate the dehydration parameters. Nsonzi and Ramaswamy (1998b) studied osmotic dehydration kinetics of the blueberry and further modeled moisture diffusivity and soluble solids diffusivity with quadratic functions of temperature and concentration. Azuara's model has the advantage of allowing the calculation of the equilibrium values of moisture loss and solids gain (ML_e and SG_e) (Ochoa-Martinez et al., 2007b).

2.5.2 Microscopic approach

The mass transfer phenomena occurring in plant tissues during osmosis involves complex mechanisms, most of them controlled by the plant cells. During osmotic dehydration of cellular material, mass transfer inside the cellular material depends on both processing variables and micro-structural properties of the biological tissue. There is a naturally wide variation in the physical nature of raw food material. When biological cellular material undergoes osmotic dehydration, mass fluxes in the system imply changes in structural and transport properties (volume, dimension, viscosity, density, porosity, etc.). As a result, these changes affect the mass transfer fluxes. The changes of material tissue volume and porosity promote the action of non-diffusional driving forces, such as a pressure gradient associated with the relaxation of a deformed cell network to release the structural stress (Lozano et al., 1983; Mayor and Sereno, 2004), and changes in mechanical properties (Telis et al., 2005) and color changes (Krokida et al., 2000b). Knowledge of and predictions about these changes are

important because they are related to quality factors and some aspects of food processing, such as food classification, process modeling and design of equipment (Perera, 2005). Most of these changes, although observed at a macroscopic level, are caused by changes occurring at the micro-structural/cellular level. In this way, the study of the micro-structural changes during dehydration is important because it can help to understand and predict the changes occurring in the physical–chemical properties at higher levels of structure. Mass transfer (and eventually heat transfer) phenomena result in changes at microscopic and macroscopic levels and consequently variations in the physical properties of the food system. These changes also produce alterations in mechanisms and kinetics in the transport phenomena (Fito and Chiralt, 2003).

2.6 Complementary drying method

Osmotic dehydration is a pretreatment which can improve nutritional, sensorial and functional properties of food without changing its integrity (Torreggiani, 1993). Osmotic dehydration is generally used as a preliminary step for further processing such as freezing (Ponting et al., 1966), freeze drying (Hawkes and Flink, 1978), vacuum drying (Dixon and Jen, 1977), microwave heating and processing applications (Nelson and Datta, 2001), and air drying (Piotrowski et al., 2004; Mandala et al., 2005). Abundant information is available on the application of an osmotic treatment before conventional air drying (Lemus-Mondaca et al., 2009; Vazquez-Vila et al., 2009). Sharma et al. (1998) studied the influence of some pretreatment parameters such as steam blanching and sulfur dioxide treatment on product quality during osmo-air dehydration processing of apples. They found greater retention of ascorbic acids in treated samples with sulfur dioxide followed by osmotic dip and vacuum drying than in non-treated samples. Riva et al. (2005) observed that vitamin C was retained higher by osmo-air dried apricot samples than by non-treated air dried samples. They attributed this phenomena to a lower phenolase activity and the protective effect of the sugar. Several authors have reported that the texture, flavor, and color stability in dried fruit and vegetables are improved. This is especially important since color may be a decisive

factor in the consumer's acceptance of a food (Krokida et al., 2000a, b; Gujral and Brar, 2003; Koyuncu et al., 2003).

2.7 Impact of osmotic dehydration on properties

Osmotic treatment of fruits and vegetables preceding convective drying may strongly affect properties of the final product (Lewicki and Lukaszuk, 2000; Lewicki and Pawlak, 2003). During osmotic dehydration, many aspects of cell structures are affected such as alteration of cell walls, splitting of the middle lamella, lysis of membranes (plasmalemma and tonoplast), tissue shrinkage (Alvarez et al., 1995; Nieto et al., 1998) which could strongly influence the transport properties of the product during processing. All these phenomena cause changes in the macroscopic properties of the sample, such as optical and mechanical properties, which are related to the product appearance and texture, respectively. All these changes greatly affect organoleptic properties of the osmo-dehydrated plant due to solute uptake and leaching of natural acids, color, and flavor compounds out of osmo-dehydrated plant tissue; as a result, natural composition of the product is modified (Lazarides et al., 1995). Although compositional changes may have a positive and negative effect on the final product, rehydration of osmotically dried fruit is lower than in the untreated fruit due to the rapid impregnation of a subsurface tissue layer with sugar (Nsonzi and Ramaswamy, 1998a); moreover, if the osmosis takes more time, the rehydration rate would be lower.

2.7.1 Impact of osmotic dehydration on color

Many investigators demonstrated that the quality (color, texture and rehydration capacity) of air, freeze or vacuum- dried fruits and vegetables could be improved by a prior osmotic step (Flink, 1975; Hawkes and Flink, 1978; Lerici et al., 1985; Nsonzi and Ramaswamy, 1998a). There have been numerous research studies on color change during osmotic dehydration. The color of the products is measured by lightness (L^* value), redness or greenness (a^* value) and yellowness or blueness (b^* value), during or after drying. Falade et al. (2007) reported transparency and color of the fruit may alter favorably due to physical and chemical changes during osmotic dehydration. They

evaluated L^* , a^* , b^* values of osmosed and osmo-oven dried watermelon, and reported that color parameters increase with an increase in osmotic solution concentration. Osmotic dehydration improves fruit quality by stabilizing color parameters and allows less color loss of fruit from enzymatic oxidative browning due to the infusion of sugars and elimination of dissolved oxygen. In addition, reducing the water activity of samples also decreases the non-enzymatic browning reaction (Krokida et al., 2000b).

Osmotic dehydration eliminates or reduces the use of preservatives such as sulfur dioxide in fruits. In addition, substantial amount of air from the tissue is removed; therefore blanching prior to osmotic dehydration also can be omitted (Torreggiani, 1993; Lenart, 1996).

2.7.2 Impact of osmotic dehydration on texture

Texture is a significant quality attribute of fruits and vegetables. During osmotic dehydration, the textural properties of osmo-dehydrated products are changed due to physical and chemical modifications occurring in the cell structure (Lewicki, 1998). Properties of the cell wall and middle lamella and the turgor pressure are the most important factors to determine the texture of plant tissue (Jackman and Stanley, 1995; Chiralt et al., 2001b). Plant tissue is affected by size and shape of the cell, volume of the vacuole, intercellular spaces volume, presence of starch granules and chemical composition (Ilker and Szczesniak, 1990). The majority of foods have visco-elastic behavior; that is why, during osmotic dehydration, the viscous nature of fruits and vegetables increases while their elasticity decreases due to the sugar uptake (Telis et al., 2005; Mayor et al., 2007). Osmotic dehydration weakens the texture of apples and makes apple tissues softer and more plastic than those of raw apple (Monsalve-GonaLez et al., 1993). Although there are numerous reports dealing with the effect of some sugars on the structural properties of osmo-treated plant material (Marcotte and LeMaguer, 1991; Maltini et al., 1993; Barat et al., 2001b), only a few reports talk about the structural changes at the cellular level which are only accessible through microscopic observations (Willis and Teixeira, 1988; Saurel et al., 1994; Martinez-

Monzo et al., 1998). Puncture force is usually used to measure the textural properties of dehydrated products which is the measure of the hardness of the product surface, and presents the extent of case hardening during drying (Lin et al., 1998). During osmotic treatments, the main changes that affect the mechanical behavior of plant tissue are changes in the air and liquid volume fractions in the sample, the size and shape of the sample (Fito, 1994), loss of cell turgor, alteration of middle lamella (Alzamora et al., 1996), alteration of cell wall resistance, establishment of water and solute concentration profiles and compositional profiles in osmotically dehydrated samples (Salvatori et al., 1998). Differences in mechanical behavior of the dried samples must be related to the differences induced in the composition of the soluble water phase and in the solid matrix during treatments. Contreras et al. (2007) reported that soluble pectin is increased during drying which alters cell bonding zone resulting in change of the solid matrix consistency. Osmotic dehydrated products have a softer texture due to leaching of calcium into the osmotic solution which in turn results in lowering the concentration of calcium content ions inside the tissue (Prothon et al., 2001).

2.7.3 Impact of osmotic dehydration on rehydration capacity

There is a need for understanding the rehydration process due to the wide variety of dehydrated foods which are available today to consumers. A particular concern is in meeting quality specifications and conserving energy. Dehydrated products are usually rehydrated by immersion in water or other liquids, such as fruit juices, sucrose or glucose solutions. Restoring the properties of the fresh product by immersing dehydrated products in a liquid phase is an important aspect during rehydration. Rehydration can reflect the physical and chemical changes that have occurred during osmotic dehydration, and can therefore be used as a quality index. In other words, any pretreatment to which the products have been subjected may have modified the composition structure of the samples (Maskan 2001a). The rehydration process is typically composed of three simultaneous steps: absorption of water into the dry material, swelling of the rehydrated product, and loss or diffusion of soluble components (Lee et al., 2006). It is reported that increasing the rehydration temperature

in the range of 40–80 °C for many fruits and vegetables, including bananas, carrots, apples, potatoes, tomatoes, and yellow, red, and green peppers markedly increased the volume of the product (Krokida and Marinos-Kouris 2003). In order to design and optimize rehydration, different mathematic models can be used to describe how certain process variables affect water transfer (Krokida and Marinos-Kouris 2003). Some researchers have assumed simple least-squares adjustment to models based on exponential models or capillary absorption theory, while others have used Fick's diffusion laws, and demonstrated that a model based on first-order kinetics can properly describe the gain of water during rehydration (García-Pascual et al.,2005; Giraldo et al.,2006; Krokida and Marinos-Kouris 2003; Lee et al.,2006; Maskan 2001a). There are three methods to estimate rehydration characteristics of dehydrated products: (1) water absorption capacity, WAC, which is the capacity of a matrix to absorb water that replaces the water lost during drying (2) dry mass retention capacity, DHC, which is the material ability to retain soluble solids after rehydration, and (3) rehydration ability or capacity, RA, which is the ability of a dehydrated product to rehydrate, and which shows total damage to tissues caused by drying and impregnation during rehydration (Maldonado et al.,2010).

CONNECTIVE STATEMENT TO CHAPTER 3

The importance of osmotic dehydration as a pretreatment to enhance the quality of fruits and vegetables was highlighted in the previous chapter. Despite the large amount of research works that have been published in the area of osmotic drying, industrial scale applications have faced some limitations such as, extensive solute uptake, difficulty in the movement of the highly viscous osmotic solution, product floating and time consuming pre-treatment. Alternate methods which enhance the performance of osmotic dehydration are therefore welcomed. In this chapter, a special method based on microwave osmotic dehydration under continuous flow medium spray condition (MWODS) was designed and compared with other existing methods (MWOD under immersion mode (MWODI) and conventional osmotic dehydration in both spray (CODS) and immersion (CODI) modes).

This research work was completed by the Ph.D. candidate under the supervision of Dr. HS. Ramaswamy.

Part of this study has been used for presentations and publications as follows:

Azarpazhooh, E and Ramaswamy, HS. 2009. Recent Technologies for the Enhancement of Osmotic Dehydration. Agricultural and Biosystems Engineering Technology Conference. March 25, Saint-Hyacinthe, Canada, (Oral presentation).

Azarpazhooh, E and Ramaswamy, HS. 2008. Microwave- assisted osmotic dehydration of apple under continuous spray mode treatment. Annual meeting of Institute of Food Technologists, June 28- July 2, New Orleans, USA. (Poster).

One manuscript has been published:

Azarpazhooh, E and Ramaswamy, HS. 2010. Microwave-Osmotic Dehydration of Apples under Continuous Flow Medium Spray Conditions: Comparison with Other Methods. *Drying Technology*, 28(1): 49-55.

CHAPTER 3. MICROWAVE OSMOTIC DEHYDRATION OF APPLES UNDER CONTINUOUS FLOW MEDIUM SPRAY CONDITIONS: COMPARISON WITH OTHER METHODS

Abstract

Microwave osmotic dehydration (MWOD) under continuous medium flow is a new technique with good potential for quality optimization. It combines microwave heating with osmotic dehydration for enhancing moisture transfer rate in the osmotic dehydration process and safeguarding product quality. This study was carried out to investigate the effects of MWOD of apple (*Red Gala*) cylinders in continuous medium-flow immersion (MWODI) and spray (MWODS) conditions, as well as compared with conventional osmotic drying (COD) under similar continuous medium-flow (immersion, CODI, and spray CODS) conditions. Two temperature-sugar concentration conditions with different contact times were employed to create 24 different test conditions for each of the four methods to test the differences between them.

The process monitored changes in moisture content, weight reduction, and solids gain. The results showed, in general, that the microwave osmotic dehydration under continuous flow medium spray conditions (MWODS) considerably enhanced the moisture transfer rate from the fruit, leading to a significant increase of moisture loss. For example, at 50°C/50°Brix for 2 h, the moisture loss with MWODS was 34-94% higher than in other methods; whereas the solids gain in MWODS was 16-46% lower than with the other methods. Overall, MWODS was far more effective than similar techniques in enhancing moisture loss and simultaneously restricting the solids gain.

3.1 Introduction

Osmotic dehydration is a process of partial removal of water from moist cellular materials. This reduces physical, chemical, and biological changes during drying at higher temperatures (Torreggiani, 1993; Sereno et al., 2001b) without involving a phase change and therefore promotes energy savings (Raoult-Wack, 1994a; Lenart, 1996). Despite the advantages of osmotic dehydration, the industrial application faces several

limitations due to the difficulty in moving the viscous and dense solution, which also often causes the product to float. On the other hand, large solute uptake has a negative effect on the nutritional profile of the product (Lazarides et al., 1995; Lazarides et al., 1997) and causes additional resistance to moisture transfer (Matuska et al., 2006).

Osmotic dehydration is relatively slow and therefore the pre-treatment is time-consuming; in order to accelerate the mass transfer, a number of techniques such as pulsed vacuum (Ito et al., 2007), ultrasound (Rodrigues and Fernandes, 2007b), pulsed electrical field (Andres et al., 2007), high pressure (Rastogi and Niranjana, 1998), and microwave (MW) drying (Krokida and Maroulis, 1997; Krokida et al., 2000c) have been employed. It has been the focus of many studies to enhance one kind of mass transfer (moisture loss) and retard the other (solids gain) (Azuara et al., 1992). In (2006c), Li and Ramaswamy achieved this by carrying out osmotic dehydration in a microwave environment under continuous-flow immersion-mode heating conditions.

Microwave drying has the specific advantage of rapid and uniform heating due to the penetration of microwaves into the body of the product (Bilbao-Sainz et al., 2006; Alibas, 2007). The most important characteristic of microwave heating is volumetric heating, which refers to the material absorbing microwave energy directly and internally and converting it into heat. Therefore, heat is generated throughout the material, leading to faster heating rates (compared to conventional heating, where heat is usually transferred from the surface to the interior) and producing rapid and uniform heating (Beaudry et al., 2004; Gowen et al., 2006). Microwave heating (MW), causing a positive out-flux of moisture from the product, not only results in greater moisture loss but also a lower solids gain. Immersion of the fruits in syrup in the MWODI mode limits the exposure of fruits to the MW field because of the surrounding syrup. However, in the MWODS mode, the same treatment provides a more direct exposure of the fruit to MW because as the continuous spray trickles down the fruit bed, it only retains a thin layer of the syrup at the fruit surface. It is interesting to note that applying spray can also overcome one of the problems with osmotic dehydration- the floating of the fruit in the solution.

In this study, microwave-osmotic dehydration was focused on a continuous-spray-medium contacting. To date, there is no published information on the effect of microwave-osmotic dehydration process under continuous-flow medium-spray conditions. The purpose of this work was to evaluate the performance of a microwave osmotic dehydration under continuous flow medium spray condition (MWODS) relative to other similar methods for achieving rapid moisture loss, limiting solids gain, and enhancing weight reduction.

3.2 Materials and Methods

3.2.1 Materials

Apples (*Red Gala*) of uniform size and ripeness were bought from the local supermarket. The fruits were refrigerated at 2-5°C and 95% relative humidity until use. Commercial sucrose (Redpath Canada Ltd., Montreal, QC) was used as the osmotic agent. After cutting calyx and pedicel ends, apple cylindroids were cut vertical to their axes, and cylinders 14 mm in diameter, 14 mm in height were prepared.

3.2.2 Microwave osmotic dehydration set-up

The experimental set-up designed to carry out the microwave osmotic dehydration is a logical alternative to that previously established by Li and Ramaswamy (2006c), for microwave osmotic dehydration under continuous flow medium immersion. Essentially the technique replaces the osmotic immersion unit with a spray unit. It consisted of a microwave transparent chamber (made out of glass) placed inside a domestic microwave oven (Danby DMW1153BL 0.031 m³, Guelph, ON, Canada; Figure 3.1) with a nominal output of 1100 W at 2450 MHz. A load test with 1 L of water, heated for 5 min, indicated that the absorbed power was in the 90-95% range. A commercial spray device 12 cm in diameter (Waterpik, CF-151-S, Canada Waterpik Technology Inc., Markham, ON) was attached at the top of the chamber to continuously spray the osmotic medium on apple samples placed inside the chamber. Test samples were held together using a thin nylon mesh for easy removal

from the chamber. A sucrose solution pre-heated to a selected temperature was pumped to the spraying device; after contacting the samples, the solution was pulled out from the bottom of the chamber using a peristaltic pump (75-211-30, Barnant Co., Barrington, IA, USA). The syrup was returned to the shower through a long copper coil placed in a steam-heated temperature-controlled water bath (Model TDB= 4, Groen Division/Dover Crop, Elk Grove Village, IL, USA). The temperature of the water bath was adjusted to the desired inlet temperature of the syrup leading to the spray device and monitored continuously immediately adjacent to the outside wall of the microwave oven. The temperature was also monitored at the exit in a similar manner. The syrup circulation system was a continuous loop from the bottom of the chamber up to the spray device. The loop was broken only during the spray, but the entire syrup was continuously recirculated, after temperature equilibration in the water bath. The flow rate was maintained at 2.80 L/min using a peristaltic pump. This flow rate level was previously determined to be sufficiently high to eliminate the influence of flow rate on mass transfer. The coil in the steam chamber was sufficiently long to provide a syrup-to-fruit ratio of over 30:1. The nearly closed loop for the medium flow prevented evaporation of water from the syrup and the large solution-to-fruit ratio allowed for maintaining a steady syrup concentration. The small amount of vapor lost during the spray treatment in the microwave chamber was nearly compensated for by the dilution of the syrup by the moisture picked up from the fruit because the solute concentration in the syrup nearly remained constant during the test runs. The rapid flow rate of the syrup also helped to prevent a large temperature change in the syrup during microwave heating. The sample and syrup load in the chamber was approximately 500 g, at which approximately 50% MW power was expected to be absorbed. The temperature difference between the osmotic solutions going in and out of the microwave oven was about 3°C, which would also account for 50-55% absorption of the microwave power ignoring the heat losses.

The same microwave-osmotic dehydration under continuous flow medium spray (MWODS) conditions was also used for simulating conventional osmotic dehydration under continuous flow medium spray (CODS) by simply turning the

microwave oven off during the run. For the microwave osmotic dehydration under medium-immersion mode (MWODI), the osmotic chamber was filled with the syrup to keep the samples fully immersed during the run; for its conventional counterpart (CODI), the MWODI set-up was run without microwave heating.

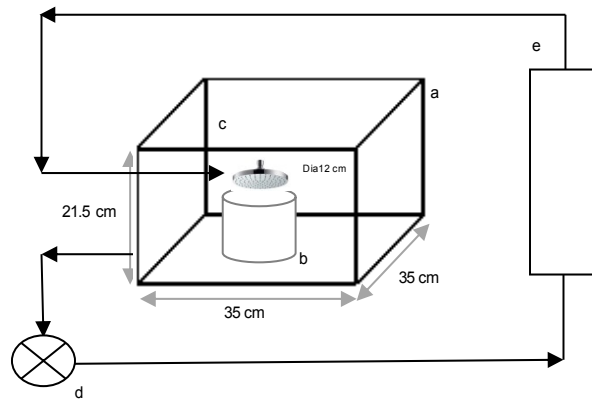
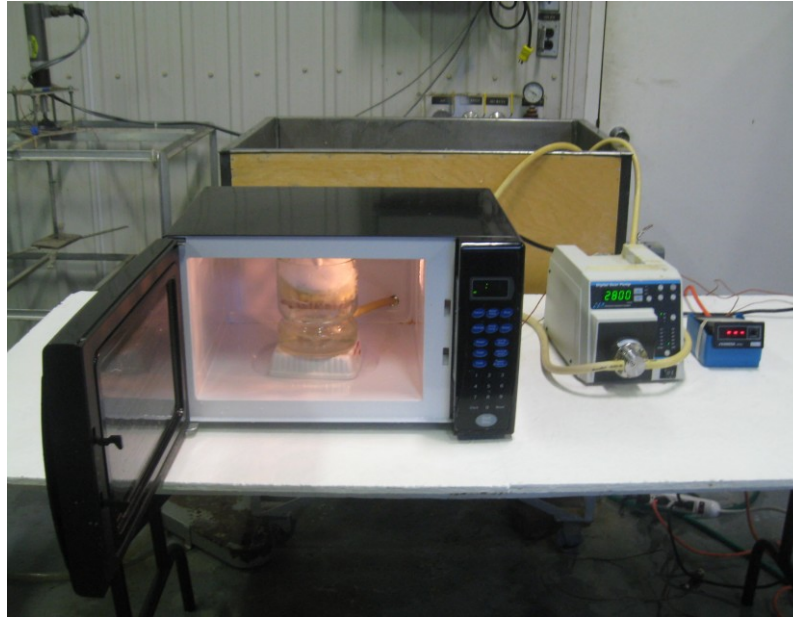


Figure 3.1 Experimental set-up for microwave osmotic drying under continuous spray mode Conditions (MWODS) (a: microwave oven; b: transparent chamber; c: spray device; d: pump; e: water bath)

3.2.3 Treatment procedure

Osmotic dehydration experiments were carried out in triplicate using all four methods: microwave osmotic dehydration under continuous flow medium spray (MWODS) and medium immersion (MWODI) conditions; conventional osmotic dehydration under continuous flow medium spray (CODS) and medium immersion (CODI) conditions. For each test run, pre-weighed apple cylinders were placed in the nylon mesh and loosely tied in a bag and placed in the osmotic chamber (total load =100 g). Over a 2-h osmotic dehydration process, replicate samples were taken after 30, 60, 90, and 120 min (each constituting a separate run), rinsed with water for a few seconds to remove adhering osmotic solution, gently blotted with a wet paper towel, and weighed. Because the primary objective of this article was to compare the different methods, the design was kept simple, employing two osmotic conditions (50°C/50°Brix and 40°C/40°Brix) for each of the four methods. Four treatment times were used and experiments were carried out in triplicate. In osmotic experiments, each testing condition is a different run, meaning that there were 96 test runs (4 methods $\times$ 4 treatment times $\times$ 2 osmotic conditions $\times$ 3 replicates). Each test required independent preparation of test samples and running the system to achieve equilibrated conditions for temperature and required a minimum 4 h.

3.2.4 Dehydration kinetics parameters

To compare the performance of the four osmotic dehydration procedures, common osmotic dehydration parameters like transient moisture loss, solids gain, and weight loss were computed. Moisture content and soluble solids content (sucrose) were determined in triplicate as follows: Samples were weighed and placed in an oven set at 105°C for approximately 24 h until a constant weight was reached (AOAC, 2000). The sucrose concentration was measured with a portable refractometer (ATAGO Co., Tokyo, Japan) at 20°C. The moisture loss (ML %), weight reduction (WR %), and solids gain (SG %) were determined from the following equations: (Li and Ramaswamy, 2006c).

$$\%ML = 100 \frac{(M_0 x_0 - M_t x_t)}{M_0} \quad (3.1)$$

$$\%WR = 100 \frac{(M_0 - M_t)}{M_0} \quad (3.2)$$

$$\%SG = 100 \frac{(M_0 s_0 - M_t s_t)}{M_0} \quad (3.3)$$

where M_0 and M_t are the sample mass (g) at time 0 and time t ; x_0 and x_t are the moisture fractions (kg/kg wet basis) at time 0 and time t ; s_0 and s_t are the solids fractions (kg/kg wet basis) at time 0 and time t . These equations are based on the assumption that no solids leaked into the solution. Moisture loss-to-solids gain ratio was used as an additional criterion because one of the purposes of optimizing osmotic dehydration is to promote better moisture gain and limit the solute gain from the syrup. Therefore, conditions with a higher ML/SG ratio are preferred. In addition, a combined process parameter, osmotic dehydration time to achieve target moisture loss, solids gain, or weight loss, was also used to compare the different methods.

3.3 Results and Discussion

3.3.1 Comparison of different methods for moisture loss

Figure 3.2(a) shows the transient moisture loss in apple samples under the different conditions of up to 2 h. throughout the treatment, the moisture loss with MWOD was consistently higher than with other methods. The four different methods could be clearly differentiated. The most effective one was MWODS followed by MWODI, CODS, and CODI, thus clearly demonstrating the superiority of MW modes over conventional modes of osmotic drying; further, in both MW and conventional systems, the spray mode proved superior to the immersion mode. Also easily demonstrated was the commonly accepted notion that higher concentrations and temperatures were more conducive to rapid moisture loss than the lower temperature-concentration combinations. Starting with a 25-35% ML within 30 min, the total moisture loss increased to 61% in the MWODS mode in 2 h (up to 73% in 3 h, not

shown). The MWODI came slightly behind with 20-25% ML in 30 min, increasing to 45% ML after 2 h. Compared to these, the ML in the conventional methods was only 10-15% during the first 30 min. Even after a 2-h treatment, the total loss in moisture was only in the 20-25% range. It is also clear that the rate of moisture loss was higher during the first 30 min, indicating the usual moderation of the osmotic diffusion-controlled moisture loss with the progress of time. Moisture loss is more prominent during the early phase of the osmotic treatment due to the existing large osmotic driving force (the gradient) between the fresh fruit and hypertonic solution. Water movement becomes more difficult during the later stages because of the accumulation of sucrose along the surface of the fruit as well as the lower osmotic difference. Similar results have been reported by other authors (Raoult-Wack et al., 1991; Souza et al., 2007).

Specific performance details of different methods with respect to moisture loss at each treatment time are compared in Table 3.1 for two of the different processing conditions. This is given as percentage increase in moisture loss associated with MWODS in relation to each of the other three methods. Compared with MWODI, the MWODS was superior by 12-31% at 40°C/40°Brix and 34-36% at 50°C/50°Brix treatment conditions, demonstrating that the spray system was better than the immersion system. With CODS, the improvement in moisture loss ranged from 56 to 71% at 40°C/40°Brix and 78 to 121% 50°C/50°Brix. These were further extended to 94-188% in comparison with CODI. Thus, the moisture loss with MWODS was considerably higher than with other methods under similar processing conditions. Thus, under each and every test condition, the moisture loss in MWODS was higher than in other methods. A t-Test comparison confirmed the significance of these differences Table 3.2(a). The t-values are also indicators of the degree of difference (all very highly significant) between the different methods; for example, the ML was highest in MWODS, followed next by MWODI and subsequently by CODS and CODI. In addition, the microwave mode was superior to the conventional mode in both spray and immersion modes, and the spray mode was better than immersion mode in both MW and conventional systems. Clearly, the microwave treatment was beneficial in speeding

up the moisture diffusion process. This is in agreement with Li and Ramaswamy (2006c). It is clear that the overall moisture loss was highly promoted by MWODS.

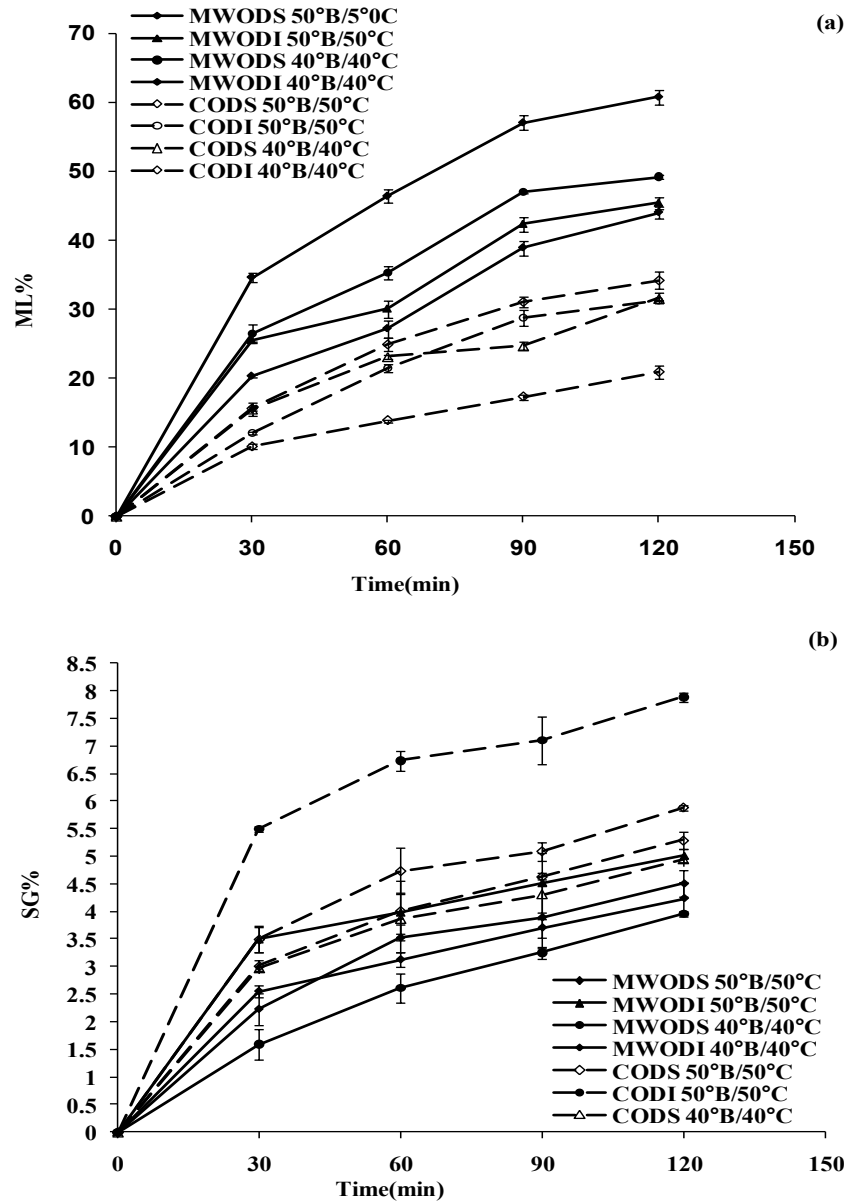


Figure 3.2 Comparison of (a) moisture loss (%ML) and (b) solids gain (%SG) under different conditions: microwave osmotic drying under spray (MWODS) and immersion (MWODI) modes and conventional osmotic drying under spray (CODS) and immersion (CODI) modes at two concentration and temperature combinations (40°B/40°C and 50°B/50°C)

Table 3.1 Percentage increase in moisture loss (ML) and percentage decrease in solids gain (SG) in MWODS relative to other methods after different osmotic treatments

Osmotic drying conditions	MWODS vs MWODI			MWODS vs CODS		MWODS vs CODI	
	Treatment time min	% Increase in ML	% Decrease in SG	% Increase in ML	% Decrease in SG	% Increase in ML	% Decrease in SG
40°C/40°Brix	30	31.3±1.91	28.5±2.81	71.2±2.15	47.3±1.81	162±1.08	47.3±1.12
	60	30.2±2.41	25.7±0.80	53.3±0.03	31.5±1.41	154±1.46	35.2±1.14
	90	20.6±0.99	17.4±2.12	83.4±0.05	24.4±2.11	171±1.64	70.2±2.34
	120	11.8±1.11	11.6±1.56	56.3±0.03	20.4±1.18	136±2.78	25.1±1.35
50°C/50°Brix	30	36.8±1.22	26.6±1.11	121±2.11	16.5±1.17	188±1.10	53.5±1.31
	60	53.6±2.79	21.6±2.90	86.3±2.6	24.5±2.16	116±2.19	53.3±2.37
	90	35.4±1.51	19.4±1.33	83.4±1.6	28.5±2.25	98±2.36	49.2±2.88
	120	33.6±1.22	15.1±2.02	78.3±1.5	28.4±1.49	94±1.97	46.1±2.24

Table 3.2 t-Test results for significance of differences in (a) moisture loss and (b) solids gain between different methods of osmotic drying

Difference	DF	t-Value	Pr > t
(a) Moisture loss			
MWODS-MWODI	23	11.72	<0.0001
MWODS-CODS	23	17.16	<0.0001
MWODS-CODI	23	26.76	<0.0001
MWODI-CODS	23	11.49	<0.0001
MWODI-CODI	23	14.57	<0.0001
(b) Solids gain			
MWODS-MWODI	23	-10.11	<0.0001
MWODS-CODS	23	-17.33	<0.0001
MWODS-CODI	23	-10.92	<0.0001
MWODI-CODS	23	-6.04	<0.0001
MWODI-CODI	23	-7.54	<0.0001
CODS-CODI	23	-5.73	<0.0001

Li and Ramaswamy (2006c) found that under MWODI at 40°C/40°Brix, the moisture loss after 2 h was 25% (a significant improvement over the conventional counterparts). In the current set-up, which is similar to that used in the previous studies, a much higher moisture loss of 44% was achieved even in the MWODI mode because

of the improvement with medium-flow rates. The spray mode (MWODS) extended the moisture loss further to 50%. These differences were further extended when higher temperatures, higher concentration, and longer treatment times were employed. The higher moisture loss in the microwave mode compared to the conventional could be explained by the microwave heating effect. Water is the most important dipole in the system (both fruit and syrup considered) and therefore will be the primary recipient of the microwave energy. The rapid heating of water molecules can be expected to create an internal pressure, promoting faster movement of water-based osmotic currents and leading to more efficient removal of moisture from the fruit to the syrup. The better performance of MWODS over MWODI is likely caused by two factors acting in synergy. First, the fruit will absorb microwaves much more than the syrup because of its higher moisture content. Second, in the immersion mode the fruit is totally submerged in the syrup and the MW absorption is rather limited. On the other hand, in the spray mode, only a thin layer of the syrup covers the surface of the fruit and hence the fruit will have direct exposure to the microwave field. Third, the spray mode is more efficient in removing the expelled moisture from the fruits than the immersion mode due to better draining ability.

As expected, in all cases moisture loss was enhanced by increasing temperatures (Lenart and Flink, 1984a; Raoult-Wack et al., 1991) and concentrations of sucrose (Table 3.1, Figure 3.2(a)) (Lazarides et al., 1997; Li and Ramaswamy, 2006c). So, all methods followed conventional osmotic dehydration trends. The osmotic pressure gradient increases with increasing temperature and solution concentration. These treatment situations cause the cell membranes to swell and plasticize, thereby causing them to be more permeable to water coming out of the product. Higher temperatures also result in lowering of viscosity of the osmotic medium, which tends to improve the mass transfer characteristics at the product surface (Contreras and Smyrl, 1981; Souza et al., 2007).

3.3.2 Comparison of methods for solids gain

The transient solids gains under different conditions using the four methods are shown in Figure 3.2(b). The solids gain with MWOD (both spray and immersion) was between 2.5 and 3% in the first 30 min and gradually increased somewhat linearly to about 3-4.5% after 120 min of osmotic dehydration. Clearly, the other methods had much more solids gain than the MWODS. During the study, unlike moisture loss, the solids gain did not show a clear trend with increasing sucrose concentration and temperature. This is in line with the findings on osmotic dehydration in reported studies (Lazarides et al., 1995; Khin et al., 2007) which indicated that higher process temperatures seem to promote faster water loss through swelling and plasticizing of cell membranes and that this results in increased solids gain. It is also true, however, that under conditions that promote higher moisture loss, the solids gain can be suppressed (Li and Ramaswamy, 2006b). This is characteristically demonstrated in the present study. Osmotic dehydration is based on the selective permeability of semi-permeable cell membranes, so any disruption of the cells will result in poor osmosis, possibly resulting in higher solids uptake and lower water removal. These results showed that the MWOD technique is as efficient as normal osmotic dehydration. The internal aqueous pressure caused by the selective absorption of microwave energy by water molecules in the fruit tissue tends to result in massive out-fluxes of moisture from the fruit counteracting the solids uptake by the fruit. Conditions favoring rapid moisture loss therefore result in a simultaneous reduction in the solids gain (Trelea et al., 1997; Li and Ramaswamy, 2006c).

The relative performances of MWODS vs. other methods in reducing the solids gain under osmotic treatment conditions are summarized in Table 3.1. The results in general demonstrated a reverse trend with respect to solids compared with the previously described moisture loss behavior. Conditions that gave the maximum moisture loss resulted in a lower solids gain. This was primarily so with the microwave osmotic drying under spray mode (MWODS). Relative to MWODI, the solids gain under MWODS (50°C/50°Brix) was 12-29% lower at 40°C/40°Brix and 15-27% lower

at 50°C/50°Brix. Relative to CODS and CODI, these were 17-28% lower at 40°C/40°Brix and 46-54% lower at 50°C/50°Brix. As with moisture loss, under each test condition the solids gain in MWODS was lower than in other methods. Again, the t-test (Table 3.2(b)) comparison confirmed the significance of these differences. These results also confirmed that MWOD methods resulted in a significantly lower solids gain than other osmotic drying methods under comparable operating conditions (Li and Ramaswamy, 2006b).

3.3.3 ML/SG ratio

The ratio of moisture loss/solids gain (ML/SG) is an important indicator for optimization of the osmotic dehydration process (Lazarides et al., 1997; van Nieuwenhuijzen et al., 2001; Li and Ramaswamy, 2006a,b,c; Matuska et al., 2006). It is desirable to maximize moisture loss and minimize solids gain. The values reported in Figure 3.3(a) indicate that the ratios of moisture loss to solids gain for apples were consistently higher under MWOD than under COD, and the spray mode resulted in a relatively higher ratio than the immersion mode. The MWODS gave the best ML/SG ratio among all modes and the ML/SG ratio was more than double relative to conventional osmotic drying conditions. These results demonstrate the superiority of MWODS over the other techniques. Earlier, Li and Ramaswamy (2006c) had demonstrated that MWODI was superior to conventional osmotic drying with respect to ML/SG ratio.

3.3.4 Weight reduction

The overall weight reduction comes from the moisture loss but is moderated as a result of the solids gain. Weight reductions under the four osmotic drying conditions are shown in Figure 3.3(b). The weight reductions were noticeably different for the four methods, with a distinctly higher value under MWODS. Similar to moisture loss, higher temperature and concentration conditions resulted in a higher weight reduction. MWODS was better than MWODI, MWOD was better than COD, and spray mode was better than immersion mode with respect to weight reduction. These results largely

confirm results from previous studies using similar systems (Van Nieuwenhuijzen et al., 2001; Li and Ramaswamy, 2006a,b, c). The new finding is that MWODS is the best performer among the four methods for achieving weight reduction in apple samples.

3.3.5 Dehydration time

To compare the effectiveness of different osmotic drying conditions, one more parameter was used that gives the cumulative time effect needed to reach moisture loss, solids gain, or weight reduction. The targets were chosen to be treatment times to achieve a 25% sample moisture loss (T_m), 20% weight reduction (T_w), and 3% solids gain (T_s) so that the treatment times were generally within the experimental domain for all conditions tested (Table 3.3).

In general, T_m and T_w showed similar trends: they decreased with increasing temperatures and sucrose concentrations. The shortest times needed to reach the target moisture loss were under MWODS, 23 min at 50°C/50°Brix and 33 min at 40°C/40°Brix treatment conditions; and the longest T_m were with CODI, 78 min at 50°C/50°Brix and 200 min at 40°C/40°Brix medium. The shortest T_w were also observed under MWODS, 20 and 24 min at 50°C/50°Brix and 40°C/40°Brix, respectively; and the longest T_w were 200 and 102 min under CODI under the same conditions. The order for both T_m and T_w was MWODS, MWODI, CODS, and CODI. The times to reach 3% solids gain under different condition (T_s) were 63 and 95 min under MWODS at 50°C/50°Brix and 40°C/40°Brix, respectively; under CODI they were 14 and 30 min under the same conditions. Because MWOD treatments would not be used for more than 60 min, the 4% SG level would never be reached in these situations, whereas conventional techniques are likely to be exposed to conditions that would exceed 4% solids gain. Li and Ramaswamy (2006c) found similar T_m and T_w values for MWODI. With a better performance than MWODI, the MWODS technique offers a better choice for MWOD treatment.

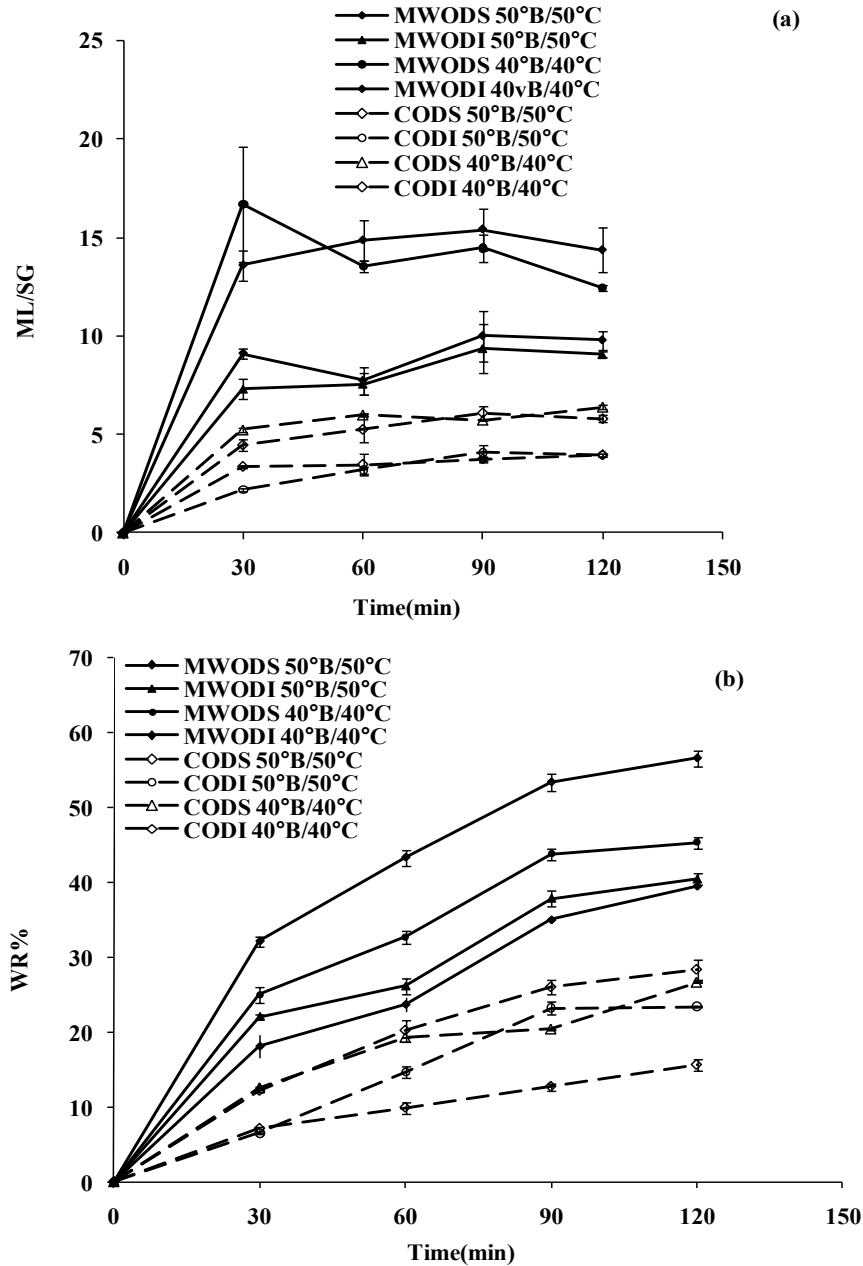


Figure 3.3 Comparison of (a) ML=SG and (b) weight reduction (%WR) under different conditions: microwave osmotic drying under spray (MWODS) and immersion (MWODI) modes and conventional osmotic drying under spray (CODS) and immersion (CODI) modes at two concentration and temperature combinations (40°B/40°C and 50°B/50°C)

Table 3.3 Relative times to achieve 25% moisture loss, 20% weight reduction, or 3% solids gain under different osmotic dehydration conditions

Conditions	Methods	25% ML Time	3% SG Time	20% WR Time
		(min)	(min)	(min)
40°C/40°Brix	MWODS	33.3±0.615	95.2±0.553	24.4±0.386
	MWODI	49.4±0.677	48.1±0.221	42.2±0.286
	CODS	75.3±0.285	24.8±0.392	72.0±0.205
	CODI	200±1.83	29.6±0.313	210±0.331
50°C/50°Brix	MWODS	22.5±0.227	62.6±0.330	20.2±0.281
	MWODI	40.1±0.201	23.1±0.293	27.3±0.167
	CODS	63.4±0.177	22.2±0.293	61.3±0.385
	CODI	78.2±0.329	13.5±0.236	102±0.196

3.4 Conclusions

Microwave osmotic dehydration under a continuous flow medium spray condition was designed and compared with three other methods: MWOD under immersion mode and conventional osmotic dehydration with both immersion and spray mode treatments, all intended to enhance the mass transfer rate during the process. Distinct differences were observed among the four methods with respect to moisture loss, weight reduction, solids gain, ML/SG ratio, and dehydration to achieve a target moisture loss, weight reduction, or solids gain. The highest moisture loss, highest weight reduction, highest ML/SG ratio, lowest solids gain, and shortest dehydration times were found with MWODS. Therefore, MWODS has a distinct advantage over the other systems and offers great potential as a novel osmotic drying pre-treatment method.

CONNECTIVE STATEMENT TO CHAPTER 4

In Chapter 3, the development of microwave-osmotic dehydration under spray medium flow (MWODS) was highlighted and the method was shown to be superior to other existing methods. This study has focused on the modeling of the mass transfer kinetics of apple cylinder under MWODS, especially to verify if the two common models - Azuara model and Fick's second law can be used as effectively for this technique as with other existing methods (MWODI, CODS and CODI). This was done essentially to demonstrate that the new method is qualitatively similar to other OD methods, however more efficient in terms of moisture loss and weight reduction as well as effective in limiting the solids gain.

Part of the results from this study has been presented at a scientific conference:

Azarpazhooh, E and Ramaswamy, HS. 2009. Mass transfer kinetics of apples in microwave-osmotic dehydration under continuous spray medium flow conditions. Annual meeting of Institute of Food Technologists, June 6-10, Anaheim, USA. (Poster and oral presentation for student competition).

One manuscript has been published:

Azarpazhooh, E and Ramaswamy, HS. 2010. Evaluation of Diffusion and Azuara Models for Mass Transfer Kinetics during Microwave-Osmotic Dehydration of Apples under Continuous Flow Medium Spray Conditions. *Drying Technology*, 28(1): 57-67.

This research work was completed by the candidate under the supervision of Dr. HS. Ramaswamy.

CHAPTER 4. EVALUATION OF DIFFUSION AND AZUARA MODELS FOR MASS TRANSFER KINETICS DURING MICROWAVE OSMOTIC DEHYDRATION OF APPLES UNDER CONTINUOUS FLOW MEDIUM SPRAY CONDITIONS

Abstract

Azuara and diffusion models were evaluated for describing the mass transfer kinetics of apple (*Red Gala*) cylinders during microwave osmotic dehydration under continuous flow medium immersion (MWODI) and medium spray (MWODS) conditions as well as conventional osmotic dehydration under continuous flow medium immersion (CODI) and medium spray (CODS) conditions without the microwave heating. Two different sets of experiments were carried out with all four methods. In the first set, osmotic treatments were given at 50°C/50°Brix and 40°C/40°Brix with a solution flow rate of 2800 mL/min and fruit-to-solution ratio of 1:30. The treatment times ranged from 0 to 120 min. In the second set, the MWODS was extended to other conditions (40°C/50°Brix and 50°C/40°Brix). The equilibrium moisture loss and equilibrium solid gains required for the diffusion model were predicted using the Azuara model. Both models well fitted the experimental data for mass transfer kinetics ($R^2 > 0.92$). Higher equilibrium moisture loss and lower solids gain were observed in samples treated with MWODS compared with other methods. The equilibrium moisture loss and solids gain under MWODS were related to solution concentration and solution temperature. The diffusion coefficients representing moisture loss (D_m) and solids gain (D_s) were computed from the diffusion model. The D_m values were higher and D_s values were lower with MWODS as compared to the other methods. D_m and D_s were dependent on temperature and concentration of the osmotic solution. Half-drying times for moisture loss and solids gain were also computed to compare the different methods. These were inversely related to diffusivity values. Overall, the highest moisture loss and the lowest solids gain were observed in MWODS.

4.1 Introduction

Osmotic dehydration is a technique for partial removal of water by direct immersion of food pieces in hypertonic solutions. The food cellular surface structure acts as a semi-permeable membrane and sets up a driving force for water transport due to a difference in the osmotic pressure between food and its surrounding solution. A simultaneous counter diffusion of solute from the osmotic solution also accompanies the outward diffusion of water. Because the membrane is not completely selective, leaching of natural solutes from the food also occurs simultaneously (Rastogi et al., 1997; Spiazzi and Mascheroni, 1997).

Considerable effort has been made toward developing models to predict the mass transfer kinetics of osmotic dehydration. The best known phenomenological model for osmotic dehydration (OD) processes at atmospheric pressures is Crank's model, which consists of a solution to the non-steady Fick's law and represents the diffusional mechanism. Several studies have focused on the modeling of mass transfer kinetics during osmotic dehydration. The diffusion model (Lazarides et al., 1997; Rastogi et al., 2000a) based on Fick's second law is perhaps the most frequently used model for the mass transfer kinetics during osmotic dehydration and generally assumes the external resistance to mass transfer to be negligible compared to the internal resistance. The model is also used to evaluate the mass diffusivities for both moisture loss and solids gain. However, comparison of mass diffusivities during osmotic dehydration from different studies is generally difficult because of variations in food composition and physical structure as well as the different methods and models employed to estimate diffusivity (Zogzas and Maroulis, 1996). These diffusion models also have a number of assumptions that are difficult to fulfill and the effective diffusivity becomes an adjustable kinetic parameter that depends strongly on the experimental conditions and the physical properties of the fruit. Nevertheless, authors have used these solutions (Lazarides et al., 1997; Rastogi et al., 2000a) to correlate experimental data in osmotic dehydration.

There are two parameters required in the diffusion model for both moisture loss and solids gain: the effective diffusion coefficient and the equilibrium values. The effective diffusion coefficient can be obtained by finding numerical or analytical solutions to experimental data, (Nguyen et al., 2006) calculating the relationship between the slope of the theoretical diffusion curve and the slope of the experimental mass transfer ratio (Rastogi et al., 2000a, Ade-Omowaye et al., 2002; Amami et al., 2005), or applying linear and nonlinear regressions (Akpinar, 2006). It is common in the literature to consider any finite food geometry as an infinite flat plate configuration, generally neglecting two- and three-dimensional diffusion in finite objects; only a few of these studies have considered unsteady-state mass transfer during osmotic dehydration (Escriche et al., 2000; Ade-Omowaye et al., 2002; Roberts et al., 2002; Mayor et al., 2007). In all these models, it is necessary to find the equilibrium values for moisture loss and solids gain (Azuara et al., 1998).

Azuara et al., 1992 developed an empirical model based on the mass balance of water and sugar to predict the kinetics of moisture loss and solids gain during osmotic dehydration. Ochoa-Martinez et al., 2007b reported that the use of Azuara's model to predict mass transfer in osmotic dehydration of fruits at atmospheric pressure should be favored relative to Page's, Magee's, and Crank's models because Azuara's model not only fits the SG data better but turns out to be good enough for fitting ML data. Further, Azuara's model has the advantage of allowing better calculation of the equilibrium values of moisture loss and solids gain (ML_e and SG_e).

Microwave osmotic dehydration is a novel technique with a good potential for more efficient osmotic drying of fruits and vegetables. Carrying out osmotic drying in a microwave environment enhances moisture removal when high moisture food is immersed in a concentrated solution of an osmotic agent (Li and Ramaswamy, 2006c). The osmotic concentration gradient effect existing between the solution and food, which is the driving force for the removal of moisture from the food into the osmotic medium, is enhanced under the microwave field. This is due to selective absorption of microwave energy by the water molecules resulting in increased moisture out-flux,

which also has the tendency to limit the simultaneous transfer of solute from the solution into the food.

Li and Ramaswamy (2006c) investigated the mass transport coefficients under microwave osmotic dehydration (MWOD, immersion medium) and compared it with the conventional continuous flow osmotic dehydration process (COD). They reported that MWOD significantly increased the rate of moisture loss and decreased the rate of solids gain. They also found that the osmotic dehydration under microwave heating made it possible to obtain a higher diffusion rate of moisture transfer at lower solution temperatures. In their experiments, they immersed the apple slices in the osmotic solution placed within the microwave field. In such an immersion medium, because the sample is surrounded by a large volume of the solution, the absorption of microwave by the sample itself will be limited, thus reducing the moisture out-flux effectiveness of the microwaves.

In a previously published paper, (Azarpazhooh and Ramaswamy, 2010a), presented in chapter 3, microwave osmotic dehydration under continuous flow medium spray condition was developed and shown to provide a means of effecting moisture loss and limiting solids gain far superior to three other techniques under similar continuous-flow conditions. It was clearly demonstrated that the spray mode microwave heating enhanced the efficiency of the system. This is likely due to the direct and more efficient exposure of the sample to the microwave field. As opposed to the large volume of solution that surrounds the sample in the MWOD immersion system, the spray mode only places a thin layer of osmotic solution that is continuously flushed down with the rapidly flowing medium and gravity. The spray mode also eliminates the problem of sample floating, which can restrict the application of immersion mode.

The purpose of this study was to evaluate Azuara and diffusion models (both moisture loss and solids gain) during microwave-osmotic dehydration under a continuous-flow medium-spray heating conditions and compare it with those under MW immersion mode heating as well as their conventional counterparts without the

microwave heating. Modeling not only helps to predict and follow the transient changes in the moisture and solids during the osmotic dehydration but to optimize the dehydration process to maximize moisture loss while limiting the solids gain.

4.1.1 Theoretical considerations

4.1.1.1 Determination of moisture and solid equilibrium

Raoult-Wack (1994) suggested the two-parameter Azuara-kinetic model (Azuara et al., 1992) based on mass balance to estimate mass transfer coefficients and the final equilibrium point. This model has been reported to accurately predict the mass transfer dynamics of osmotic dehydration and the dynamic period solids gain kinetics (Salvatori and Alzamora, 2000; Azoubel and Murr, 2004). The proposed model for moisture loss and solids gain is shown by Eqs. (4.1) - (4.4):

$$ML_t = \frac{S_1 t (ML_e)}{1 + S_1 t} = \frac{t (ML_e)}{\frac{1}{S_1} + t} \quad (4.1)$$

$$\frac{t}{ML_t} = \frac{1}{S_1 (ML_e)} + \frac{t}{ML_e} \quad (4.2)$$

where ML_t moisture loss fraction at any time, t ; S_1 is a constant related to the rate of water diffusion out from product; and ML_e is moisture loss fraction at equilibrium. To determine the constant and solids gain at equilibrium during osmotic dehydration, similar equations can be used.

$$SG_t = \frac{S_2 t (SG_e)}{1 + S_2 t} = \frac{t (SG_e)}{\frac{1}{S_2} + t} \quad (4.3)$$

$$\frac{t}{SG_t} = \frac{1}{S_2 (SG_e)} + \frac{t}{SG_e} \quad (4.4)$$

where SG_t is the solids gain fraction at any time, t ; S_2 is a constant related to the rate of solids diffusion in the product; and SG_e is the solids gain fraction at equilibrium. The equilibrium moisture, ML_e , and solids contents, SG_e , can be obtained as the reciprocal slopes of t/ML_t and t/SG_t against reciprocal of time plots, respectively.

4.1.1.2 Determination of effective diffusion coefficients of water and solute

Fick's second law is generally used to model the mass transfer during osmotic dehydration (Lazarides et al., 1997; Mayor et al., 2007), which neglects the external mass transfer resistance to the internal resistance. In Fick's law of diffusion, a relationship between the flux of a component and the concentration gradient of that component exists, which is given below (Crank, 1975).

$$\frac{\partial C}{\partial t} = D \left(\frac{\partial^2 C}{\partial x^2} \right) \quad (4.5)$$

where C is the concentration of diffusing substance (kg/m^3), x is the space coordinates measured normal to the section (m), and D is the diffusion coefficient (m^2/s).

Analytical solutions to this equation for different shapes (spheres, infinite cylinders, and infinite slabs) as well as for semi-infinite and finite solids have been summarized by Crank (1975). For a spherical particle, Nsonzi and Ramaswamy (1998a) simplified the equations for moisture and solids transfer as:

$$\frac{M_e x_e - M_t x_t}{M_e x_e - M_0 x_0} = \frac{6}{\pi^2} e^{\left(\frac{-D_m \pi^2 t}{a^2} \right)} \quad (4.6)$$

$$\frac{M_e s_e - M_t s_t}{M_e s_e - M_0 s_0} = \frac{6}{\pi^2} e^{\left(\frac{-D_s \pi^2 t}{a^2} \right)} \quad (4.7)$$

where x_t and x_e are the water content at time t and equilibrium; M_0 , M_t , and M_e are the initial sample mass and those at time t and equilibrium; s_0 , s_t , and s_e are the initial solids

content and those at time t and equilibrium, respectively; and D is the diffusion coefficient (m^2/s).

Similar equations have been employed for other particle shapes. In this study, samples are cut into finite cylinders. Equations for finite cylinders can be obtained as a cross between equations for infinite cylinders and infinite plates. Based on heat-mass transfer analogy and equations given by Ramaswamy et al., (1982) for mass average temperature ratios, the following formulae were derived for the transient mass average moisture content in a finite cylinder (length being equal to diameter) (Ramaswamy and Van Nieuwenhuijzen, 2002).

$$M_{\text{mfc}} = 0.56e^{-\frac{8.25}{a^2}Dt} \quad (4.8)$$

where (M_{mfc}) is the unsteady mass concentration (mass average moisture ratio) in a finite cylinder. The transient moisture ratio (M_{mfcw}) in the finite cylinder ($a = r$) is defined as follows for water transfer:

$$M_{\text{mfcw}} = \frac{M_e x_e - M_t x_t}{M_e x_e - M_0 x_0} = \frac{ML_e - ML_t}{ML_e - ML_0} \quad (4.9)$$

where ML_e , ML_t , and ML_0 are the initial sample moisture loss and those at time t and equilibrium, respectively.

The transient solids gain ratio (M_{mfcs}) in a finite cylinder ($a = r$).

$$M_{\text{mfcs}} = \frac{M_e s_e - M_t s_t}{M_e s_e - M_0 s_0} = \frac{SG_e - SG_t}{SG_e - SG_0} \quad (4.10)$$

where SG_0 , SG_t , and SG_e are the initial sample moisture loss and those at time t and equilibrium, respectively.

The effective diffusion coefficients of water and solute, D_m and D_s (m^2/s), can be determined, respectively, from the slope of $\text{Ln} \frac{ML_e - ML_t}{ML_e - ML_0}$ and $\text{Ln} \frac{SG_e - SG_t}{SG_e - SG_0}$, against immersion time, t , for samples in the osmotic solution. The equilibrium moisture, ML_e , and solids gain contents, SG_0 , can also be inferred from the slopes of the plots of rate of change of moisture and solids content against mean moisture and solids content, respectively. The slope of Eq. (4.8) is equal to $-8.25 D/d$ (Rastogi and Raghavarao, 1997), with D representing the diffusivity coefficient of either moisture or solids.

4.1.1.3 Determination of half-drying time (Z)

The description of half-drying time with respect to moisture loss (Z_m) or solids gain (Z_s) is analogous to half-cooling time in Newton's law of cooling (Van Nieuwenhuijzen et al., 2001). The equations used for half-drying time are based on the assumption that the rate of moisture loss and solids gain during dehydration is directly proportional to the moisture in the apple and the sugar in the solution available for the dehydration process. This can be obtained either graphically or from regression slopes used for computing the D values:

$$\text{Ln} \frac{ML_e - ML_t}{ML_e - ML_0} \text{ and, vs. time for moisture } (Z_m) \text{ and } \text{Ln} \frac{SG_e - SG_t}{SG_e - SG_0} \text{ vs. time for}$$

solids (Z_s). At half-time (Z), both expressions $\text{Ln} \frac{SG_e - SG_t}{SG_e - SG_0}$ on the left hand side will reduce to 0.693 [i.e., $\ln(1/2)$]. On the right hand side, this will be $-8.25 Dt/a^2$ with t representing the half-time. Hence Z values can be computed from the evaluated diffusion coefficients taking in to account the product size. On the right-hand side, this will be $-8.25 DZ/a^2$ with Z representing time t at the half-time. Hence, Z values can be computed from the evaluated diffusion coefficients taking into account the product size.

$$z = \left(\frac{0.693}{8.25} \right) \left(\frac{a^2}{D} \right) \quad (4.11)$$

Alternatively, it can be found graphically from the plot of residual moisture ratio vs. time as the time interval between which the residual moisture ratio reduces by one half.

4.2 Material and Methods

4.2.1 Materials

Apples (*Red Gala*) of uniform size and ripeness, bought from a local supermarket, were used in the study. The fruits were stored and refrigerated at 2-5°C and 95% relative humidity until use. Commercial sucrose (Redpath Canada Ltd., Montreal, QC) was used as the osmotic agent. The initial moisture content of the fresh apples varied from 85 to 89% (wet basis). The fruits were washed with tap water and cylinders were punched out using a cork borer with a diameter of 14 mm and cut to a length of 14 mm. The weight of each cylinder was about 3 g.

4.2.2 Osmotic dehydration procedure

Information related to the methodology; general preparation of materials; details of microwave-osmotic dehydration setup with continuous-flow medium-spray (MWODS), medium-immersion (MWODI), similar conventional spray-osmotic drying in spray (CODS), and immersion (CODI) modes; treatment details; and data gathering and procedures used to compute ML and SG are detailed in chapter 3 and published paper (Azarpazhooh and Ramaswamy, 2010a). Apple cylinders were in 100 g pre-weighed batches, tied in a mesh bag, and placed in the osmotic dehydration chamber inside the microwave oven. The osmotic medium was a sucrose solution of 40°Brix at 40°C or 50°Brix at 50°C with a flow rate of 2800 mL/min. Solution-to-fruit ratio was maintained at 30:1 in order to minimize changes in the sucrose concentration during the osmotic dehydration. Osmotic dehydration was carried out in triplicate using each of

the four different methods: MWODS, MWODI, CODS, and CODI. In addition, experiments with MWODS included two other conditions: 40°Brix at 50°C and 50°Brix at 40°C to further study the effect of these variables on the mass transfer kinetics. Separate runs were carried out for each treatment time of 30, 60, 90, and 120 min. The samples were rinsed with distilled water, blotted with a paper towel, and weighed.

4.2.3 Diffusion coefficient (D) and half-drying time (Z)

The equilibrium moisture content and solids content of samples needed for calculating mass average moisture, residual moisture, and solids ratios were found from Eqs. (4.2) and (4.4), respectively. The diffusion coefficient associated with water loss and solids gain was obtained from Eqs. (4.8)-(4.10). The half drying time Z was computed using Eq. (4.11). Mass transfer kinetics was modeled using the empirical Azuara model and the traditional diffusion model based on Fick's law (detailed in the Theoretical Considerations section).

4.3 Results and Discussion

4.3.1 Equilibrium moisture loss and solids gain

These were obtained by fitting the ML% vs. t and SG% vs. t data to Azuara model. The fitted curves under different conditions are shown in Figure 4.1 for both (a) moisture loss and (b) solids gain. Table 4.1 shows the values of parameters S_1 , ML_e , S_2 , and SG_e . The equilibrium values were obtained as the reciprocal slopes of t/ML vs. t and t/SG vs. t plots for each osmotic drying condition and the intercepts were used to compute the second parameter. The high R^2 value (> 0.92) indicated the acceptability of the model and the computed equilibrium values. Other published works also indicate excellent results for the Azuara model and its usefulness in predicting the equilibrium values of moisture loss and solids gain (Waliszewski et al., 2002; Ochoa-Martinez et al., 2007a).

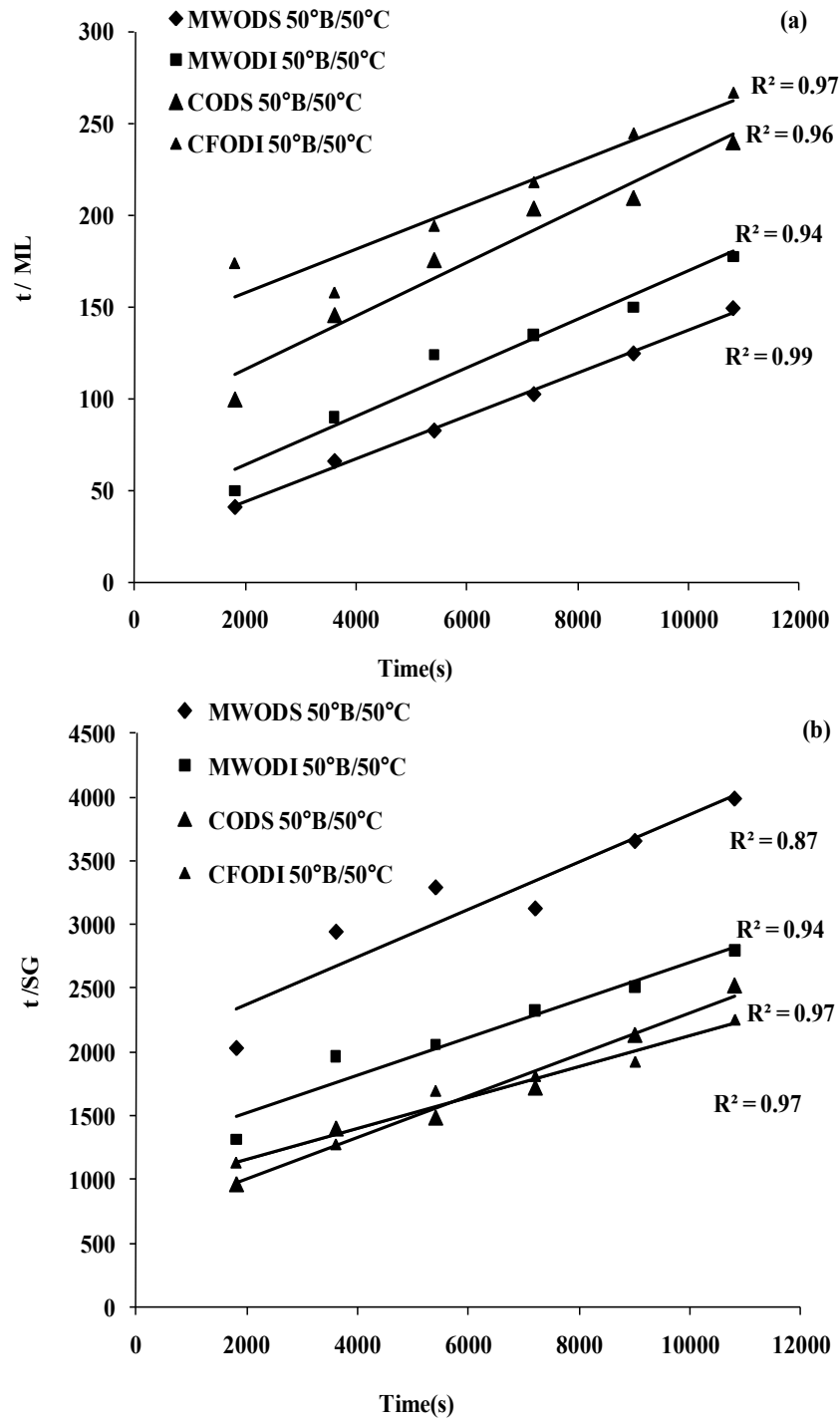


Figure 4.1 Linear plots of Azuara model for determination of ML (a) and SG (b) at 50°C/50°Brix for different methods.

Table 4.1 also compares the equilibrium values of moisture loss and solids gain under different osmotic solution concentrations and temperatures under the spray and immersion modes with and without the use of microwave heating. It can be observed that ML_e under each method increased with increasing concentration of osmotic solution and solution temperature. On the other hand, SG_e values showed the opposite trend. Similar results have been reported by Hawkes and Flink (1978) and Lazarides et al. (1995). MWODS showed the highest ML_e and lowest SG_e compared to the other methods, and in general osmotic solutions of higher concentration and higher temperature resulted in a higher ML_e and lower SG_e . The equilibrium values were dependent on not only the osmotic solution temperature and concentration but on the method because the methods have varying potential for achieving the equilibration.

Table 4.1 Azuara model parameters and equilibrium values for ML_e and SG_e during osmotic drying of apples at different conditions

Processing Conditions	Methods*	ML_{∞} (%)	$S_1 (\times 10^{-4})$ min^{-1}	R^2	SG_{∞} (%)	$S_2 (\times 10^{-4})$ min^{-1}	R^2
40°C/50°Brix	MWODS	72.50	3.6	0.99	5.59	3.2	0.97
40°C/40°Brix	MWODS	72.46	3.02	0.99	7.63	1.44	0.98
40°C/40°Brix	MWODI	77.76	1.76	0.98	6.56	2.94	0.98
40°C/40°Brix	CODS	46.08	2.72	0.98	6.25	4.64	0.98
40°C/40°Brix	CODI	32.57	2.27	0.96	7.04	5.27	0.99
50°C/40°Brix	MWODS	72.73	4.3	0.98	5.49	3.8	0.99
50°C/50°Brix	MWODS	83.03	3.8	0.99	5.43	4.26	0.97
50°C/50°Brix	MWODI	62.30	2.96	0.92	5.84	7.17	0.99
50°C/50°Brix	CODS	57.14	2.2	0.99	7.42	4.70	0.98
50°C/50°Brix	CODI	46.8	1.02	0.94	9.08	7.97	0.99

*MWODS: Microwave-Osmotic Dehydration under spray medium; MWODI: Microwave-Osmotic Dehydration under immersion medium; CODS: Conventional Osmotic Dehydration under spray medium; CODI: Conventional Osmotic Dehydration under immersion medium.

4.3.2 Azuara model for ML and SG

The two-parameter Azuara et al. (1992) was used not only for computing the equilibrium values but also for describing the mass transfer in osmotic dehydration of apple cylinder with different methods. Figure 4.2 shows the experimental vs. model

predicted transient moisture loss and solids gain under different conditions. All points were scattered evenly and tightly around the diagonal line, indicating an excellent model performance ($R^2 > 0.98$) and a good predictor of the ML and SG% for all four methods and under the different testing conditions.

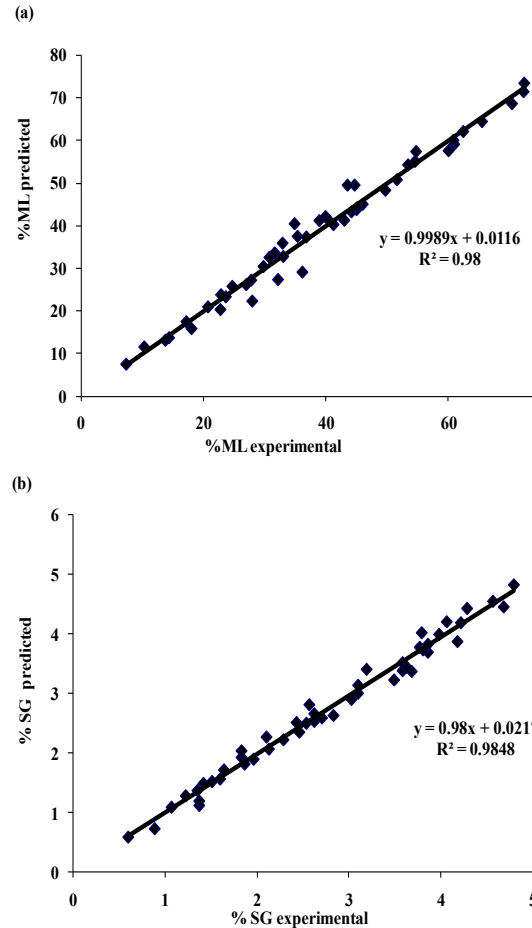


Figure 4.2 Performance of Azuara model (predicted vs. experimental) for (a) moisture loss (%ML) and (b) solids gain (%SG)

Figure 4.3(a-d) shows the trend curves for predicted moisture loss and solids gain based on the Azuara model with superimposed experimental values under MWODS, MWODI, CODS, and CDOI at 50°B/50°C treatment. The results and the smooth curves demonstrate the acceptability of the model for mass transport studies in dynamic period. As can be seen clearly in Figure 4.3(a), ML% was distinctly higher

with MWODS compared to the other methods. The moisture loss was also favored by an increase in solution concentration and process temperature (results are not shown), due to higher osmotic pressure at the product solution interface. A similar effect was verified in the osmotic dehydration of melon and pear (Park et al., 2002; Ferrari and Hubinger, 2008). Figure 4.3(b) shows similar trends but with opposing results, with solids gain indicating the MWODS to yield the least SG% compared to the other methods, which was demonstrated in a previous study (Azarpazhooh and Ramaswamy, 2010a). Figures 4.3(c,d) show the dynamic changes in ML% and SG% under different solution concentration-temperature combinations for MWODS of apples as predicted by the Azuara model. The model parameters were sensitive to the differences in the methods and processing conditions. The smooth and trendy curves, especially with ML%, show the excellent potential of Azuara model predictions right from the start. It is especially important to have a good prediction in the first hour or two because OD is mostly used as short pre-treatment prior to second-stage drying using one of the drying methods.

4.3.3 Moisture (D_m) and solids diffusivity (D_s)

The effective diffusion coefficients of moisture loss and solids gain, D_m and D_s , were determined from the slopes of residual moisture ratio and solids fraction vs. time (Eq. 4.8). The semi-logarithmic plots are shown in Figure 4.4 (a,b) for moisture loss and solids gain, demonstrating a fairly good fit of the data with fairly high R^2 values. The computed effective diffusivity D_m and D_s values from the slopes of the semi-logarithmic plots are detailed in Table 4.2. This diffusion model showed a good fit to experimental data with R^2 higher than 0.95.

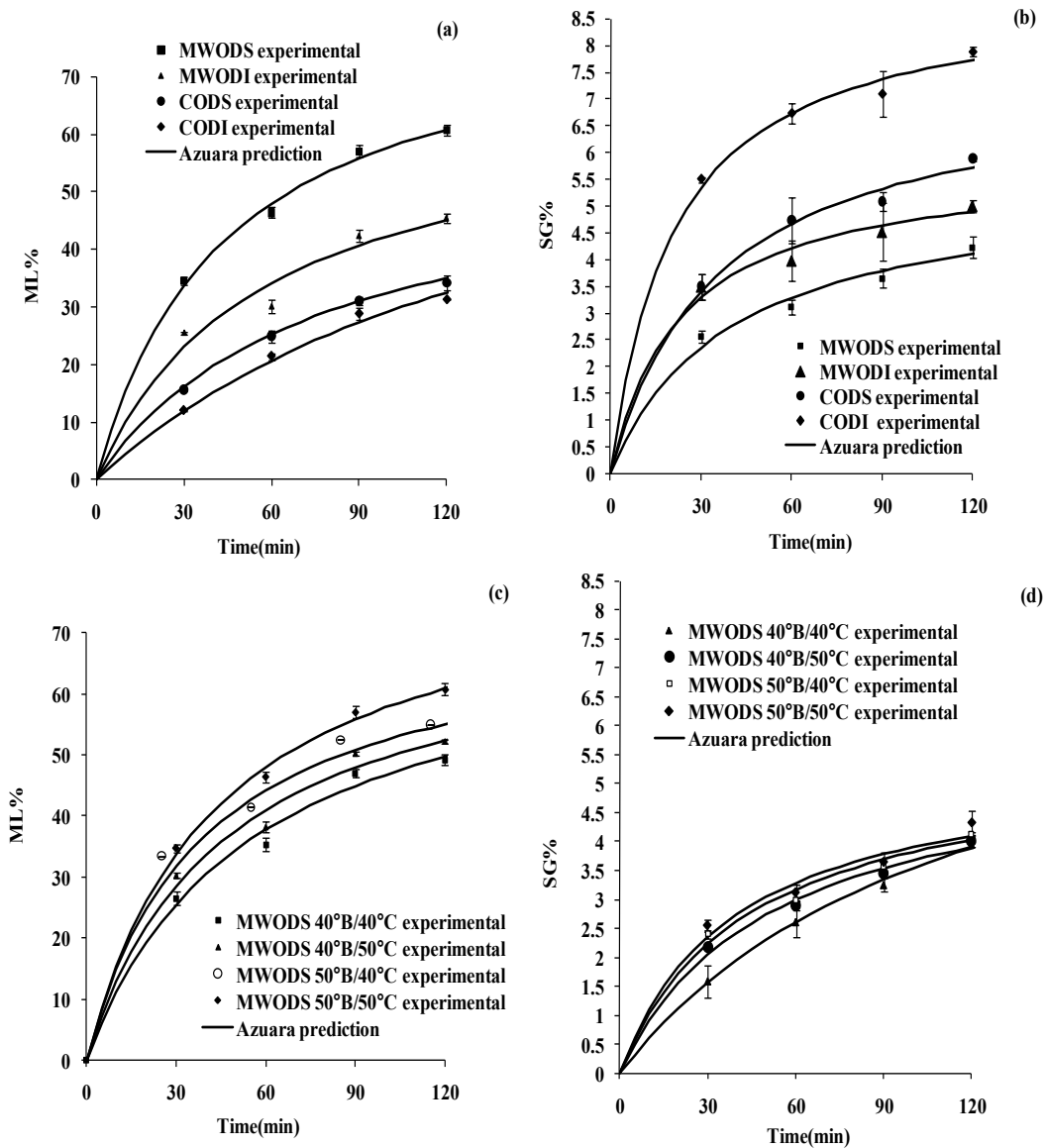


Figure 4.3 Azuara model prediction for transient moisture loss (a) and solids gain (b) with different methods at 50°C/50°Brix: Microwave-osmotic dehydration under medium-spray (MWODS) and medium-immersion (MWODI) and conventional osmotic dehydration under medium-spray (CODS) and medium-

immersion (CODI); moisture loss (c) and solids gain (d) with MWODS at different temperature and concentrations

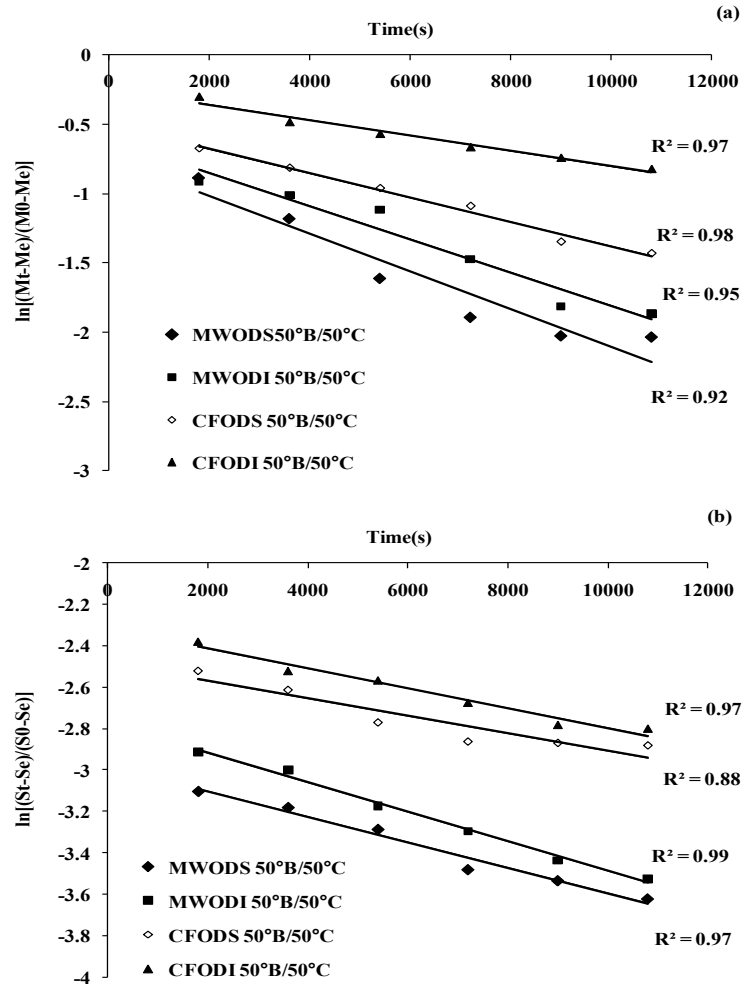


Figure 4.4 Residual moisture loss ratio (a) and solids gain ratio (b) as a function of contact time during osmotic dehydration at 50°C/50°Brix in different methods

The moisture diffusivity values associated with microwave-osmotic drying (MWODS and MWODI) were higher than with the other methods. Between the two MW techniques, moisture diffusivities under MWODS were $7.98 \times 10^{-10} \text{ m}^2/\text{s}$ and $9.79 \times 10^{-10} \text{ m}^2/\text{s}$, at 40°B/40°C and 50°B/50°C, respectively, much higher than

$6.11 \times 10^{-10} \text{ m}^2/\text{s}$ and $7.99 \times 10^{-10} \text{ m}^2/\text{s}$, respectively, with the MWODI process. Relative to these values, the moisture diffusivity under conventional modes of osmotic drying was much smaller.

Table 4.2 Moisture (D_m) and solids (D_s) diffusivity coefficients during osmotic drying of apples at different conditions

Processing Conditions	Method	$D_m (\times 10^{-10}) \text{ m}^2/\text{s}$	Intercept	R^2	$D_s (\times 10^{-10}) \text{ m}^2/\text{s}$	Intercept	R^2
40°C/50°Brix	MWODS	8.72	-0.277	0.95	8.4	-0.223	0.99
40°C/40°Brix	MWODS	7.98	-0.221	0.95	5.33	-0.126	0.99
40°C/40°Brix	MWODI	6.11	-0.100	0.98	7.77	-0.229	0.97
40°C/40°Brix	CODS	5.72	-0.235	0.97	9.68	-0.344	0.98
40°C/40°Brix	CODI	3.09	-0.138	0.99	10.97	-0.285	0.99
50°C/40°Brix	MWODS	9.01	-0.201	0.98	9.07	-0.269	0.97
50°C/50°Brix	MWODS	9.79	-0.291	0.98	9.5	-0.306	0.98
50°C/50°Brix	MWODI	7.99	-0.210	0.94	9.74	-0.551	0.96
50°C/50°Brix	CODS	6.53	-0.151	0.97	10.9	-0.357	0.97
50°C/50°Brix	CODI	4.04	-0.061	0.96	11.4	-0.593	0.96

*MWODS: Microwave-Osmotic Dehydration under spray medium; MWODI: Microwave-Osmotic Dehydration under immersion medium; CODS: Conventional Osmotic Dehydration under spray medium; CODI: Conventional Osmotic Dehydration under immersion medium.

The overall range of values of diffusion coefficients was found to be in the order of $10^{-10} \text{ m}^2/\text{s}$, which is in agreement with values reported in the literature (Conway et al., 1983; Azuara et al., 1998; Kaymak-Ertekin and Sultanoglu, 2000; Li and Ramaswamy, 2006a). Rastogi et al. (1997) reported that the moisture diffusivity coefficient of banana in a 70% concentration syrup at 45°C was $2.34 \times 10^{-9} \text{ m}^2/\text{s}$. Park et al. (2002) found D_m for pear cubes to vary between 0.35×10^{-9} and $1.92 \times 10^{-9} \text{ m}^2/\text{s}$ and D_s to vary between 0.2×10^{-9} and $3.6 \times 10^{-9} \text{ m}^2/\text{s}$ at different temperatures (40-60°C). Lazarides et al. (1997) found moisture diffusivity values of apple slices at different temperatures (20-50°C) and sucrose concentrations (45-65%) to range from 1.42×10^{-10} to $4.69 \times 10^{-10} \text{ m}^2/\text{s}$ and solute diffusivity to range from 0.73×10^{-10} to $2.41 \times 10^{-10} \text{ m}^2/\text{s}$. It can also be observed from the above that the value of the effective diffusion coefficient was

found to be dependent on the concentration and temperature of the osmotic solution. The osmotic pressure gradient is the driving force for osmotic mass transfer and an increase in osmotic solution concentration increases the gradient and in turn the driving forces (Li and Ramaswamy, 2006c). Moisture diffusivity (D_m) at 50°C/50°Brix was higher than at 40°B/40°C, thus indicating that an increase in concentration and temperature will enhance the moisture diffusivity. This result is in agreement with other works (Lazarides et al., 1995; Rastogi et al., 1997; Li and Ramaswamy, 2006c). The solids diffusivity under MWOD (both immersion and spray mode) was much lower than with the conventional methods, indicating that MW exposure helps to limit the solids gain (Table 4.2). Solids diffusivity (D_s) at 40°C/40°Brix and 50°C/50°Brix under MWODS was lower than with the other three methods, at $5.33 \times 10^{-10} \text{ m}^2/\text{s}$ and $9.5 \times 10^{-10} \text{ m}^2/\text{s}$, respectively, whereas in the MWODI under the same conditions, the solid diffusivity increased to 7.7×10^{-10} and $9.74 \times 10^{-10} \text{ m}^2/\text{s}$, respectively. Thus, osmotic treatment under MWODS limits the solids gain better than MWODI and conventional methods. The primary reason for the lower D_s with MWODS appears to be associated with better MW absorption, resulting in a greater out-flux of moisture (also demonstrated by higher D_m values). This rapid out-flux of moisture will certainly counter the influx of solids coming from the opposite direction. It was observed that at the highest D_m value was associated with MWODS with 50°Brix and 50°C and the lowest D_m was found under CODI at 40°Brix/40°C. These results are logical if one keeps in mind that microwave treatment accelerates moisture out-flux, and the spray medium is more efficient than the immersion medium. Additional data related to the effect of osmotic solution concentration and temperature on the moisture diffusivity (D_m) and solids diffusivity (D_s) under MWODS are given in Table 4.2. These effects demonstrated a common trend and dependence of diffusion coefficients with process parameters; i.e., increasing the osmotic solution concentration and temperature results in an increasing of moisture diffusivity (D_m) and solids diffusivity (D_s).

4.3.4 Half-drying time

Half-drying time is the time required to remove half of the available moisture or accomplish half of the potential solids gain. Although half-drying under different conditions will mean different extents of drying, the parameter is still an effective measure of the rate of drying. Figure 4.5(a,b) show the half-drying time with respect to moisture loss (Z_m) and solids gain (Z_s) for different methods.

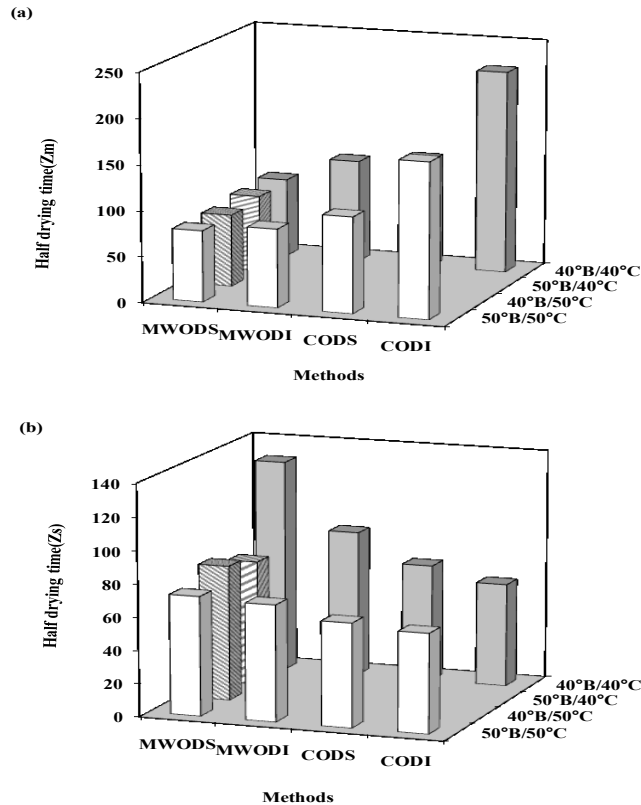


Figure 4.5 Half-drying time for moisture loss (a) and solids gain (b) with different methods at 50°C/50°Brix: Microwave-osmotic dehydration under medium spray (MWODS) and medium immersion (MWODI), and conventional osmotic dehydration under medium spray (CODS) and processing temperature-concentrations combinations

Figure 4.5a compares the different methods for moisture loss and Figure 4.5(b) for solids gain. The half-drying time for moisture loss (Z_m) was 78 and 170 min under MWODS and CODI at 50°C/50°Brix, respectively, the first one showing the shortest time or fastest drying condition and the second one the slowest among the four methods. With respect to the half-drying time for solids gain (Z_s data), the trends were opposite to those observed for Z_m . The Z_s was 129 and 63 min under 50°B/50°C with MWODS vs. CODI. For each method, the higher concentration and higher temperature conditions resulted in lower Z_m values and higher Z_s values. Overall, the half-drying times were reciprocally related to the diffusion coefficients.

4.3.5 Diffusion model for ML and SG

Diffusion coefficients are obtained from the slopes of the straight line portion of the residual moisture loss ratio or the solids gain ratio curves. In order to use the diffusion coefficients to predict the transient moisture loss or solids gain, the underlying assumptions of the diffusion model need to be recognized. The original analytical solution (Crank, 1975) to the diffusion model is based on an infinite summation series that is approximated to the first term. This happens only after a time lag, which represents the system resistance of the particle moisture loss or solids gain. When the resistance at the particle surface is negligible (an assumption used), the lag will generally be minimal and depend on the internal resistance.

There is still a time lag between the surface and the center that increases with the size of the particle. Again, this is often neglected and the plot is assumed to begin with a ratio at 1.0. For mass average moisture loss or solids gain, this intercept is expected to be different ($\ln(0.56)$) as indicated in Eq. (4.8). The regression details for the different curves (Table 4.2) clearly indicates that the real intercepts deviate from this value. This will cause a time shift between the experimental and predicted curves. In order to match the two curves, it becomes necessary to use the computed intercept rather than the theoretical intercept of -0.58. Even so, the predictions will be valid only

after a time interval representing a Fourier number ($F_o = \text{Diffusivity} \times \text{time/square of the radius}$) beyond 0.2 due to the first term approximation.

Figure 4.6 shows the diffusion model prediction for moisture loss and solids gain. Only predictions from treatment times 30 min and beyond are shown. Within this time frame of 30 min to 2 h, the prediction for both moisture loss and solids gain show fairly good performance. However, the way it is used, and like the empirical Azuara model, the diffusion model also becomes a two-parameter model involving the diffusion coefficient and an intercept factor. As with the Azuara model, the diffusion model predictions clearly differentiate the different methods used for osmotic drying as well as the different temperature-concentration treatment conditions with MWODS, confirming the experimental trends discussed earlier.

4.4 Conclusions

This study has focused on the modeling of the mass transfer kinetics of apple cylinder under MWODS. The results showed that the two-parameter Azuara model can be used to describe the transient mass transfer kinetics in the osmotic dehydration process of apple cylinder satisfactorily. This model also is useful in computing the equilibrium point for the moisture loss and solids gain based on the short duration osmotic treatments, rather than waiting for the real equilibration to be achieved. The diffusion model is used to compute the diffusion coefficients and in order to successfully use it for model prediction, it is necessary to add the intercept parameter. Even so, the model deviates from actual during the short treatment times (less than 30 min). In both cases, the model parameters were sensitive to changes in process parameters. Overall the results confirm the conclusion that the use of MWODS improves mass transfer rate during the process and has a higher diffusion rate of water while decreasing solids gain.

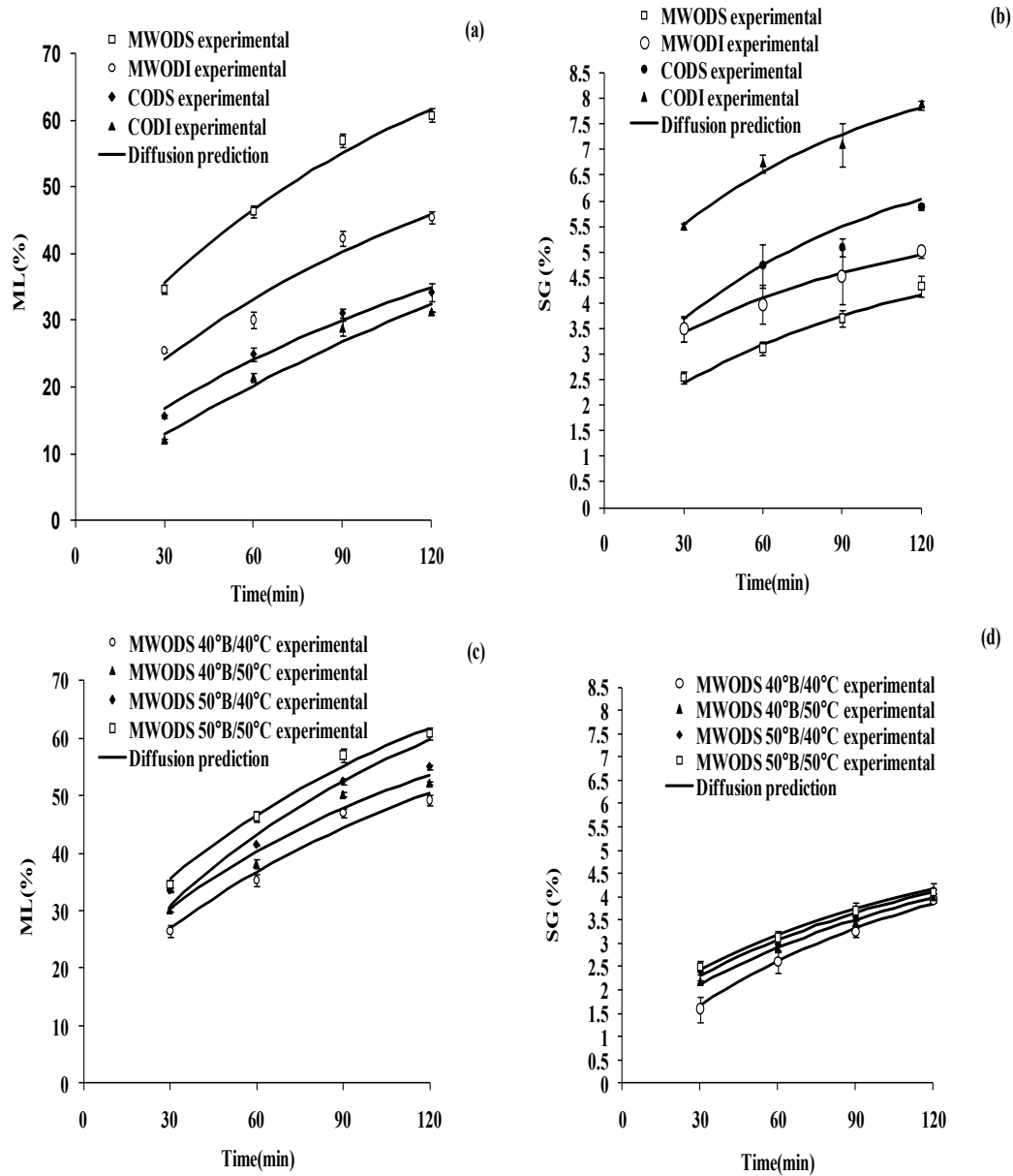


Figure 4.6 Diffusion model prediction for transient moisture loss (a) and solids gain (b) with different methods at 50°C/50°Brix: Microwave-osmotic dehydration under medium-spray (MWODS) and medium-immersion (MWODI) and conventional osmotic dehydration under medium-spray (CODS) and medium-immersion (CODI); moisture loss (c) and solids gain (d) with MWODS at different temperature and concentrations

CONNECTIVE STATEMENT TO CHAPTER 5

In the previous two Chapters (Chapter 3 and 4), the microwave osmotic dehydration under spray medium flow (MWODS) was developed and shown to permit better exchange of moisture and solids between apple cylinders and osmotic solution than the conventional osmotic drying process. It was also demonstrated the traditional OD models could be successfully employed to describe the mass transfer kinetics. This chapter is devoted to the detailed evaluation of different MWODS process variables such as sucrose concentration, osmotic medium temperature, flow rate and contact time on the moisture loss, solids gain and weight reduction.

Part of the results of this study has been presented at the following conference:

Azarpazhooh, E and Ramaswamy, HS. 2010. Effect of different variables on microwave osmotic dehydration under spray mode (MWODS) of apple cylinder using response surface methodology. The 17th World Congress of the International Commission of Agricultural and Biosystems Engineering (CIGR). July 13-17, Québec City, Canada. (Poster)

Based on results from Chapter 5, a manuscript has been accepted for publication.

Azarpazhooh, E and Ramaswamy, HS. 2010. Evaluation of factors influencing microwave osmotic dehydration of apples under continuous flow medium spray (MWODS) conditions. Food and Bio-products Technology (Accepted ; Manuscript Number FABT-868).

This research work was completed by the Ph.D. candidate under the supervision of Dr. HS. Ramaswamy.

CHAPTER 5. EVALUATION OF FACTORS INFLUENCING MICROWAVE OSMOTIC DEHYDRATION OF APPLES UNDER CONTINUOUS FLOW MEDIUM SPRAY (MWODS) CONDITIONS

Abstract

Microwave osmotic dehydration under continuous flow medium spray (MWODS) conditions is an innovative concept with high potential for enhancing moisture loss as well as improving product quality. Quantification of mass transfer kinetics under different processing conditions is important for managing and optimizing the osmotic dehydration process. A response surface methodology was used for evaluating and quantifying the moisture loss and solids gain kinetics of apples during the MWODS process. Experiments were designed according to a central composite rotatable design with all independent variables included at five levels (sucrose concentration, 33.3 - 66.8 °Brix; temperature, 33.3 - 66.8 °C; flow rate, 2120-3480 ml/min and contact time, 5-55 min). The process responses were moisture loss (ML), solids gain (SG) and weight reduction (WR) and were related to process variables using second order polynomial regression models. The lack of fit was not significant ($P > 0.05$) for any of the developed models. For ML, SG and WR, the contact time was the most significant factor during the MWODS process followed by temperature and sucrose concentration. The effect of flow rate was significant only with moisture loss. The quantity of ML, SG or WR achieved over a 30 min treatment time was chosen as the drying rate. These rates were shown to be responsive to the osmotic treatments increasing with sucrose concentration, flow rate and temperature.

5.1 Introduction

Different techniques are used to produce of high quality shelf-stable fruit products so as to enhance the product availability and extend the marketability, since fresh fruits locally are not always available. Osmotic dehydration is a mild process in which the product texture is only moderately affected, nutritional value is generally well maintained and the product quality is often elevated. It results in partial removal of water from food tissues by immersion in a hypertonic (osmotic) solution. The driving

force for the moisture diffusion is the high osmotic pressure exerted by the osmotic medium. The moisture diffusion is accompanied by a simultaneous counter diffusion of solutes from the osmotic solution into the fruit tissue. Since the membrane responsible for the osmotic process is not perfectly selective, other solutes present in the cells can also be leached into the osmotic solution (Dixon and Jen, 1977). However, unlike in conventional drying, there is no need to supply the latent heat because the moisture is removed by a physical diffusion process rather than vaporization. The process therefore is energy-efficient. Several studies have been carried out to evaluate the influence of process variables (concentration and composition of the osmotic solution, temperature, contact time, agitation, nature of food and its geometry, medium/sample ratio, flow rate etc.) on the mass transfer kinetics of conventional osmotic dehydration processes (Nsonzi and Ramaswamy, 1998; van Nieuwenhuijzen et al., 2001; Rastogi et al., 2002; Li and Ramaswamy, 2006a)

Since osmotic dehydration is a slow process, there has always been a need to develop supplementary techniques to enhance the mass transfer without adversely affecting the quality (Rastogi et al., 2002). Several techniques have been used to improve the mass transfer rates. These include application of partial vacuum, high hydrostatic pressure and high intensity electrical field pulse treatments, and using centrifugal force, ohmic heating, ultrasound and microwave (MW) during or after the osmotic dehydration process (Fito, 1994; Eshtiaghi et al., 1994; Ade-Omowaye et al., 2002; Contreras et al., 2005; Li and Ramaswamy, 2006a; Paes et al., 2007; Deng and Zhao, 2008; Allali et al., 2008).

Microwave heating has been used in many drying studies (Orsat et al., 2005; Li and Ramaswamy, 2006c; Gowen et al., 2006). Microwave heating involves conversion of electromagnetic energy into heat by selective absorption and dissipation. Microwave heating is attractive for thermal processing due to its volumetric nature of heating, rapid temperature rise in the product, and controllable heat deposition. The microwaves generate heat in the food by friction due to rotation of dipolar molecules (mostly water) and polarization ionic salts, which try to orient and align with themselves with the MW

field. Microwaves transmitted through a solid or liquid medium produce a variety of effects that can influence mass transfer. In the context of microwave-assisted osmotic dehydration, there can be rapid and differential heat generated within the product as a result of the MW absorption. These results in a pressure build up within the product thereby accelerating the moisture loss. The principal component absorbing MW radiation is the water molecule which exists in higher concentration in the fruit as compared to the osmotic medium. Again, during the MW osmotic drying, the increased outward flux of moisture from the product resists the counter-acting solids flow thereby limiting the solids gain. However, immersion of the fruits in osmotic medium during the MW heating prevents the full exposure of fruits to the MW. On the other hand, the same treatment under a medium spray mode would provide a more direct exposure of the fruit to MW since the spray drains off, leaving only a thin layer of the osmotic medium at the fruit surface. Applying the medium as a spray also overcomes the problem floating of the fruit in the solution (Gowen et al., 2006).

In chapter 3 and 4 and published papers (Azarpazhooh and Ramaswamy 2010 a,b), MW osmotic dehydration concept [under medium immersion (MWODI) and medium spray (MWODS) flow conditions] of apples (*Red Gala*) was tested and compared with conventional continuous osmotic dehydration [medium immersion (CODI) and medium spray (CODS) flow] under similar conditions. The MWODS process was reported to significantly enhance the moisture loss. Distinct differences were observed between the four methods with respect to moisture loss, weight reduction, solids gain, ML/SG ratio and dehydration time to achieve a target moisture loss, weight reduction or solids gain. The highest moisture loss, highest weight reduction, highest ML/SG ratio, lowest solids gain and shortest dehydration times were reported to be associated with MWODS. Thus, the MWODS process was shown to have a distinct advantage over the other systems and to have a good potential as a novel osmotic drying pre-treatment.

The objective of the present study was to extend the work conducted in chapters 3 and 4 to quantify the effect of sucrose concentration, temperature, flow rate

and contact time on the moisture loss, solids gain and weight reduction during the microwave-osmotic dehydration of apples under continuous flow medium spray heating conditions (MWODS) using a central composite rotatable design (CCRD) of experiments and a response surface methodology (RSM) for data analysis.

5.2 Materials and Methods

5.2.1 Materials

Apples (*Red Gala*) of uniform size and ripeness were obtained from the local market. The fruits were stored and refrigerated at 2-5°C and 95% relative humidity until use. Samples were cut as cylinders of 14 mm diameter and 14 mm height from the paranchymatic tissue with a metallic cork borer and oriented parallel to the natural apple axis. The weight of each cylinder was about 3g. Commercial sucrose (Redpath Canada Ltd., Montreal, QC) was used as the osmotic agent. The initial moisture content of the fresh apples varied from 85 to 89% (wet basis).

5.2.2 Microwave osmotic dehydration set- up

The microwave osmotic dehydration set-up was based on the system previously described in chapter 3 for the spray mode heating. It consisted of a microwave transparent chamber (made from glass) placed inside a domestic microwave oven with a maximum output of 1100 W at 2450 MHz (Danby DMW1153BL 0.031 m³ Guelph, ON. Canada). A spray device (CF-151-S, Waterpik Technology Inc., Markham, ON. Canada) was attached at the inside top of the chamber for continuously spraying the osmotic medium on apple samples placed in the chamber. Test samples were held together using a thin nylon mesh that could be easily removed. Sucrose solution, preheated to a selected temperature, was pumped to the spraying device and sprayed on the material, collected below the samples, and pulled out from the bottom of the chamber using a peristaltic pump (75-211-30, Barnant CO., Barrington, IO). The returning osmotic medium was circulated through a long coil placed in a temperature controlled water bath (Model TDB/4, Groen Division, Dover Crop, and IL). The water

bath temperature was adjusted to the desired inlet temperature of the osmotic medium, and monitored continuously at the entry and exit points of the oven. The osmotic medium circulation system was a continuous loop from the bottom of the chamber up to the spray device. The loop is broken only during the spray, but the entire osmotic medium was continuously recirculated, after temperature equilibration in the water bath. The flow rate was maintained using the peristaltic pump. A schematic of the set-up is shown in Figure 3.1 in chapter 3. The coil in the steam chamber was sufficiently long so as to provide a osmotic medium/ fruit ratio of over 30:1. The nearly closed loop for the medium flow prevented evaporation of water from the osmotic medium and the large solution to fruit ratio allowed for maintaining a steady sucrose concentration in the osmotic medium. The small amount of vapor lost during the spray treatment in the microwave chamber was nearly compensated for by the dilution of the osmotic medium by the moisture picked up from the fruit since the solute concentration in the osmotic medium nearly remained constant through the duration of the tests [measured using a hand held refractometer (ATAGO Co., Tokyo, Japan)]. The rapid flow rate of the osmotic medium also helped to prevent large temperature buildup in the osmotic medium during the microwave heating. The temperature difference between the osmotic solution going in and out of the microwave oven was about (3-5°C) accounting for nearly 70% absorption of the microwave power.

5.2.3 Osmotic dehydration procedure and the experimental plan

Apple cylinders, in 100 g pre-weighed batches, tied in mesh bags, were placed in the glass chamber inside the microwave oven. Each testing condition was a pre-selected specific test run and involved a specified sucrose concentration, temperature and flow rate. Under each testing condition, osmotic dehydration was carried out to a selected contact time. After the specified treatment, the MW was turned off, osmotic medium circulation was stopped and the test sample was removed from the solution and drained. The excess solution on the surface was removed using a paper towel and then the sample was weighed. A central composite rotatable design with four factors (sucrose concentration, temperature, flow rate and contact time) at five coded levels (-

1.68, -1, 0, 1, 1.68), 7 central points and 8 axial points was used (Myers and Montgomery, 2002). Test conditions employed are shown in Table 5.1.

Table 5.1 Experimental design of process in coded^a and actual variables and values of experimental data for microwave osmotic dehydration under spray (mean values plus standard deviation in parenthesis)

Run No.	Sucrose concentration (°B)	Temperature (°C)	Flow rate (ml/min)	Time (min)	Moisture Loss (%)	Solids Gain (%)	Weight Reduction (%)
1	60(+1)	40(-1)	3200(+1)	15(-1)	23.3(1.3)	1.77(1.04)	21.5(2.20)
2	40(-1)	40(-1)	2400(-1)	45(+1)	28.4(1.39)	1.63(1.19)	26.7(2.44)
3	60(+1)	60(+1)	3200(+1)	45(+1)	47.0(1.94)	3.96(2.13)	43.0(3.90)
4	40(-1)	60(+1)	2400(-1)	15(-1)	22.2(1.59)	2.48(1.53)	19.7(2.97)
5	60(+1)	60(+1)	3200(+1)	15(-1)	29.5(1.69)	2.43(1.70)	27.1(3.24)
6	60(+1)	40(-1)	3200(+1)	45(+1)	36.3(2.05)	3.13(2.32)	33.2(4.19)
7	60(+1)	60(+1)	2400(-1)	45(+1)	45.5(2.15)	3.76(2.49)	41.8(4.45)
8	60(+1)	60(+1)	2400(-1)	15(-1)	29.0(2.06)	2.57(2.34)	26.5(4.21)
9	60(+1)	40(-1)	2400(-1)	45(+1)	35.8(2.18)	2.87(2.55)	32.9(4.53)
10	40(-1)	40(-1)	3200(+1)	45(+1)	30.5(2.45)	2.78(3.01)	27.7(5.25)
11	40(-1)	40(-1)	2400(-1)	15(-1)	18.8(1.27)	1.60(0.99)	17.2(2.12)
12	60(+1)	40(-1)	2400(-1)	15(-1)	22.7(1.39)	2.10(1.19)	20.6(2.44)
13	40(-1)	60(+1)	3200(+1)	45(+1)	37.6(1.94)	3.33(1.18)	34.2(3.90)
14	40(-1)	60(+1)	2400(-1)	45(+1)	35.3(1.5)	2.87(1.38)	32.5(2.73)
15	40(-1)	60(+1)	3200(+1)	15(-1)	24.4(1.69)	1.94(1.70)	22.5(3.24)
16	40(-1)	40(-1)	3200(+1)	15(-1)	20.7(1.83)	1.68(1.94)	18.9(3.61)
17	50(0)	67(+1.68)	2800(0)	30(0)	37.7(1.60)	3.09(1.55)	34.6(3.00)
18	33(-1.68)	50(0)	2800(0)	30(0)	27.2(1.69)	1.83(1.70)	25.4(3.24)
19	67(+1.68)	50(0)	2800(0)	30(0)	37.7(1.28)	3.21(1.01)	34.5(2.15)
20	50(0)	33(-1.68)	2800(0)	30(0)	26.3(1.51)	2.40(1.39)	23.9(2.76)
21	50(0)	50(0)	2800(0)	5(-1.68)	17.7(1.61)	2.01(1.57)	15.7(3.02)
22	50(0)	50(0)	2800(0)	55(+1.68)	40.3(1.94)	4.01(2.13)	36.2(3.90)
23	50(0)	50(0)	2120(-1.68)	30(0)	30.8(1.59)	1.74(1.53)	29.1(2.97)
24	50(0)	50(0)	3480(+1.68)	30(0)	38.6(1.27)	3.25(0.99)	35.3(2.12)
25	50(0)	50(0)	2800(0)	30(0)	34.6(1.69)	2.99(1.70)	31.7(3.24)
26	50(0)	50(0)	2800(0)	30(0)	33.0(1.49)	3.01(1.36)	30.1(2.71)
27	50(0)	50(0)	2800(0)	30(0)	34.6(2.04)	3.20(2.31)	31.9(4.16)
28	50(0)	50(0)	2800(0)	30(0)	35.2(1.60)	2.89(1.55)	32.5(3.00)
29	50(0)	50(0)	2800(0)	30(0)	35.1(1.83)	3.22(1.94)	32.2(3.61)
30	50(0)	50(0)	2800(0)	30(0)	35.0(1.69)	3.06(1.70)	32.1(3.24)
31	50(0)	50(0)	2800(0)	30(0)	33.0(1.28)	2.66(1.00)	30.4(2.15)

^a Code 0 is for center point of the parameter range investigated, ± 1 for factorial points, and ± 1.68 for augmented points

5.2.4 Osmotic dehydration kinetic responses

Evaluation of mass exchange between the solution and sample during the osmotic dehydration were made by using the traditional parameters such as moisture loss (%ML), weight reduction (%WR) and the solids gain (% SG) from the following equations:

$$\%ML = 100 \frac{(M_0 x_0 - M_t x_t)}{M_0} \quad (5.1)$$

$$\%WR = 100 \frac{(M_0 - M_t)}{M_0} \quad (5.2)$$

$$\%SG = 100 \frac{(M_0 s_0 - M_t s_t)}{M_0} \quad (5.3)$$

Where M_0 and M_t are the sample mass (g) at time 0 and time t ; x_0 and x_t are the moisture fractions (kg/kg wet basis at time 0 and at time t ; S_0 and S_t are the solid fractions (kg/kg wet basis) at time 0 and time t . These equations are based on the assumption that no solid leaked into the solution.

Moisture content was determined in triplicate as follows: samples were weighed and placed in an oven set at 105°C for approximately 24 h until a constant weight was reached (AOAC, 2000). The solids content and moisture content (by difference) in the test samples were estimated from the difference in weight before and after the oven drying. The sucrose concentration in the syrup was measured with a portable refractometer (ATAGO Co., Tokyo, Japan) at 20°C. During experiments, it was assumed that the amount of solid leaching out of apples during osmosis was negligible (Nsonzi and Ramaswamy, 1998a; Rastogi et al., 2002).

5.2.5 Rate of moisture loss and solids gain

To compare the effectiveness of different osmotic drying conditions, the moisture loss and solids gain following a 30 min treatment was calculated from the

model for different processing conditions involving a combination of temperature (40-60°C), sucrose concentration (40-60°Brix) and flow rate (2400-3200 ml/min) and used as a measure of the rate of moisture loss and solids gain. The process variables were evaluated then with respect to their influence on the rate of ML and SG.

5.2.6 Data analysis

The second-order polynomial equation models were fitted to the experimental data for each dependent variable (moisture loss, weight reduction and solids gain) as shown below

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_{11} X_1^2 + b_{22} X_2^2 + b_{33} X_3^2 + b_{44}X_4^2 + b_{12} X_1X_2 + b_{13}X_1X_3 + b_{14} X_1X_4 + b_{23} X_2X_3 + b_{24} X_2X_4 + b_{34} X_3X_4 \quad (5.4)$$

where b_0 , b_1 , b_2 , b_3 , b_4 , b_{11} , b_{22} , b_{33} , b_{44} , b_{12} , b_{13} , b_{14} , b_{23} , b_{24} and b_{34} are regression coefficients of the model; Y represents the experimental response- either moisture loss, solids gain or weight reduction of apples; X_1 , X_2 , X_3 and X_4 are sucrose concentration (°Brix), temperature (°C), flow rate (ml/min) and contact time (min), respectively. The polynomial regression coefficients in Eq. (5.4) were determined using a commercial statistical package Design-Expert version 6.01 (StatEase Inc., Minneapolis, MN) and used for generating the response surface and contour plots. The significant terms in the model were found by analysis of variance (ANOVA) for each response. In order to check the adequacy of the model, the non-significant ($P > 0.05$) terms were removed by using a step-wise “backward” multiple reduction algorithm and the associated R^2 , adj- R^2 , pre- R^2 , Adeq. Precision parameters were computed (Myers Myers and Montgomery, 2002).

5.3 Results and Discussion

5.3.1 Experimental results and model fitting

Data on moisture loss, solids gain and weight reduction evaluated under the different CCRD experimental conditions are tabulated as mean values (with standard

deviations in parentheses) in Table 5.1. The data acquisition procedure and analysis employed in this study are unique and different from conventional osmotic dehydration studies. In conventional studies, under a given set of experimental conditions (fixed levels of sucrose concentration, temperature and flow rate), the sample is generally subjected to a series of osmotic contact times (usually at 15-30 min intervals) until some level of equilibrium is reached. Under the testing conditions employed in this study, with each of the three process variables at 5 levels, this would mean 625 test runs if five time steps are used. This is almost prohibitive. Hence, a CCRD design was used with all four factors at five levels, plus a few selected levels as additional tests. The total number of tests was reduced to 31 and replicated once. According to the design, even the replication is not essential since the central point is replicated seven times to get an estimate of the experimental variability. Experiments are statistically designed so that each experiment is carried out with at only one variable at a different condition except for the central point replicates. This permits to model the response parameters (dependant variables) as a function of the four independent variables through polynomial regression.

A second-order polynomial response surface model Eq. (5.4) was fitted to each response variable (Y). The statistical parameters were program generated and are summarized in Table 5.2 indicating a quadratic model to give the best performance for all response variables. In order to determine the significant effects of process variables on each response, an analysis of variance procedure was used. The significant terms and their coefficients in the final model are summarized in Table 5.3. An important aspect of such a model is to verify the appropriateness of the model to make sure that the lack of fit was not significant ($P > 0.1$). This basically means that the model is significant in adequately predicting the response variables. This can be demonstrated by the satisfactory correlation between actual and fitted values. The R^2 for the different models ranged from 0.87 to 0.99 which were high (ML and WR models were better than SG model).

Table 5.2 Sequential model sum of squares for moisture loss, solids gain, weight reduction

Source	DF	Moisture Loss		Solids Gain		Weight Reduction	
		Sum of squares	Pr>F	Sum of squares	Pr>F	Sum of squares	Pr>F
Mean	1	31214.96		225.12		26209.75	
Linear	4	1450.38	< 0.0001	10.56	< 0.0001	1215.97	< 0.0001
Interaction	6	37.16	0.4579	1.07	0.2670	31.43	0.5179
Quadratic	4	107.71	< 0.0001	1.31	0.0169	104.39	< 0.0001
Cubic	8	10.65	0.2614	1.04	0.0203	6.76	0.4377
Residual	8	6.67		0.22		6.03	
Total	31	32828		239		27574	

Table 5.3 Analysis of variance (ANOVA) for the fit of experiment data to response surface model.

Source	Moisture Loss (%)			Solid Gain(%)			Weight Reduction (%)		
	Coefficient	Sum of squares	P-Value	Coefficient	Sum of squares	P-Value	Coefficient	Sum of squares	P-Value
Model	-36.8	1593	< 0.0001***	-13.1	12.3	< 0.0001***	-31.5	1349	< 0.0001***
Linear									
C	0.597	220	< 0.0001***	0.209	2.01	< 0.0001***	0.455	180	< 0.0001***
T	0.775	249	< 0.0001***	0.032	2.21	< 0.0001***	0.701	203	< 0.0001***
F	2.83E-03	28.0	< 0.0001***	5.88E-03	0.63	0.0116*	2.41E-03	20.2	0.0001***
t	0.391	954	< 0.0001***	-0.053	5.71	< 0.0001***	0.433	811.6	< 0.0001***
Quadratic									
C.C	-8.24E-03	11.4	0.0027**	-1.78E-03	0.53	0.0188*	-6.89E-03	73.6	0.0037*
T.T	-9.83E-03	16.2	0.0006**			NS	-9.28E-03	14.4	0.0003**
F.F			NS	-1.14E-06	0.58	0.0148*			NS
t.t	-9.26E-03	71.0	< 0.0001***			NS	-9.43E-03	73.8	< 0.0001***
Interaction									
C.T	7.28E-03	8.47	0.0078**			NS	7.32E-03	8.58	0.0027*
C.F			NS			NS			NS
C.t	6.05E-03	13.2	0.0015**			NS	5.24E-03	9.89	0.0015**
T.F			NS			NS			NS
T.t	6.11E-03	13.4	0.0013**			NS	5.59E-03	11.3	0.0009**
F.t			NS	3.13E-05	0.564	0.0159*			
Statistic analysis for the model									
Lack of fit		13.9	0.49NS		1.70	0.11NS		9.59	0.66NS
R-squared		0.988			0.865			0.989	
Adj R-squared		0.982			0.824			0.984	
CV		3.1			10.7			3.0	

C, T, F and t, are sucrose concentration ($^{\circ}$ Brix), process temperature ($^{\circ}$ C), flow rate (ml/min) and contact time (min). *Significant at 0.05 level. **Significant at 0.01 ***Significant at 0.001 level; NS: Non significant.

Figure 5.1 shows the comparison between the observed and the model predicted values. The results demonstrate that the polynomial regression models were in good agreement with the experimental data.

Figure 5.2 shows the quality of RSM model prediction for ML and SG at selected conditions demonstrating the effect of sucrose concentration, temperature and flow rate. With both ML and SG, the curves were very representative of the normal MW osmotic behavior (Azarpazhoooh and Ramaswamy, 2010a) and demonstrated higher ML and SG as the sucrose concentration, temperature and flow rate of the osmotic medium increased. The ML curves demonstrated a smooth increase in ML at the beginning and approached the equilibrium values at longer treatment times. The SG curves mostly described a linear increase in SG after a step change in SG following the first treatment. In most osmotic drying situations, the SG never forms a smooth curve from time zero. It is probably due to some residual solids, present at the surface of the fruits following the treatment, which does not get removed prior to moisture determination. The flow rate effect was small with ML within the range of experiments studied and small and somewhat mixed with respect to the SG.

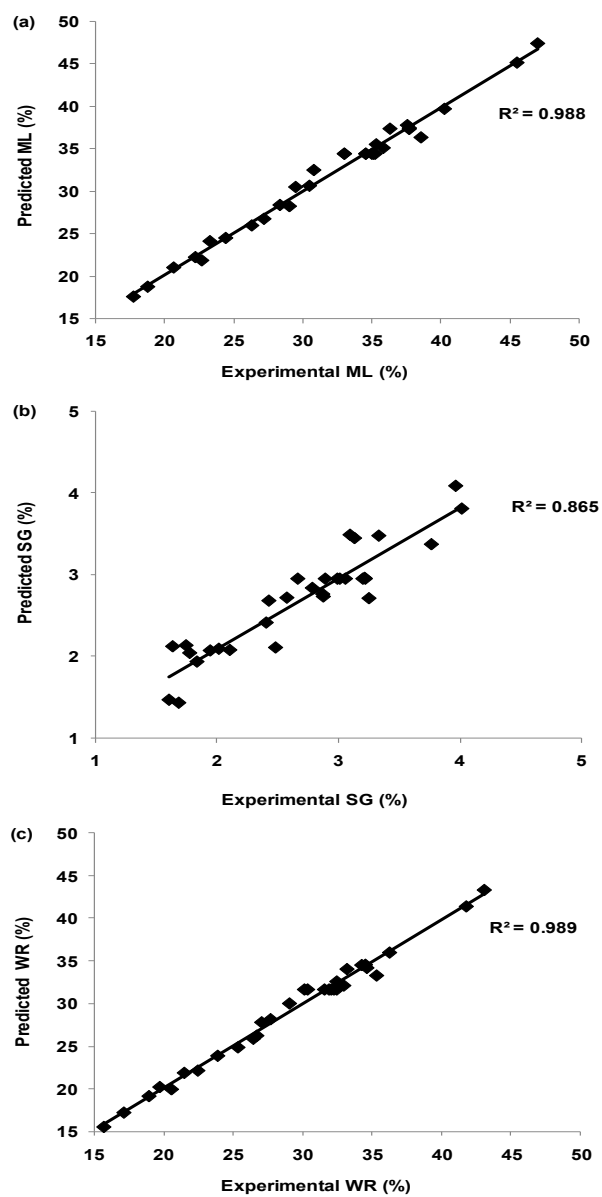


Figure 5.1 Comparison between experimental and predicted values for (a) moisture loss, (b) solids gain, (c) weight reduction under MWODS processing conditions

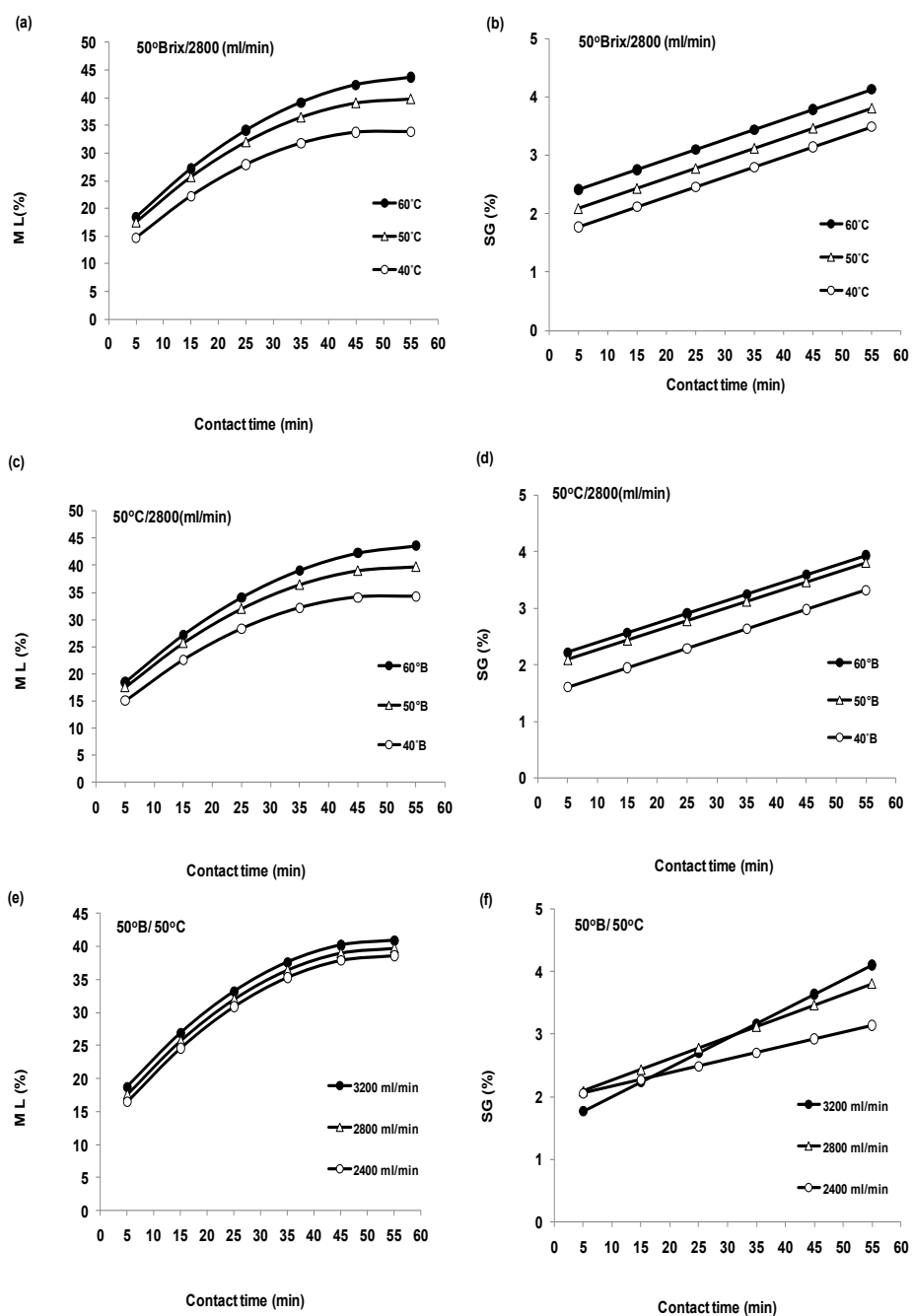


Figure 5.2 Typical model predicted ML and SG curves under different MWOD processing conditions demonstrating the effect of sucrose concentrations, temperatures and flow rate

5.3.1 Effect of process variables on transient ML, SG and WR

An analysis of variance was used to evaluate the effect of different process variables; sucrose concentration, temperature, flow rate and contact time and their interactions on moisture loss, solids gain and weight reduction. The P -values were used to get the relative importance of the influence of individual variables and their interactions. Table 5.3 summarizes the ANOVA results for ML, SG and WR. The coefficients of the 2nd order polynomial equation [Eq. (5.4)] for the different process parameters (computed on the coded values) are detailed in Table 5.3.

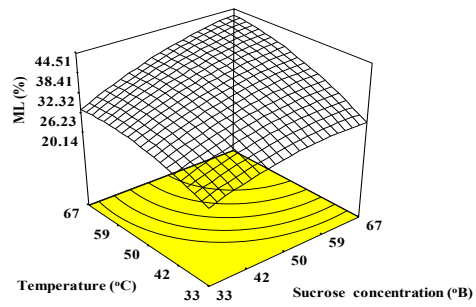
5.3.2 Moisture loss (ML)

It was observed that all linear terms of process variables had a significant effects ($P < 0.0001$), and all the quadratic terms except flow rate, and the interaction of sucrose concentration with temperature, sucrose concentration with contact time and temperature with contact time had a significant effect ($P < 0.05$) on moisture loss during osmotic dehydration. Based on the sum of squares, the importance of the independent variables on moisture loss could be ranked in the following order: contact time > temperature > sucrose concentration > flow rate. All linear terms of process variables had positive effects on ML whereas the quadratic terms of sucrose concentration, temperature and contact time had a negative influence. The interaction of sucrose concentration with temperature, sucrose concentration with contact time and temperature with contact time had a positive effect and lead to enhance the magnitude of moisture loss.

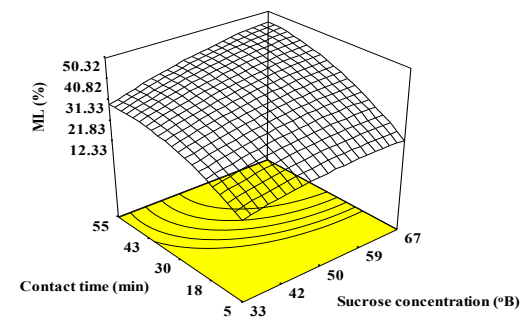
Figure 5.3 shows the combined effects of sucrose concentration, temperature and contact time (taken two at a time with the third being maintained at the central level). The flow rate effect was small and hence was fixed at the mid-level for all plots. All three plots show somewhat similar results as described earlier with respect to the main variables with only marginal interaction effects.

Between sucrose concentration and temperature (Figure 5.3a), the temperature effect was more significant than concentration effect; between temperature and contact time (Figure 5.3b), and sucrose concentration and contact time (Figure 5.3c), the time effect was more prevalent. The individual effects and their interactions can be easily explained because of the nature of influence of different process variables on osmotic parameters. The sucrose concentration effect generally favors higher ML with an increase in sucrose concentration, except that at very high levels, with its increased viscosity, which limits the solution mobility and its power to accelerate the ML. Hence, the ML increase is moderated at higher sucrose concentration levels. It is also possible that at very high sucrose concentrations, the solutes could block the pores in the fruit tissue thereby restricting the ML. With temperature, generally higher temperatures favor higher ML because higher temperatures contribute to enhanced kinetic energy and mobility of water molecules. Further, at any given sucrose concentration, a higher temperature decreases the viscosity of the solution thereby facilitating greater mobility and extraction ability of the solution. Higher temperatures can also overcome the viscosity problems associated with sucrose concentration effect thereby providing some interactive synergy. The flow rate effect in general favors higher ML at higher flow rate, but the magnitude depends on the level and range of the flow rate used. While a certain minimum is essential to achieve uniformity of operation, higher flow rate levels especially in continuous flow systems help to quickly replenish the contact surface with original solution by removing the contact fluid as it picks up the moisture from the fruit. The flow can also help to reduce the solids build-up at the surface of the product. However, flow rate beyond a certain level, may not provide additional enhancement because of lowered contact time. Time effect is again moderated by the prevailing sucrose concentration difference between the fruit and the solution which decreases with time. Hence the ML is much more effective early in the process and tends to reach an equilibrium level beyond a couple of hours of contact time. These general explanations can be seen in many previous publications (Jokic et al., 2007; Eren and Kaymak-Ertekin, 2007; Li and Ramaswamy, 2006 (a,b,c)).

(a)



(b)



(c)

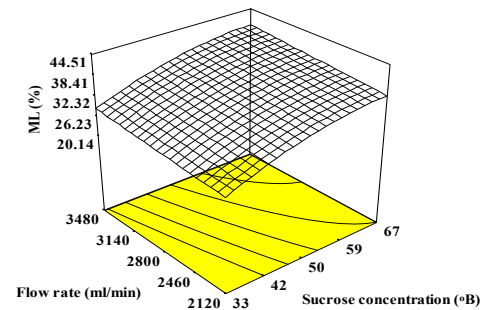


Figure 5.3 Response surface plots for ML showing the interaction effects of two variables by keeping the other two at their central points which are 50°Brix for sucrose concentration, 50°C for temperature, 30 min for the contact time and 2800 ml/min for the flow rate

5.3.3 Solids gain (SG)

All linear terms of process variables had significant effects ($P < 0.0001$) (Table 5.3) on solids gain, and in addition, the quadratic terms of sucrose concentration and flow rate had a significant effect ($P < 0.05$). Only the interaction of contact time with flow rate had a significant effect ($P < 0.05$) on solids gain. The importance of the independent variables on solids gain was ranked in the following order: contact time > temperature > sucrose concentration > flow rate.

The individual effects of all process variables were significant and the SG increased with an increase in sucrose concentration, temperature and contact time as well as flow rate. The interaction effect of flow rate and contact time on the SG is shown in Figure 5.4. The synergistic effect of increase in solids gain by the combination of contact time and flow rate is clearly evidenced in this figure with the maximum SG achieved at the highest combination level of these two independent variables. This can be attributed to the increased mobility of the solution at higher flow rates and solids accumulation as the time increased. Some studies report that the enhancement of solids gain is due to the possible membrane swelling/plasticizing effect, which might increase the cell membrane permeability to sucrose molecules (Lazarides et al., 1997; Li and Ramaswamy, 2006c). At higher flow rates, the accumulation of sucrose molecules along the surface of cytoplasm could also result in formation of a dense superficial layer which could actually decrease the solids gain (Ruiz-López et al., 2008; Shi et al., 2008; Eren Kaymak-Ertekin, 2007; Jokic et al., 2007; van Nieuwenhuijzen et al., 2001). This is more evident at short contact times.

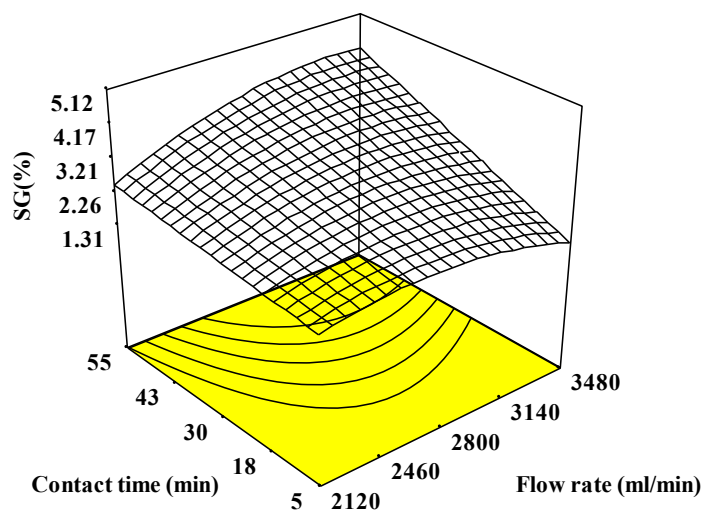


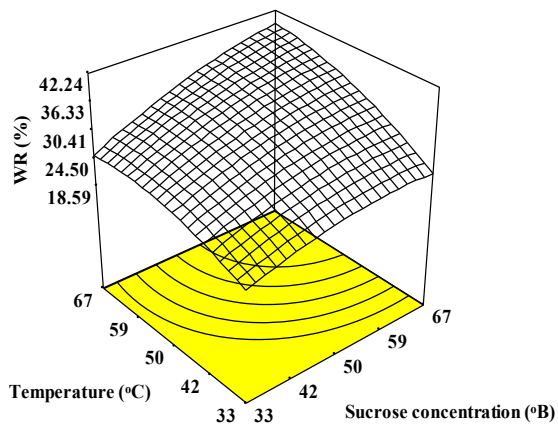
Figure 5.4 Response surface plots for SG showing the interaction effects of flow rate and contact time with sucrose concentration (50°Brix) and temperature (50°C) at their central points

5.3.4 Weight reduction (WR)

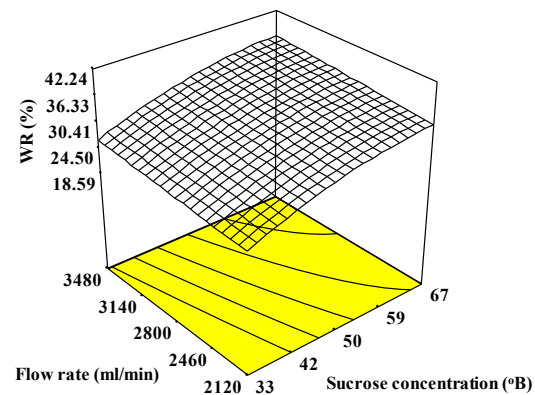
Linear effects of all process variables on weight reduction were highly significant ($P < 0.0001$). The quadratic effects of sucrose concentration, temperature and contact time were also significant ($P < 0.05$). Among the interaction terms, sucrose concentration with contact time and temperature with contact time were significant ($P < 0.05$). With respect to the WR, the importance of the independent variables was ranked as follows: contact time > temperature > sucrose concentration > flow rate.

Weight reduction is the result of moisture loss moderated by the countering solids gain. Since ML is higher than SG by almost an order of magnitude, the influence of variables on ML and WR can be expected to be similar. Figure 5.5 presents the effect of sucrose concentration, temperature and contact time on weight reduction. In each of the subplots, the other two variables were kept at their midpoint levels. As expected, it can be observed that Figures 5.3 and 5.5 are quite similar.

(a)



(c)



(b)

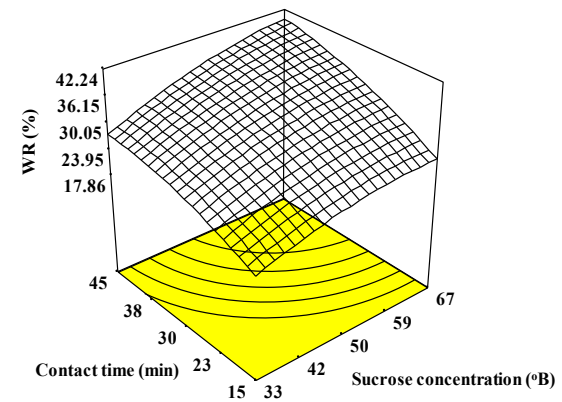


Figure 5.5 Response surface plots for weight reduction (WR %) showing the interaction effects of two variables by keeping the other two at their central points which are 50°Brix for the sucrose concentration, 50°C for the temperature, 30 min for the contact time and 2800 ml/min for the flow rate.

5.3.5 Effect of process variables on rates of ML and SG

The influence of the process variables on the rate of moisture loss (ML-30) and solids gain (SG-30) are shown in Figure 5.6. These are represented as 3-D bar graphs demonstrating the influence of sucrose concentration and temperature at three selected levels of flow rate (separate sub-figures, a-c for moisture loss rate and d-f for solids gain rate). At each flow rate, an increase in both sucrose concentration and temperature contributed to an increase in rates of ML and SG. Further, higher flow rates consistently resulted in a higher ML rate; however, the flow rate influence on SG rate was somewhat mixed.

Another interesting observation that can be made from Figure 5.6 is that the ML rates are almost ten times higher than the SG rates in almost all cases. This means that in general, the ML/SG ratio is maintained at a high level. Generally, one of the problems with osmotic drying is the high level of solids gain which diminishes the effect of moisture loss. Conditions that give relatively higher ML/SG ratios are generally favored because they generally tend to give better quality products. It was shown in previous studies that MWOD gives a considerably higher ML/SG ratio as compared to conventional osmotic dehydration techniques (Li and Ramaswamy, 2006a, b, c; Azarpazhooh and Ramaswamy, 2010a). This study not only confirms that finding but also demonstrates that under the MWODS, the ML/SG ration is significantly enhanced.

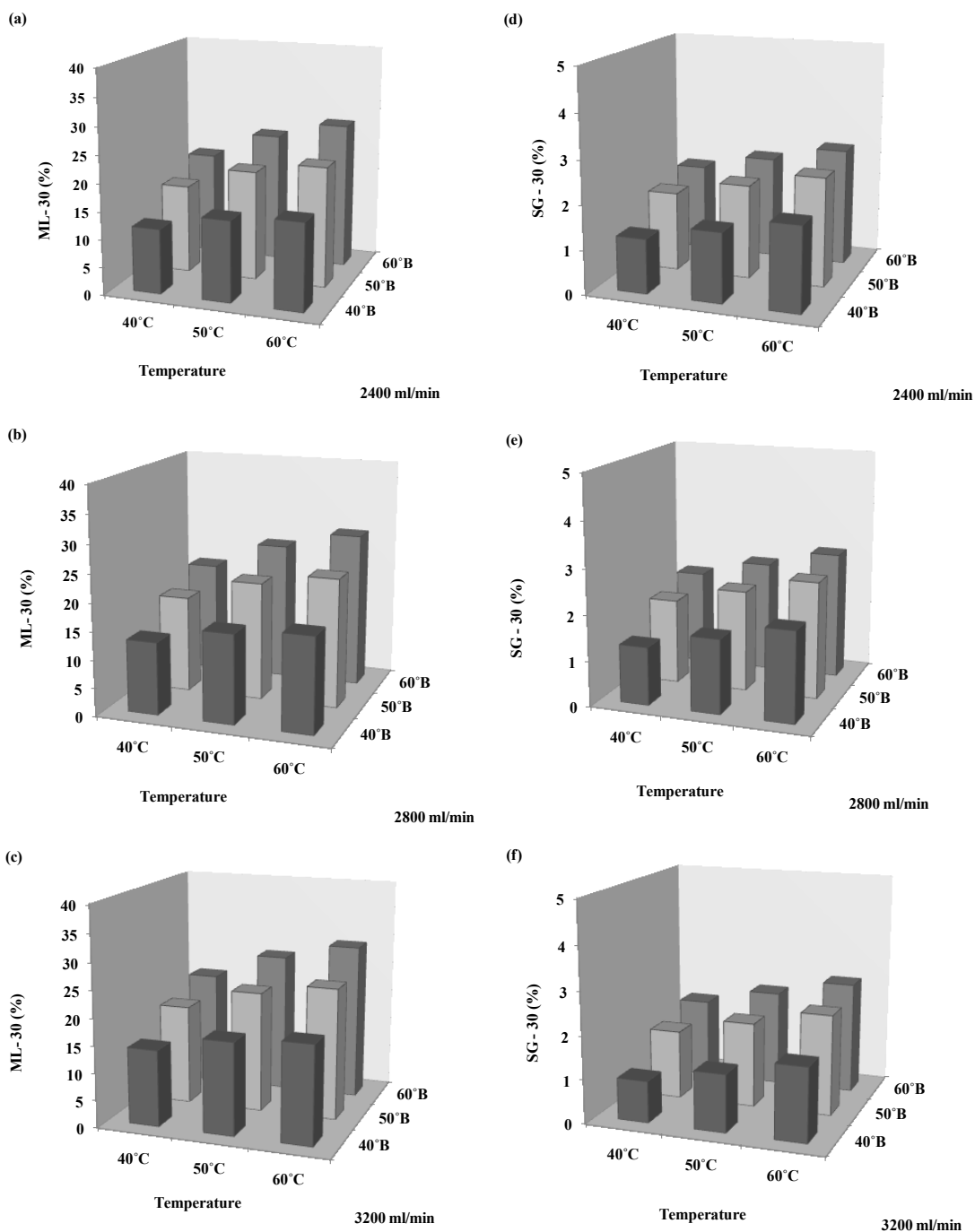


Figure 5.6 3-D bar graphs moisture loss rate (ML-30) and solids gain rate (SG-30) demonstrating the effect of sucrose concentration, temperature and flow rate

5.4 Conclusions

A CCRD model combined with RSM was used effectively to evaluate mass transfer kinetics of apples under microwave osmotic dehydration under spray mode (MWODS) conditions. The methodology was effective in developing second order polynomial models for ML, SG and WR to demonstrate the influence of sucrose concentration, temperature, flow rate and contact time. As compared to conventional methodology this would help reducing the number of experiments required to develop a comprehensive model. The study demonstrated that moisture loss, solids gain and weight loss were higher at higher sucrose concentration, higher temperature, and longer contacts time. Flow rate effects were not significant ($P > 0.05$) on solids gain. With ML and WR, all variables except flow rate had interaction effects, while with SG, only the contact time –flow rate interaction was significant. A rate of ML and SG were related to sucrose concentration, temperature and flow rate. Under the MWODS processing conditions, the ML/SG was high, demonstrating relatively much higher ML compared to SG. These conditions are more desirable because it prevents large accumulation of solutes from the osmotic medium.

CONNECTIVE STATEMENT TO CHAPTER 6

In the previous chapter (Chapter 5) the effect of different parameters during microwave osmotic dehydration under continuous flow medium spray conditions on moisture loss, solids gain and weight reduction was evaluated and discussed. In this chapter, the mass transfer behavior during MWODS process was further explored and modeled, and the MWODS kinetic parameters were related to the process variables. Further, based on desirability function models, optimal processing conditions were identified under user imposed restrictions.

Part of the results of this study has been presented at the following conference:

Azarpazhooh, E and Ramaswamy, HS. 2010. Optimization of microwave osmotic dehydration process for apple cylinders under continuous flow medium-spray conditions. Annual meeting of Institute of Food Technologists. July 17-20, Chicago, USA. (Poster)

Based on results from Chapter 6, a manuscript has been accepted for publication.

Azarpazhooh, E and Ramaswamy, HS. 2010. Modeling and optimization of microwave osmotic dehydration of apple cylinders under continuous flow spray mode processing conditions. Food and Bio-products Technology (Accepted; Manuscript Number FABT-869).

This research work was completed by the Ph.D. candidate under the supervision of Dr. HS. Ramaswamy.

CHAPTER 6. MODELING AND OPTIMIZATION OF MICROWAVE OSMOTIC DEHYDRATION OF APPLE CYLINDERS UNDER CONTINUOUS FLOW SPRAY MODE PROCESSING CONDITIONS

Abstract

The objective of this study was to model and optimize the mass transfer behavior during microwave osmotic dehydration of apple cylinders under continuous flow spray mode processing conditions. Data needed for the model development and optimization were obtained using a central composite rotatable experimental design involving sucrose concentration (33.3- 66.8°B), temperature (33.3-66.8°C), flow rate (2120-3480 ml/min) and contact times (5-55 min) and the response variables were moisture loss, solids gain and weight loss. Mass transfer kinetics was evaluated based on the empirical Azuara model and the conventional diffusion model. Diffusivities of both moisture loss (D_m) and solids gain (D_s) obtained from the diffusion model were related to sucrose concentration, temperature and flow rate. Optimization was evaluated using a desirability function model which could be used with several imposed constraints. The optimum conditions obtained depended on the imposed constraints. A set of constraints involving maximizing moisture loss and weight reduction while keeping the solids gain below 3.5% gave the following optimal conditions: a 30 min osmotic treatment at 65°B, 60°C and 2800 ml/min flow rate yielding a moisture loss of 40.9%, weight reduction of 37.7% with a solids gain of 3.32%.

6.1 Introduction

During the past decade, osmotic drying treatment of food has been attracting increased interest as a mild treatment which can improve food quality. Since osmotic drying provides only partial moisture removal, such treatments need to be coupled with other drying methods to complete the process; however, the quality of the product is generally enhanced as compared to the primary drying method alone. In osmotic dehydration, food is immersed in a hypertonic aqueous solution, leading to the diffusion of the product's moisture through cell membrane and inter-cellular network. This is always accompanied by a counter flow solute diffusion into the food due to non-

ideal selectiveness of the membrane (Hawkes & Flink, 1978). The solute uptake not only modifies the composition of the product, but also blocks the surface layers of the material, thereby contributing to increase the mass transfer resistance (Eren & Kaymak-Ertekin, 2007). In general, the solute gain from the osmotic solution is not very desirable except when dealing with tart fruits.

Osmotic dehydration in a microwave environment is an emerging technology which has an excellent potential for enhancing the rate of moisture loss from the product, limit solids gain and for enhancing product quality (Li & Ramaswamy, 2006c). The process is a combination of microwave and osmotic dehydration under the continuous flow of the osmotic medium with fruit pieces fully posited in a fully immerse mode and has been shown offer a significant advantage over the conventional counterparts. The microwave field helps to enhance the driving force for the removal of moisture from the fruit into the osmotic medium and thus results in increasing the rate of moisture removal. More recently, this concept was improved further by replacing the immersion system with a spray system (Azarpazhooh & Ramaswamy, 2010a). The spray system is more advantageous because it provides greater exposure of the fruit to MW heating and eliminates the problem of the floating of fruit pieces. In both systems, moisture loss (ML) is significantly enhanced while the solids gain (SG) is suppressed providing a better ratio of ML/SG than possible with conventional osmotic drying treatments. Such conditions have been shown previously to promote better sensory quality in the product.

Understanding the mass transfer process during osmotic dehydration and modeling the kinetics of process has been the focus of several research activities (Azuara et al., 1992; Fernandes, Gallão & Rodrigues, 2009; Magee, Hassaballah & Murphy, 1983). Fickian unsteady state diffusion has been introduced as the most appropriate mechanism for the estimation of diffusion coefficients during the osmotic process (Azuara, Flores & Beristain, 2009; Li & Ramaswamy, 2006a). However, the application of this model has its drawback which is related to the need for long experimental procedures for finding equilibrium moisture loss and equilibrium solids

gain. Moreover, unlike their heat transfer counterparts, the mass transfer coefficients are not easy to determine. In order to simplify the process, usually unlimited mass transfer potential is assumed which often deviates from reality. Hence, in order for the model to perform well within the range of experimental conditions, the primary parameter, which is the diffusion coefficient, is combined with the model intercept coefficient. Two parameter empirical models for both moisture loss and solids gain have been proposed by Azuara et al. (1992) to describe the mass transfer patterns during short osmotic treatments. The benefit of these models is predicting the equilibrium moisture loss and equilibrium solid gain during osmotic dehydration. Azarpazhooh & Ramaswamy (2010b) found good results for the Azuara model and they demonstrated its usefulness in predicting the equilibrium values of moisture loss and solids gain during microwave assisted osmotic dehydration. These results show that MWOD (both immersion and spray medium) enhances the moisture diffusion rate even at low solution temperatures as compared with conventional osmotic dehydration (Azarpazhooh & Ramaswamy, 2010b; Li & Ramaswamy, 2006b). Implementation of osmotic dehydration on an industrial scale has to deal with problems such as process optimization, solution management and designing continuous process equipment. The efficiency of these large scale treatments are complicated by the problem of floating behavior of the fruits during the treatment. The MWOD with a spray system (MWODS) (Azarpazhooh & Ramaswamy, 2010a) effectively helps to solve the floating problem.

Response surface methodology (RSM) has been widely used as an effective tool in industrial processing to develop or improve processes/products through optimization process (Floros & Chinnan, 1988). This methodology is fundamental for finding certain desirable operating conditions by considering maximum or minimum region in the total space of the factors (Myers Myers and Montgomery, 2002). Optimization of osmotic dehydration helps to reduce production and energy costs and minimize undesired effects during process. There are several studies on optimization of fruit and vegetable processing by RSM methods (Eren and Kaymak-Ertekin, 2007; Koocheki & Azarpazhooh, 2010; Rodrigues & Fernandes, 2007a; Singh, Panesar & Nanda, 2008). During optimization, some variables may need to be maximized while some others may

need to be minimized. However, in competing situations, one response may have an opposite effect on another one resulting in complications (Eren and Kaymak-Ertekin, 2007).

Several approaches have been used to solve the optimization problem including: (i) constrained optimization, (ii) superimposed contour diagrams of the different response variables, (iii) combination of all responses into one model by using desirability functions. The desirability function approach is one of the most widely used methods in industry for the optimization of multiple response processes. Desirability concept for multi-criteria optimization in industrial quality management was introduced by Harrington (1965). It is based on the idea that the "quality" of a product or process that has multiple quality characteristics, is unacceptable when one of them stays outside of some "desired" range. The method finds operating conditions that provide the "most desirable" response values. The desirability function model has the potential to compare responses with different scales, transforming easily the responses to one measurement in order to be applied for both qualitative and quantitative responses (Shi et al., 2008). It is measured by a desirability index (DI). DI is the multivariate optimization problem which is converted into a univariate one. Based on one of the design of experimental methods, the optimal levels of process influencing factors can be determined by simultaneously taking into consideration all competing constraints (Trautmann and Weihs, 2006).

In chapter 5, a new procedure for evaluating osmotic dehydration process kinetics was elaborated and the effect of different osmotic variables associated with MWODS on moisture loss, solids gain and weight reduction was evaluated. The present study is an extension of this work for process and process optimization. Through the use of response surface methodology and desirability function various optimization scenarios are discussed for the application of MWODS process for apple slices.

6.2 Material and Methods

6.2.1 Experimental data and RSM models

Experimental data gathering and preliminary data handling for this manuscript is essentially the same as detailed in chapters 3 and 5. Briefly, apples (*Red Gala*) were peeled and cut into a cylindrical shapes with both diameter and length of 14 mm. Each cylinder had a weight of 3 ± 0.03 g. In order to avoid changes in osmotic solution concentration during the treatment, a solution to sample mass ratio of 30:1 was used. Test samples were subjected to a continuous medium flow osmotic treatment under spray mode inside a glass chamber positioned inside a microwave oven operating at full power during the treatment. The osmotic solution was continuously circulated through the system using a pump allowing it to enter the treatment chamber as a spray over the top of test samples and leaving the microwave oven from below. The syrup temperature adjusted back to the initial by making it flow through a long coil positioned in a water bath set at the desired temperature. The medium temperature at the entrance and exit of the MW oven was continuously monitored using T-type thermocouples attached to a data logger. The rapid medium flow rate allowed the temperature difference between the medium entering and leaving the MW system to be kept within 3-5°C. Additional details about the technique are presented in chapters 3 and 5. A rotatable central composite design of four factors [sucrose concentration (33.3 - 66.8°B), process temperature (33.3 - 66.8°C), flow rate (2140 - 3480 ml/min) and time (5-55 min)] at five levels, 7 central points and 8 axial points to 24 full factorial designs (Myers and Montgomery, 2002) was used. The actual factors variable chosen from preliminary studies and the corresponding coded value (-1.68, -1.0, 1, 1.68) are given in Table 6.1.

Table 6.1 Experimental design with actual and coded^a values (parenthesis) of process variables for microwave osmotic dehydration

Experiment No.	Sucrose concentration (°B)	Temperature (°C)	Flow rate (ml/min)	Time (min)
1	60(+1)	40(-1)	3200(+1)	15(-1)
2	40(-1)	40(-1)	2400(-1)	45(+1)
3	60(+1)	60(+1)	3200(+1)	45(+1)
4	40(-1)	60(+1)	2400(-1)	15(-1)
5	60(+1)	60(+1)	3200(+1)	15(-1)
6	60(+1)	40(-1)	3200(+1)	45(+1)
7	60(+1)	60(+1)	2400(-1)	45(+1)
8	60(+1)	60(+1)	2400(-1)	15(-1)
9	60(+1)	40(-1)	2400(-1)	45(+1)
10	40(-1)	40(-1)	3200(+1)	45(+1)
11	40(-1)	40(-1)	2400(-1)	15(-1)
12	60(+1)	40(-1)	2400(-1)	15(-1)
13	40(-1)	60(+1)	3200(+1)	45(+1)
14	40(-1)	60(+1)	2400(-1)	45(+1)
15	40(-1)	60(+1)	3200(+1)	15(-1)
16	40(-1)	40(-1)	3200(+1)	15(-1)
17	50(0)	67(+1.68)	2800(0)	30(0)
18	33(-1.68)	50(0)	2800(0)	30(0)
19	67(+1.68)	50(0)	2800(0)	30(0)
20	50(0)	33(-1.68)	2800(0)	30(0)
21	50(0)	50(0)	2800(0)	5(-1.68)
22	50(0)	50(0)	2800(0)	55(+1.68)
23	50(0)	50(0)	2120(-1.68)	30(0)
24	50(0)	50(0)	3480(+1.68)	30(0)
25	50(0)	50(0)	2800(0)	30(0)
26	50(0)	50(0)	2800(0)	30(0)
27	50(0)	50(0)	2800(0)	30(0)
28	50(0)	50(0)	2800(0)	30(0)
29	50(0)	50(0)	2800(0)	30(0)
30	50(0)	50(0)	2800(0)	30(0)
31	50(0)	50(0)	2800(0)	30(0)

^a Code 0 is for center point, ± 1 for factorial points, and ± 1.68 for augmented points

6.2.2 Mathematical modeling

Fick's law of diffusion is the most used phenomenological model which expresses mathematically the results of observed phenomena without paying detailed attention to their fundamental significance. Crank (1975) developed solutions of Fick's law for slabs, cylinders, and spheres at different boundary conditions (Ochoa-Martinez et al., 2007b). However, in this model, it is necessary to know the experimental value of moisture loss at equilibrium (ML_e) and the value of effective diffusivity (D). Azuara et al, (1998) proposed an adjustable two-parameter model capable of predicting ML_e . In this model, using a mass balance on water movement inside the food, Eq. (6.4) was obtained giving the rate of water loss as a function of time.

$$ML_t = \frac{S_1 t (ML_e)}{1 + S_1 t} = \frac{t (ML_e)}{\frac{1}{S_1} + t} \quad (6.1)$$

where ML_t is the moisture loss fraction at any time, t , S_1 is a constant related to the rate of water diffusion out from product, and ML_e is moisture loss fraction at equilibrium. Eq. (6.2) can be linearized as:

$$\frac{t}{ML_t} = \frac{1}{S_1 (ML_e)} + \frac{t}{ML_e} \quad (6.2)$$

Similarly for solid gain, it can also be written as,

$$\frac{t}{SG_t} = \frac{1}{S_2 (SG_e)} + \frac{t}{SG_e} \quad (6.3)$$

Where: SG_t is the solid gain fraction at any time, t , S_2 is a constant related to the rate of solids diffusion in the product and SG_e the solid gain fraction at equilibrium. The equilibrium moisture loss (ML_e), and solids gain (SG_e), can be obtained as the

reciprocal slopes of t/ML_t and t/SG_t against reciprocal of time plots, respectively. Using the equilibrium moisture loss (ML_e) and equilibrium solids gain SG_e , the diffusion coefficient associated with moisture loss and solids gain was obtained from Eqs. (6.4), (6.5) and (6.6) (Ramaswamy & van Nieuwenhuijzen, 2002).

$$M_{mfc} = 0.56e^{-\frac{8.25}{a^2}Dt} \quad (6.4)$$

where (M_{mfc}) is the unsteady mass concentration (mass average moisture ratio) in a finite cylinder. The transient moisture ratio (M_{mfcw}) in the finite cylinder ($d = r$) is defined as follow for water transfer:

$$M_{mfcw} = \frac{M_e x_e - M_t x_t}{M_e x_e - M_0 x_0} = \frac{ML_e - ML_t}{ML_e - ML_0} \quad (6.5)$$

where ML_0 , ML_e , and ML_t are the initial sample moisture loss and that at time t and equilibrium, respectively. The transient solids gain ratio (M_{mfcs}) in a finite cylinder ($d = r$) is:

$$M_{mfcs} = \frac{M_e s_e - M_t s_t}{M_e s_e - M_0 s_0} = \frac{SG_e - SG_t}{SG_e - SG_0} \quad (6.6)$$

where SG_0 , SG_t and SG_e are the initial sample moisture loss and that at time t and equilibrium, respectively. The effective diffusion coefficients of water and solute, D_m

and D_s ($m^2 s^{-1}$) can be determined, respectively, from the slope of $\ln \frac{ML_e - ML_t}{ML_e - ML_0}$

and $\ln \frac{SG_e - SG_t}{SG_e - SG_0}$, against contact time, t , for samples in the osmotic solution.

6.2.3 Optimization of MWODS

In order to optimize a process, several response variables are to be maximized or minimized. Graphical method is an overlay of the contour plots for each response

which is a relatively straightforward approach for optimizing. For finding the best view of surface, trial and error attempts may be necessary (Myers Myers and Montgomery, 2002). Constrained optimization problem (non-linear programming methods) is a popular approach for formulating and solving the problem (Corzo & Gomez, 2004). For solving this problem, a desirability optimization has been developed and used (Koocheki & Azarpazhooh, 2010; Trautmann and Weihs, 2006).

In the present study, response surface regression models were used to find optimum conditions valid within the range of experimental conditions. The Design-Expert version 6.04 software (Statease Inc., Minneapolis, USA) was used implement the desired function methodology. Desirability functions were developed under different user selectable constraints.

6.2.4 Statistical analysis

A rotatable central composite design of three factors (sucrose concentration, process temperature, and flow rate) with five levels, 7 central points and 8 axial points to 24 full factorial design (Myers and Montgomery, 2002) was used. The actual factors variable chosen from preliminary studies and the corresponding coded value (-1.68, -1.0, 1, 1.68) are given in Table 6.1. Moisture diffusivity (D_m) and solid diffusivity (D_s) were response variables for the purpose of modeling. Response surface methodology (RSM) was used to estimate the main effect of the process variables on mass transfer variables describing osmotic dehydration of fresh apple. The second-order polynomial equation model consist of the linear, quadratic and cross-product regression coefficients was fitted to the experimental data of each dependent variable of given next:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 + b_{44}X_4^2 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{14}X_1X_4 + b_{23}X_2X_3 + b_{24}X_2X_4 + b_{34}X_3X_4 \quad (6.7)$$

where $b_0, b_1, b_2, b_3, b_4, b_{11}, b_{22}, b_{33}, b_{12}, b_{13}, b_{23},$ and b_{24} are regression coefficients of the mode; Y represents the experimental response- either moisture diffusivity and solid diffusivity; X_1, X_2, X_3 and X_4 are sucrose concentration ($^{\circ}\text{Brix}$), temperature ($^{\circ}\text{C}$), flow

rate (ml/min) and contact time(min), respectively. All statistical analysis was carried out using Design-Expert version 6.04 software. The significant terms in the model were found by analysis of variance (ANOVA) for each response. In order to check the adequacy of the model, the effects that are not significant ($P > 0.05$) were eliminated by using “backward” reduction algorithm and then R^2 , adjusted R^2 , prediction R^2 and coefficient variance (CV) were computed (Myers Myers and Montgomery, 2002).

6.3 Results and Discussion

6.3.1 Experimental data handling

The following second order polynomial models were developed for moisture loss, solids gain and weight reduction based on the CCRD design (Table 6.1) as a function of sucrose concentration (C in °Brix), temperature (T in °C), flow rate (F in ml/min)) and treatment time (t in min).

$$\begin{aligned} \text{ML \%} = & -36.78 + 0.598C + 0.775T + 0.002F + 0.390t + 0.0082C^2 - 0.0098T^2 - 0.0092t^2 \\ & - 0.0073C \times T + 0.00605C \times t + 0.0061T \times t \end{aligned}$$

$$R^2 = 0.99 \quad (6.8)$$

$$\begin{aligned} \text{SG \%} = & -13.2 + 0.28C + 0.032T + 0.005F - 0.053t - 1.78C^2 + 0.000031F \times t \end{aligned}$$

$$R^2 = 0.87 \quad (6.9)$$

$$\begin{aligned} \text{WR \%} = & -31.5 + 0.455C + 0.701T + 0.0024F + 0.433t + 0.00689C^2 - 0.0092T^2 - \\ & 0.0094t^2 + 0.00732C \times T + 0.0052C \times t + 0.00559T \times t \end{aligned}$$

$$R^2 = 0.99 \quad (6.10)$$

Since CCRD designs are statistics-based experimental optimization models, they rely on carrying out a minimum number of experiments. For example, the 4 factor at five levels CCRD design (Table 6.1) makes use of only 31 test runs while a full factorial design would require 625 experimental conditions. Hence, it is necessary to develop response surface model equations like those presented above (Eqs. 6.8 – 6.10)

before attempting to model process kinetics using diffusion or Azuara models. These models can be used to generate time specific moisture loss and solids gain under different experimental conditions.

6.3.2 Azuara model and equilibrium values

Moisture loss and solids gain values at different contact times were fitted to the empirical Azuara model Eqs. (6.2 and 6.3) , and equilibrium moisture loss and equilibrium solids gain were obtained as reciprocal slopes of t/ML vs. t and t/SG vs. t plots for each osmotic drying condition and the intercepts were used to compute the second Azuara parameter (S_1 or S_2). A sample t/ML vs. t plot has been shown in Figure 6.1 in order to demonstrate the suitability of Azuara model. The results show a minimum R^2 value 0.91 (often as high as 0.97) making the models quite acceptable (they explain more than 91% of the experimental variability) (Table 6.2). The equilibrium moisture loss increased with temperature (41.4 to 51.9%) and with sucrose concentration (41.7 to 51.8%), but moderately changed with flow rate (46.5 to 48.1%). These values were computed under conditions set at the mid-level for the other two variables; for example, the average effect of temperature is estimated 50°B syrup and 2800 ml/min flow rate (Table 6.2). These results are consistent with the fact that higher temperatures and higher solute concentrations which provide the driving force for osmotic dehydration. This will permit greater amount of moisture to come out at equilibrium and hence will result in a lower equilibrium moisture contents in the sample which translates to a higher equilibrium moisture loss. The flow rate mostly helps to facilitate the equilibrium conditions faster and hence its effect is mostly minimal.

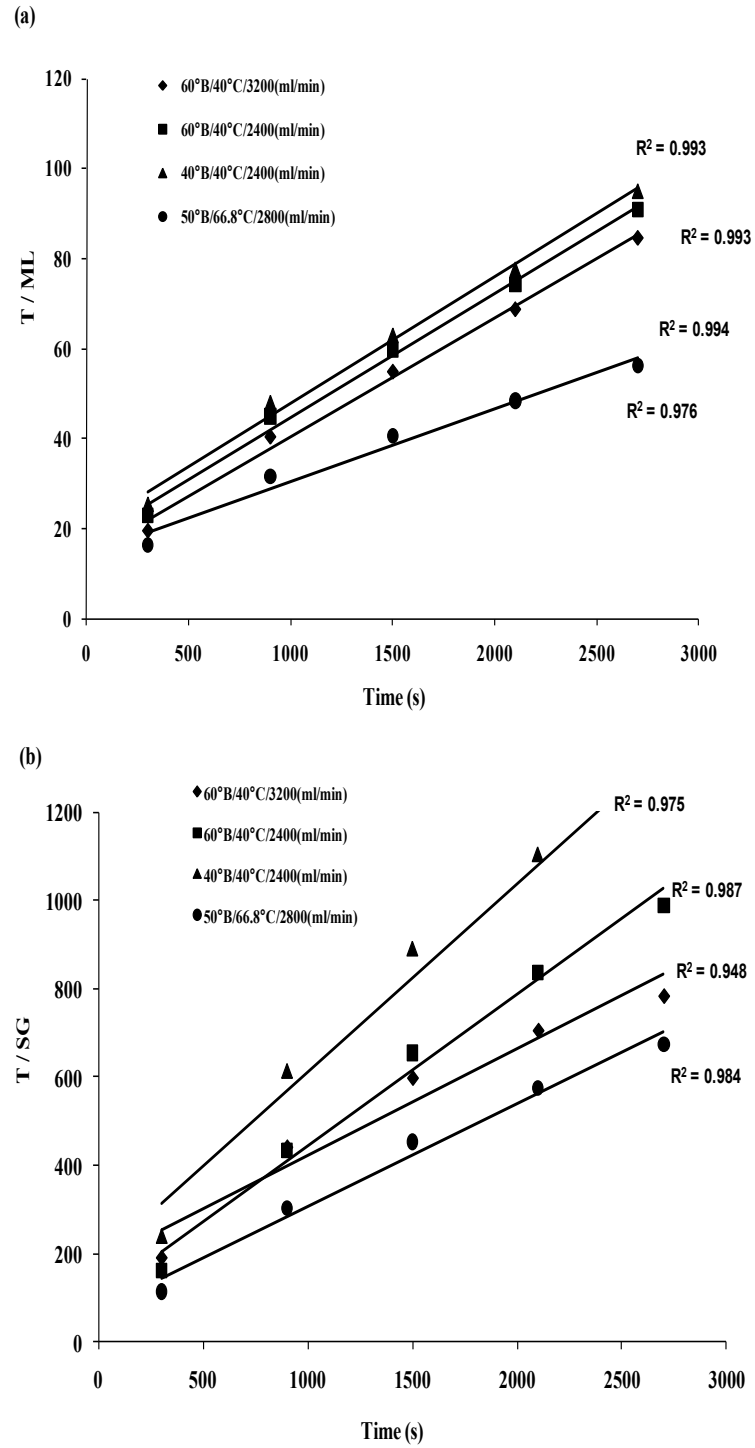


Figure 6.1 Linear plots of Azuara model for determination of ML_e (a) and SG_e (b) at different conditions

Table 6.2 Azuara model parameters and equilibrium values for moisture loss and solids gain during MWODS of apples at different conditions

Sucrose concentration (°B)	Temperature (°C)	Flow rate (ml/min)	ML _e	S ₁ *10 ⁻³	R ²	SG _e	S ₂ *10 ⁻³	R ²
50	40	2800	41.4	1.55	0.99	3.52	2.10	0.97
50	50	2800	47.9	1.54	0.98	3.81	2.44	0.97
50	60	2800	51.9	1.47	0.96	4.10	3.71	0.97
40	50	2800	41.7	1.11	0.99	3.38	1.87	0.96
50	50	2800	47.9	1.54	0.97	3.80	2.44	0.97
60	50	2800	51.8	1.58	0.98	3.92	2.59	0.97
50	50	2400	46.5	1.41	0.97	3.11	3.7	0.93
50	50	2800	47.9	1.54	0.98	3.81	2.44	0.97
50	50	3200	48.1	1.65	0.96	4.27	1.55	0.96

For solids gain at equilibrium, the temperature and sucrose concentration effects (Table 6.2) were similar to those observed with moisture loss at equilibrium (but more moderate). The mean values of equilibrium solids gain increase from 3.52 to 4.1% (temperature effect) and 3.38 to 3.92% (concentration effect). However, the flow rate had a much higher effect on the equilibrium solids gain with the mean value increasing from 3.10 to 4.27% in the same range of experiments demonstrating a deviation from the moisture loss behavior. As with equilibrium moisture, an increase in equilibrium solids gain was observed at higher solute concentrations and temperatures because of the increased partial pressure gradient between the sample and osmotic medium (Khin, Zhou & Yeo, 2007). The higher equilibrium solids gain at higher flow rates indicates the possibility of a secondary mechanism, like for example capillary flow, for solids gain in addition to the diffusion.

The prediction of transient moisture loss and solids gain within the range of experimental conditions based on Azuara model are shown in Figure 6.2 (a-d) at selected conditions. The smooth curves demonstrate the usefulness of such simple models for estimating mass transfer pattern during the dynamic period of osmotic

drying. The figures also demonstrate the common trends with moisture loss and solids gain favored by increasing sucrose concentration, temperature and flow (Table 6.2).

The two Azuara model coefficients can be related to the process variables by fitting a second order polynomial model by using data through an expanded Table 6.2 to different levels of a CCRD design (shown in detail for the diffusivity parameters in the next section). Equations (6.11)-(6.14) provide such models for equilibrium moisture loss and equilibrium solids gain, as well as the rate parameters obtained for conditions within the experimental range:

$$ML_e = -28.1 + 0.406C + 1.38T + 0.0014F - 0.00504C^2 - 0.0059T^2 - 0.0034CT \quad R^2=0.99 \quad (6.11)$$

$$SG_e = -912 + 0.286C + 0.035T + 0.0012F - 0.0025C^2 \quad R^2=0.88 \quad (6.12)$$

$$S_1 = 0.285 + 0.07C - 0.000267T + 0.00026F - 0.00059C^2 \quad R^2 = 0.95 \quad (6.13)$$

$$S_2 = -32.82 + 0.384C + 0.019F - 0.0036C^2 - 0.0000037F^2 \quad R^2 = 0.65 \quad (6.14)$$

With these models the equilibrium ML and SG values and the intercept coefficients can be computed for any set levels (within the experimental range) of sucrose concentration, temperature and flow rate, and then the transient ML and SG gain can be predicted using Eqs. (6.2) and (6.3). The associated high R^2 values for the above models were generally high for moisture loss predictions while they were slightly lower with solids gain. Most osmotic drying studies show that solids gain data inherently have a high variability due to different practices used in the osmotic drying research. The solids gain range is also generally small relative to the moisture loss range making them relatively more sensitive.

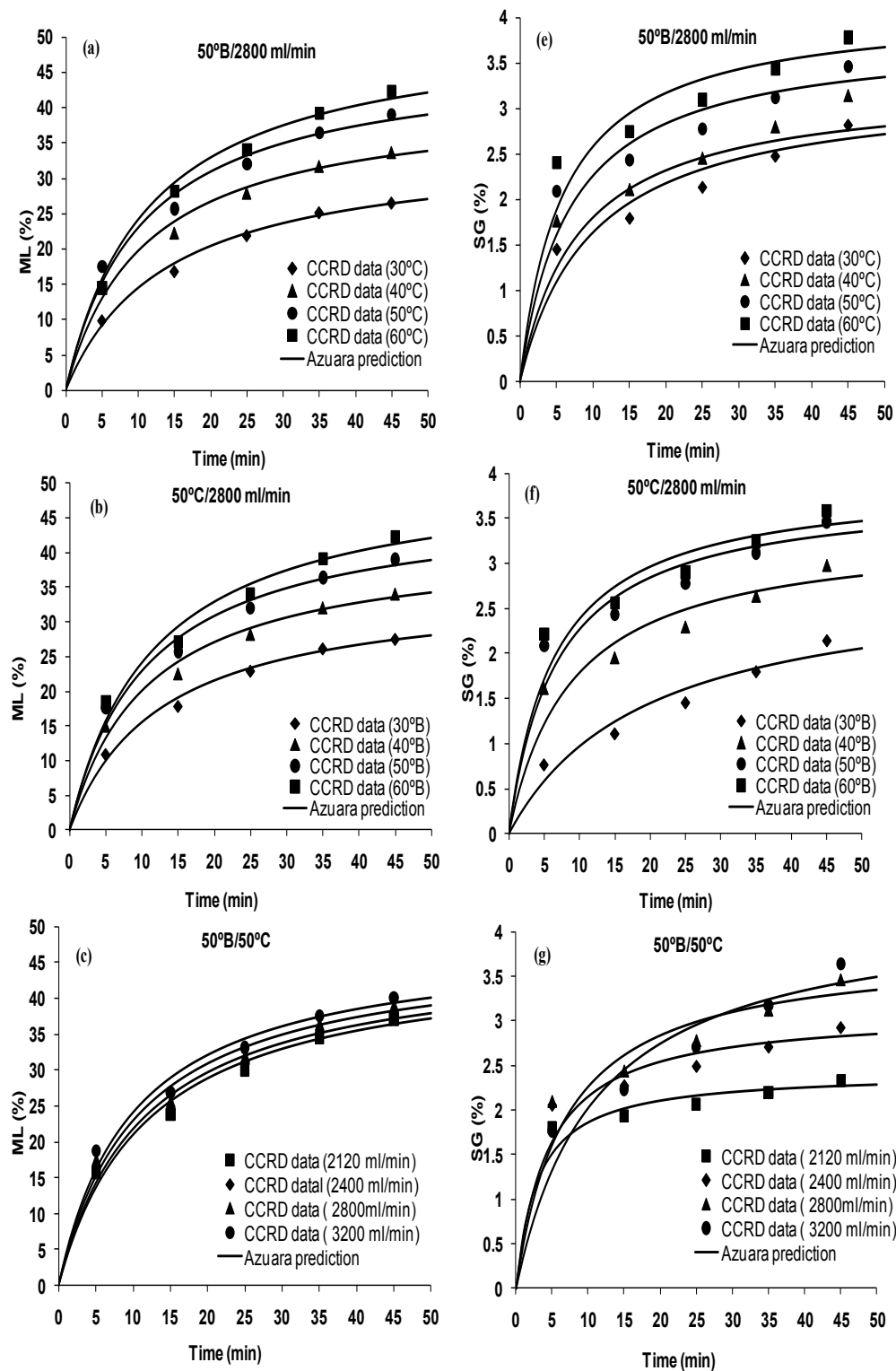


Figure 6.2 Performance of Azuara model (predicted vs. experimental) for (a,b,c) moisture loss(%ML) and (e,f,g) solids gain (%SG) at different conditions

6.3.3 Diffusion Model

One of the most widely studied kinetic parameter on osmotic dehydration is the effective diffusivity (D). Normally, together with D and the associated processing conditions, it is possible to model the mass transfer kinetics provided the equilibrium values of moisture loss and solids gain are known or predicted. In the present study, these were obtained from the Azuara model. Knowing the equilibrium values, the moisture loss and solids gain data (Eqs. 6.8-6.10) can be fitted to the diffusion model. The effective diffusion coefficients of moisture loss and solids gain, D_m and D_s , are computed from the slopes of logarithms of residual moisture ratio and solids fraction vs. time (Eqs. 6.4 - 6.6). The linearized plots of the diffusion model are shown in Figure 6.3 and data for selected conditions are presented in Table 6.3 demonstrating a good fit with fairly high $R^2 > 0.92$ values.

Table 6.3 Experimental design of process in coded^a and actual variables and values of predicted data for microwave osmotic dehydration under spray

Exp.	Sucrose concentration (°B)	Temperature (°C)	Flow rate (ml/min)	D_m (10^{-9}) m ² /s	D_s (10^{-9}) m ² /s
1	40(-1)	40(-1)	2400(-1)	2.43	3.80
2	60(+1)	40(-1)	2400(-1)	2.62	4.17
3	40(-1)	60(+1)	2400(-1)	2.63	4.18
4	60(+1)	60(+1)	2400(-1)	3.04	4.38
5	40(-1)	40(-1)	3200(+1)	2.71	3.30
6	60(+1)	40(-1)	3200(+1)	2.83	3.17
7	40(-1)	60(+1)	3200(+1)	2.87	3.17
8	60(+1)	60(+1)	3200(+1)	3.23	4.03
9	33(-1.68)	50(0)	2800(0)	2.55	3.08
10	66(1.68)	50(0)	2800(0)	3.04	3.86
11	50(0)	33(-1.68)	2800(0)	2.24	3.54
12	50(0)	66(+1.68)	2800(0)	2.89	4.06
13	50(0)	50(0)	2127(-1.68)	2.85	3.28
14	50(0)	50(0)	3472(+1.68)	3.21	2.70
15	50(0)	50(0)	2800(0)	3.04	3.81
16	50(0)	50(0)	2800(0)	3.45	4.40
17	50(0)	50(0)	2800(0)	3.12	4.24
18	50(0)	50(0)	2800(0)	3.34	4.59
19	50(0)	50(0)	2800(0)	3.24	4.87
20	50(0)	50(0)	2800(0)	3.20	4.56
21	50(0)	50(0)	2800(0)	3.26	4.28

^a Code 0 is for center point, ± 1 for factorial points, and ± 1.68 for augmented points

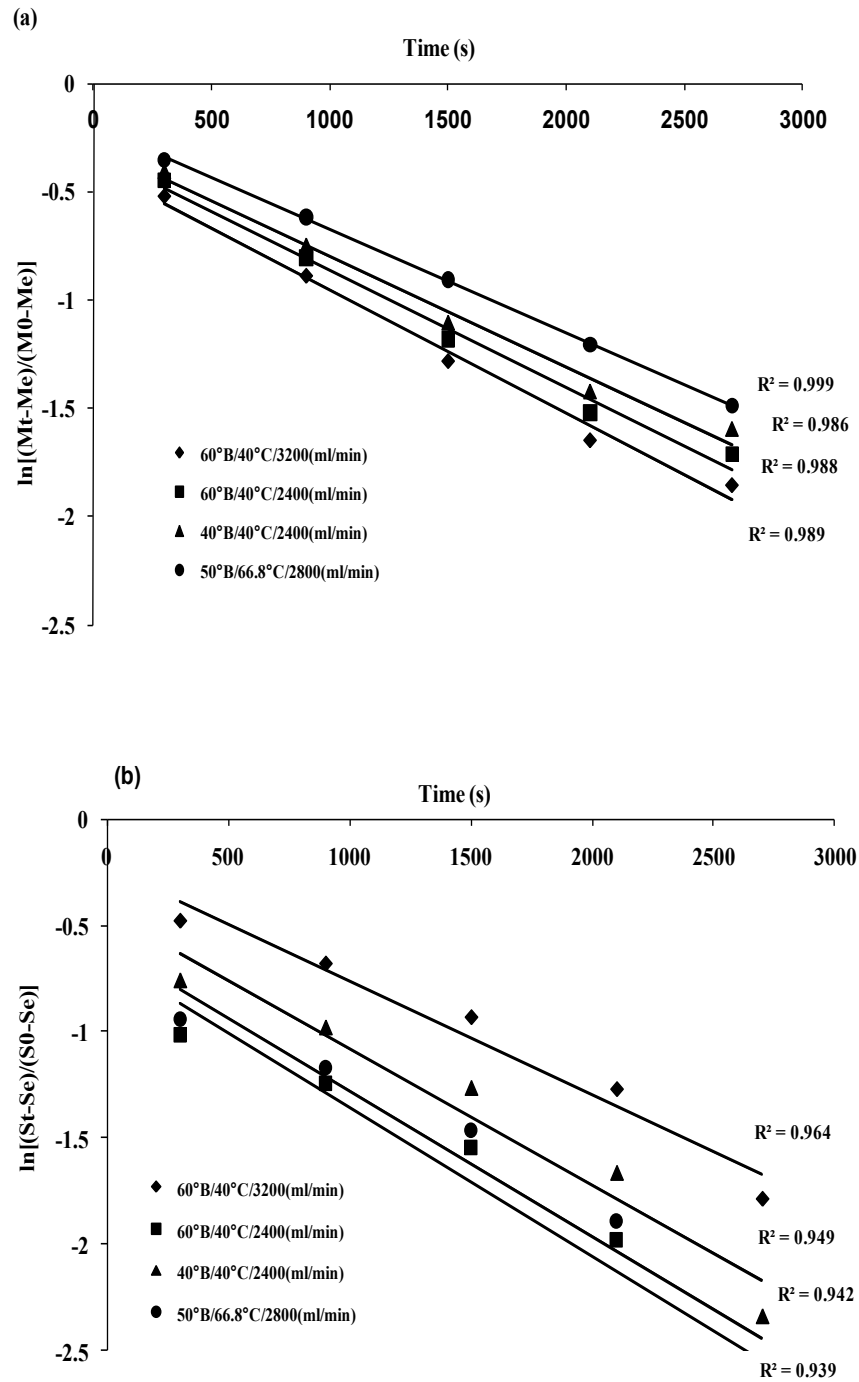


Figure 6.3 Residual moisture loss ratio (a) and solids gain ratio (b) as a function of contact time during MWOD at different conditions

Second-order polynomial response surface models were also fitted for both diffusivity coefficients (moisture loss and solids gain) as a function of process variables. The sum of squares of the sequential model was analyzed to check how the variability of moisture diffusivity (D_m) and solid diffusivity (D_s) were accommodated. The accompanying ANOVA results (Table 6.4) demonstrated that a second order quadratic model well described the relationship between the diffusivity coefficients and process variables.

Table 6.4 Analysis of variance (ANOVA) for the fit of experiment data to response surface model

Source	Moisture diffusivity			Solid diffusivity		
	m ² /s			m ² /s		
	Sum of squares	DF	p	Sum of squares	DF	p
Model	1.760	6	< 0.0001***	4.66	4	0.0131*
Linear						
C	0.264	1	0.0002**	0.5	1	NS
T	0.382	1	< 0.0001***			NS
F	0.169	1	0.0013**	1.07	1	0.013*
Quadratic						
C.C	0.343	1	< 0.0001***	0.82	1	0.026*
T.T	0.707	1	< 0.0001***			NS
F.F	0.040	1	0.0243*	2.44	1	0.0006**
Interaction						
C.T			NS			NS
C.F			NS			NS
T.F			NS			NS
Statistic analysis for the model after backward elimination						
Lack of fit	0.037	8	0.849NS	1.51	10	0.371 NS
R-squared	0.947			0.681		
Adj R-squared	0.924			0.602		
Pred R-squared	0.901			0.443		
Adeq precision	19.2			8.919		

C, T and F are sucrose concentration (°Brix), process temperature (°C) and flow rate (ml/min).

** Significant within a 99%confidence interval.* Significant within a 95% confidence interval.

NS: Non significant

Again, the associated R^2 value was especially high for D_m (0.947) while D_s had a somewhat lower R^2 value of 0.681. The lack-of-fit in both cases was not significant ($P > 0.05$). The "Pred R-Squared" of moisture diffusivity (D_m) was 0.901 was in reasonable agreement with the "Adj R-Squared" of 0.924. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. Here, the ratio was 19.2 indicating an adequate signal. With respect with solid diffusivity (D_s), the "Pred R-Squared" of 0.443 is in reasonable agreement with the "Adj R-Squared" of 0.602. "Adeq Precision" was 8.92 is higher than 4. Therefore, this model can be used to navigate the design space for D_m and D_s . Figure 6.4 shows the comparison between the observed and the model predicted values. The results demonstrate that the polynomial regression models were in good agreement with the experimental data, especially for D_m .

6.3.4 Influence of MWODS on Moisture diffusivity (D_m)

Table 6.4 shows that among the three independent variables, temperature exerted the highest significance on the D_m value ($P < 0.0001$; $SS=0.382$) followed by sucrose concentration ($P < 0.0001$; $SS=0.264$) and flow rate ($P < 0.001$, $SS=0.169$). The quadratic terms of temperature ($P < 0.0001$; $SS=0.811$), sucrose concentration ($P < 0.0001$; $SS=0.343$), and flow rate ($P < 0.05$; $SS=0.068$) were significant. The following polynomial equation describes the relationship between D_m and process variables:

$$D_m = -10.48 + 0.149C + 0.233T + 0.00209F - 0.00135C^2 - 0.00210T^2 - 0.000000323F^2$$

$$R^2 = 0.95 \quad (6.15)$$

Figure 6.5 shows the effect of MWODS process variables on moisture diffusion. The effect of sucrose concentration and temperature on D_m is presented in the Fig 6.5a at a flow rate of 2800 ml/min. It can be observed that the moisture effective diffusivity increased with increasing sucrose concentration and temperature. These effects agree with those previously reported by Allali et al. (2008).

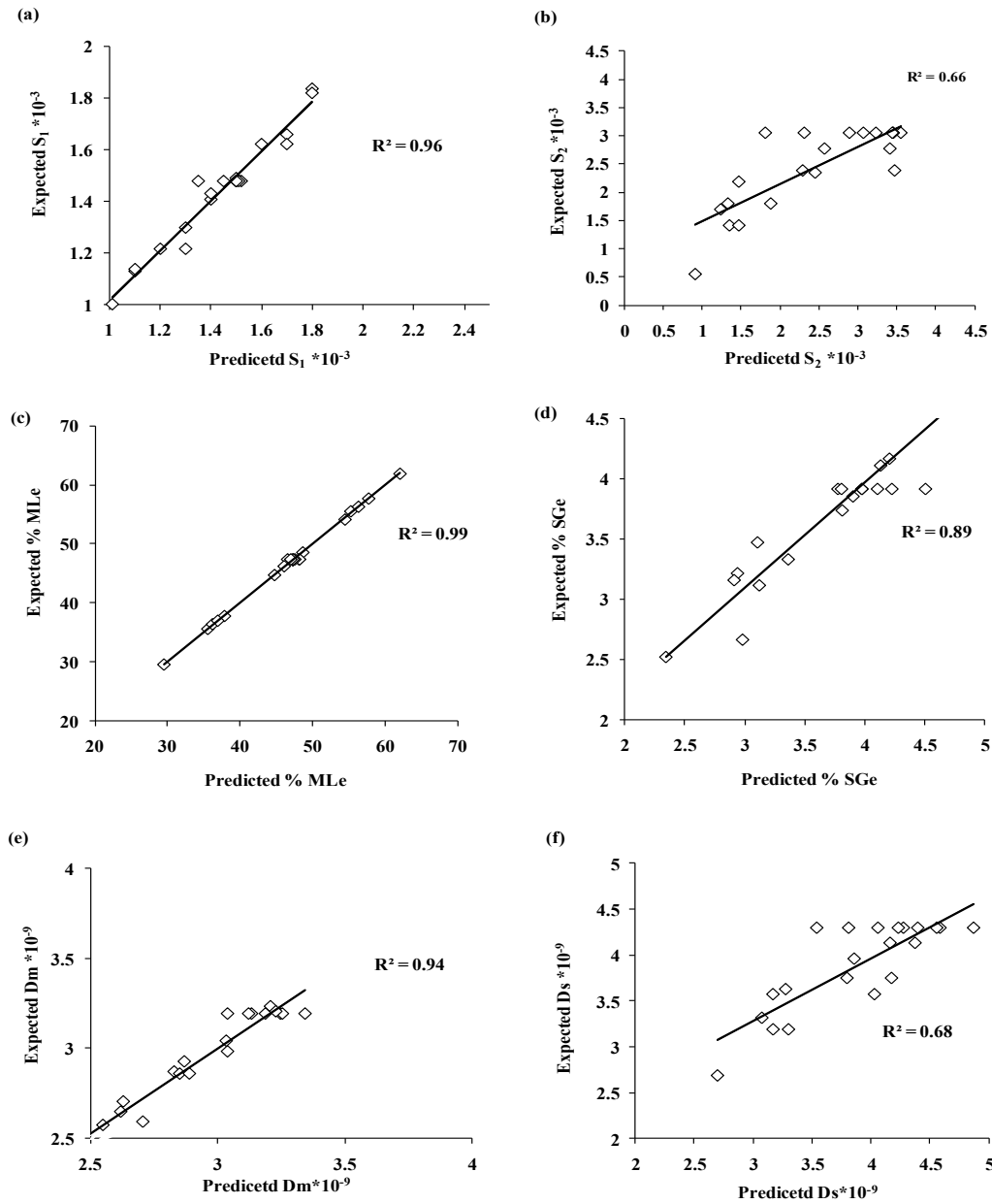


Figure 6.4 Comparison between predicted and expected values of (a) S1 parameter (b) S2 parameter; (c) Moisture loss equilibrium (ML_e);(d) Solids Gain equilibrium (SG_e); (e)Moisture diffusivity (D_m) and (f) Solids diffusivity (D_s)

The effective diffusivity of moisture varied from 2.24×10^{-9} to $3.45 \times 10^{-9} \text{ m}^2/\text{s}$ over the sucrose concentration studied. This order of magnitude is ten times higher than the values reported by Kaymak-Ertekin & Sultanoglu (2000). D_m for dehydrated food

are variable in the range of 10^{-8} to 10^{-12} m²/s due to compositions and physiological of food and experimental procedures used for determining moisture diffusivity (Allali et al., 2008; Corzo, Bracho & Alvarez, 2008).

As can be seen, increasing sucrose concentration leads to an increase D_m , however, at higher sucrose concentrations ($> 50^\circ\text{B}$), D_m increased only slightly and then showed a tendency of decreasing. This is can be explained as the effect of the surface blocking at higher sucrose, which reduces the concentration gradient between the product and osmotic solution, imposing an additional resistance to mass exchange and lowering the rates of moisture loss change (Azarpazhooh & Ramaswamy, 2010b; Eren and Kaymak-Ertekin, 2007; Li & Ramaswamy, 2006b; Ruiz-López et al., 2008).

Increasing temperature generally is expected to result in increasing D_m due to lowering the viscosity of osmotic solution which promotes the water transfer (Azarpazhooh & Ramaswamy, 2010b; Jokic et al., 2007; Li & Ramaswamy, 2006 b). The influence of sucrose concentration and flow rate on moisture loss is shown in Figure 6.5b. The results show that increasing flow rate reduced the mass resistance and increased osmotic pressure gradient therefore D_m , is increased (Li & Ramaswamy, 2006b).

It may not be easy to see the combined effects of three variables through response surface plots. These are usually better described using perturbation plots. The perturbation plot demonstrating the effect different independent variables are shown in Figure 6.6a indicating a convex increasing effect of sucrose concentration and temperature reaching peak values around mid-way between the center and highest concentrations used while the flow rate effect was a bit less convex. The peak values for each variable at the intermediate levels demonstrate some interaction effects at the high ends with other process variables. For example, at higher sucrose concentrations, while the increased viscosity could normally limit the fluid flow at lower temperatures, the viscosity effect diminishes at higher temperature. Similarly flow rate could

positively affect up to a certain level, but at further higher flow rates the diffusion effect could be lowered due to the shorter contact time.

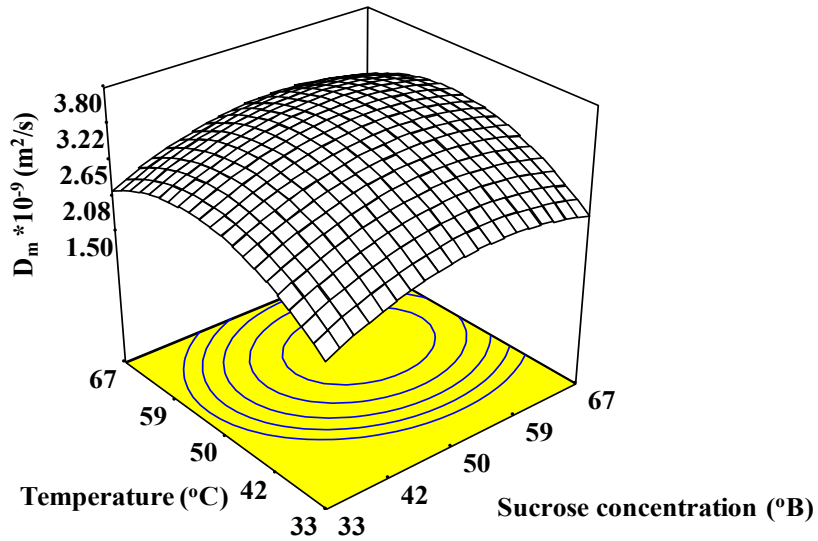
6.3.5 Influence of MWODS on Solid Diffusivity (D_s)

From the results presented in Table 6.4, it can be seen that the flow rate had significant effect on the D_s value ($P < 0.05$, $SS=1.06$) while the linear effects of sucrose concentration and temperature were not significant. The quadratic terms of flow rate ($P < 0.001$; $SS=2.43$), and sucrose concentration ($P < 0.005$; $SS=0.81$) were, however, significant. The second order polynomial model relating D_s to process variables is given below:

$$D_s = -20.24 + 0.2519C - 0.01233F - 0.00233C^2 - 0.0000025F^2 \quad R^2 = 0.68 \quad (6.16)$$

Figure 6.7 represents the effect of MSWOS process variables on solids gain in to the product. Sucrose concentration had a significant effect ($P < 0.05$) on solid diffusivity (Fig 6.7a) while temperature had no effect. Solid diffusivity showed a pattern similar to moisture diffusivity showing an off center peak toward the high end. Figure 6.7b presents the interaction between sucrose concentration and flow rate on D_s . Increasing flow rate generally resulted in increasing D_s , but again peak values were observed at intermediate levels. Increasing flow rate results in decreasing the mass transfer resistance thereby contributing to the increasing of solids gain; however at high flow rates the surface layer of the cell could be blocked leading to decrease the D_s (Eren and Kaymak-Ertekin, 2007). Again, the perturbation plots demonstrate these individual effect plots more clearly (Figure 6.6b).

(a)



(b)

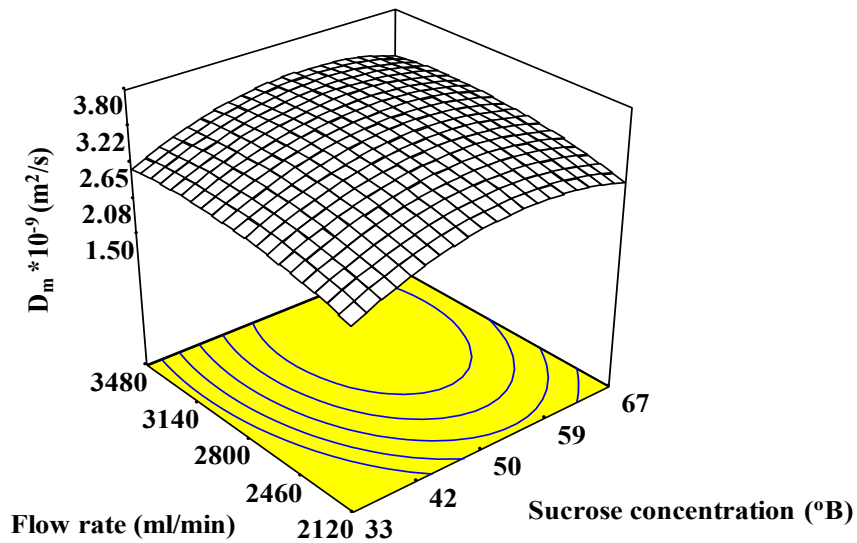


Figure 6.5 Three-dimensional (3D) response surface plots showing the effect of the variable on the response: (a) the effect of sucrose concentration and temperature on the moisture diffusivity (flow rate =2800 ml/min); (b) the effect of sucrose concentration and flow rate on the moisture diffusivity (temperature =50°C)

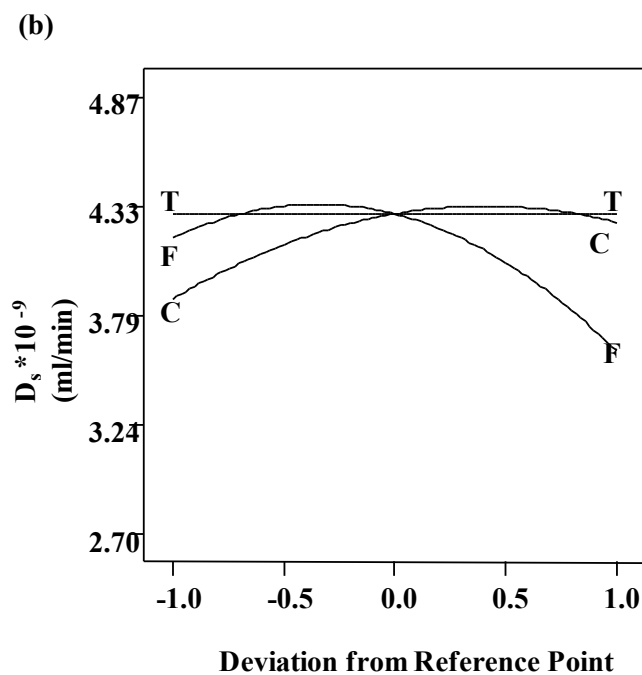
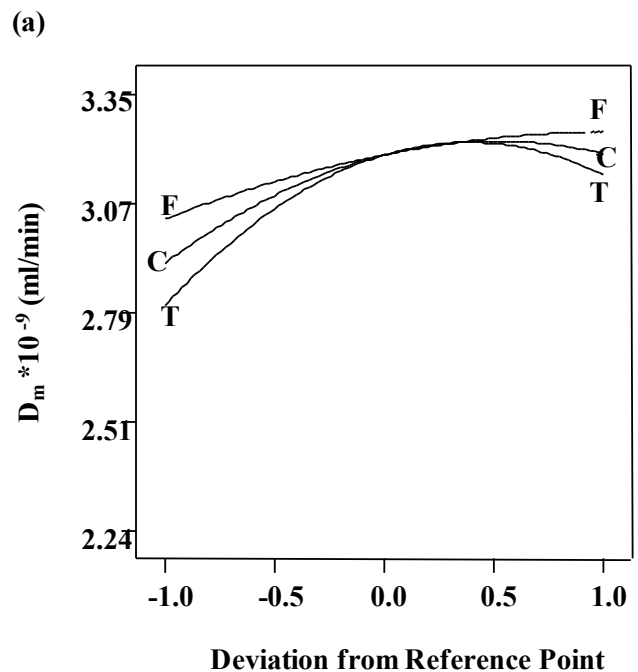


Figure 6.6 Perturbation plot (a) Moisture diffusivity (b) Solid diffusivity; Sucrose concentration=50°B Temperature=50°C and Flow rate=2800 ml/min).C: Sucrose concentration, T: Temperature and F: Flow rate

6.3.6 Influence of MWODS on Solid Diffusivity (D_s)

From the results presented in Table 6.4, it can be seen that the flow rate had significant effect on the D_s value ($P < 0.05$, $SS=1.06$) while the linear effects of sucrose concentration and temperature were not significant. The quadratic terms of flow rate ($P < 0.001$; $SS=2.43$), and sucrose concentration ($P < 0.005$; $SS=0.81$) were, however, significant. The second order polynomial model relating D_s to process variables is given below:

$$D_s = -20.24 + 0.2519C - 0.01233F - 0.00233C^2 - 0.0000025F^2 \quad R^2 = 0.68 \quad (6.16)$$

Figure 6.7 represents the effect of MSWOS process variables on solids gain in to the product. Sucrose concentration had a significant effect ($P < 0.05$) on solid diffusivity (Fig 6.7a) while temperature had no effect. Solid diffusivity showed a pattern similar to moisture diffusivity showing an off center peak toward the high end. Figure 6.7b presents the interaction between sucrose concentration and flow rate on D_s . Increasing flow rate generally resulted in increasing D_s , but again peak values were observed at intermediate levels. Increasing flow rate results in decreasing the mass transfer resistance thereby contributing to the increasing of solids gain; however at high flow rates the surface layer of the cell could be blocked leading to decrease the D_s (Eren and Kaymak-Ertekin, 2007). Again, the perturbation plots demonstrate these individual effect plots more clearly (Figure 6.6b).

6.3.7 Process optimization by desirability functions methodology

In this study, the optimization was applied within the experimental ranges of sucrose concentration, temperature, flow rate and contact time for selected dependent variables to be maximized or minimized either independently or in combination. Second-order polynomial models obtained in this study were utilized for each response in order to determine the specified optimum drying condition. Different scenarios based on economical and industrial constraints were considered. After finding the best solution, a graphical method was applied for mapping the optimum conditions range.

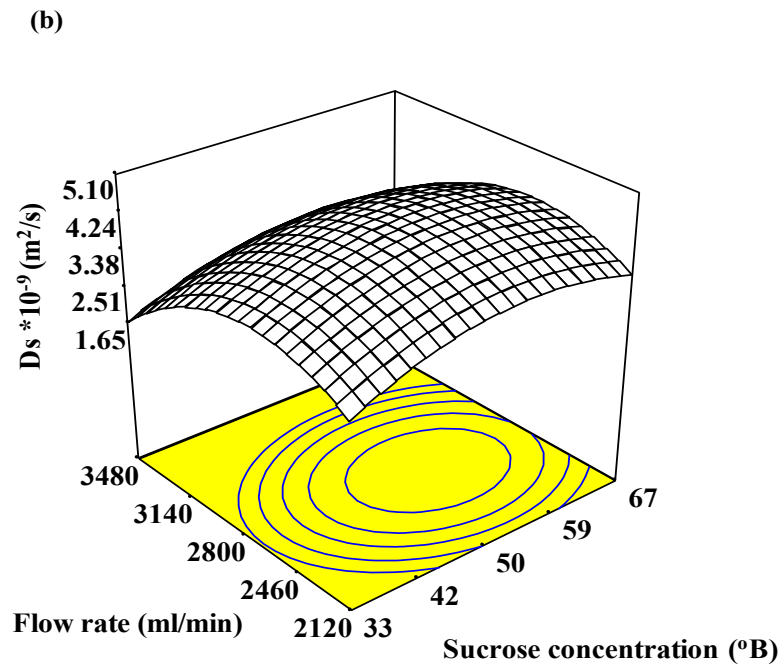
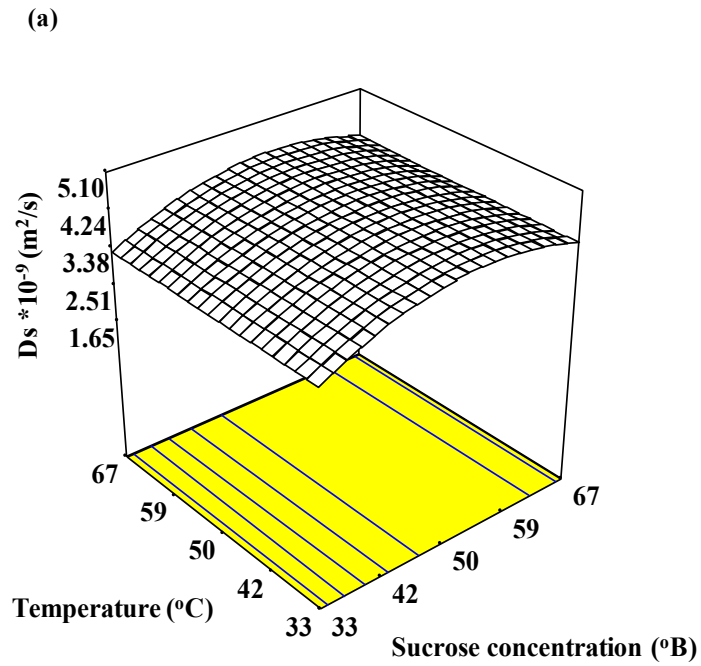


Figure 6.7 Three-dimensional (3D) response surface plots showing the effect of the variable on the response: (a) the effect of sucrose concentration and temperature on the solids diffusivity (flow rate = 3200 ml/min); (b) the effect of sucrose concentration and flow rate on the solids diffusivity (temperature = 50°C)

In the first set of analysis, different constraints for the responses were considered. As can be seen in the Table 6.5, moisture loss was maximized while other parameters were allowed to be in the experimental range (Run 1).

Table 6.5 Results of optimization of different constraints by desirability function

Run	Constraints	Sucrose concentration (°B)	Temperature (°C)	Flow rate (ml/min)	Contact time (min)	ML (%)	SG (%)	WR (%)	Desirability
1	Maximize(ML)	60	59	3193	44	46.62	4.01	42.41	0.987
		59	60	3200	44	46.59	4.01	42.43	0.986
		60	59	3200	43	46.44	3.96	42.28	0.981
2	Minimize(SG)	43	40	2400	15	19.75	1.68	18.01	0.970
		40	40	3200	15	21.05	1.75	19.46	0.939
		40	40	3200	15	21.04	1.75	19.45	0.939
3	Maximize(WR)	60	59	3200	44	46.85	4.01	42.63	0.985
		59	60	3200	43	46.79	4.01	42.60	0.984
		60	59	3200	44	46.76	4.01	42.55	0.982
4	Maximize (ML) and Minimize (SG)	40	47	2400	44	31.77	2.16	29.25	0.608
		40	48	2400	44	32.25	2.22	29.65	0.607
		40	46	2400	45	31.39	2.11	28.91	0.607
5	Maximize (ML) and Maximize (WR)	60	59	3200	44	46.82	4.01	42.60	0.989
		60	60	3146	43	46.75	4.01	42.58	0.987
		60	60	3200	43	46.66	3.97	42.50	0.984
6	Minimize (SG) and Maximize (WR)	40	46	2400	44	31.43	2.12	28.96	0.618
		40	45	2400	45	30.71	2.02	28.33	0.617
		40	46	2403	44	31.30	2.10	28.84	0.617

The results show for this condition, 46.6(%) ML, 4.0 (%) SG and 42.4(%) WR with a high desirability of 0.99. In Run (2), SG was minimized while keeping other variables in the range. In this constraint, again the desirability was very high (0.97), and as can be expected, the SG was the lowest 1.65%; however, the associated ML and WR were too low to be practical value (19.8% ML and 18.0% WL). In Run (3), the weight reduction was maximized. In general, this shows the same trend as maximizing ML. Run (4) is a combination of maximizing ML and WR, and the results show a high moisture loss and weight reduction with a high desirability value. Run (5) and (6) gave mixed results and had much lower desirability values (0.6). Comparing the results of

different runs demonstrate that maximizing weight reduction while keeping other parameters within the range is probably the optimum solution if no other external constraints are necessary. It is important to notice that weight reduction is a combination of moisture loss and solid gain; therefore it can cover both areas. In the final analysis it was considered to maximize moisture loss and weight reductions while minimize the solids gain. Since complete minimization of solids gain would result in poor moisture loss and weight reductions (Runs 2, 4, 6) (Table 6.5). Hence a constraint was added that the solids gain minimum be set at 3.5%. A range of MWODS pre-treatment conditions meeting these constraints are shown in Table 6.6. The treatment time of 30 min at 65°B sucrose concentration, 60 °C, and a flow rate of 2800 ml/min had the best desirability function. Under these constraints, the ML, WR and SG were calculated as 40.9%, 37.7% and 3.32%, respectively (Table 6.6).

6.3.8 Graphical overlay

The optimal conditions imposed by the above conditions as demonstrated in Table 6 can also be visualized graphically as shown in Figure 6.8. The overlaid contours were created using sucrose concentration as the major variable, and shown in combinations with other three variables taking one at a time. For example, Figure 6.8a describes the overlay plot for sucrose concentration and temperature at a flow rate of 2800 ml/min and contact time of 30 min. Figure 6.8b describes a similar plot for sucrose concentration and contact time at the same flow rate (2800 ml/min) but at temperature (60°C) and Figure 8c is plot for sucrose concentration and flow rate at 60°C and 30 min. The optimal zone for a given set of variables has been shown in the shaded area within the overlay plot. The optimal range drawn from the overlay plot was found to be 64-66°B for sucrose concentration, 60-62°C temperature, 30-32 min immersion time and 2800-3000 (ml/min) flow rate.

Table 6.6 Results of optimization by desirability function

Constraints								
Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance		
Sucrose concentration(°B	is target = 65	50	65	1	1	3		
Temperature (°C)	is target = 60	50	65	1	1	3		
Flow rate(ml/min)	is target = 2800	2400	3200	1	1	5		
Contact time(min)	is target = 30	15	45	1	1	5		
ML(%)	maximize	17.74	47.005	1	1	5		
SG(%)	minimize	3.5	4.014	1	1	5		
WR(%)	maximize	15.726	43.041	1	1	5		
Number	Sucrose concentration (°B)	Temperature (°C)	Flow rate (ml/min)	Contact time (min)	ML (%)	SG (%)	WR (%)	Desirability
1	65.000	60.000	2800.002	30.00	40.9	3.32	37.7	0.930
2	65.000	60.000	2800.000	30.07	40.9	3.33	37.7	0.929
3	64.999	60.000	2798.938	30.00	40.9	3.32	37.7	0.929
4	64.981	59.879	2799.994	30.00	40.8	3.32	37.7	0.928
5	65.000	60.000	2800.005	30.74	41.3	3.35	38.1	0.927
6	64.999	60.000	2800.001	30.91	41.4	3.36	38.2	0.927
7	64.595	60.000	2800.002	30.00	40.8	3.33	37.6	0.926
8	65.000	60.000	2799.995	31.20	41.6	3.37	38.3	0.926
9	64.511	60.000	2800.001	30.00	40.8	3.33	37.6	0.926
10	65.000	60.000	2819.813	30.00	40.9	3.33	37.7	0.923
11	65.000	60.744	2799.996	30.00	41.1	3.35	37.9	0.917

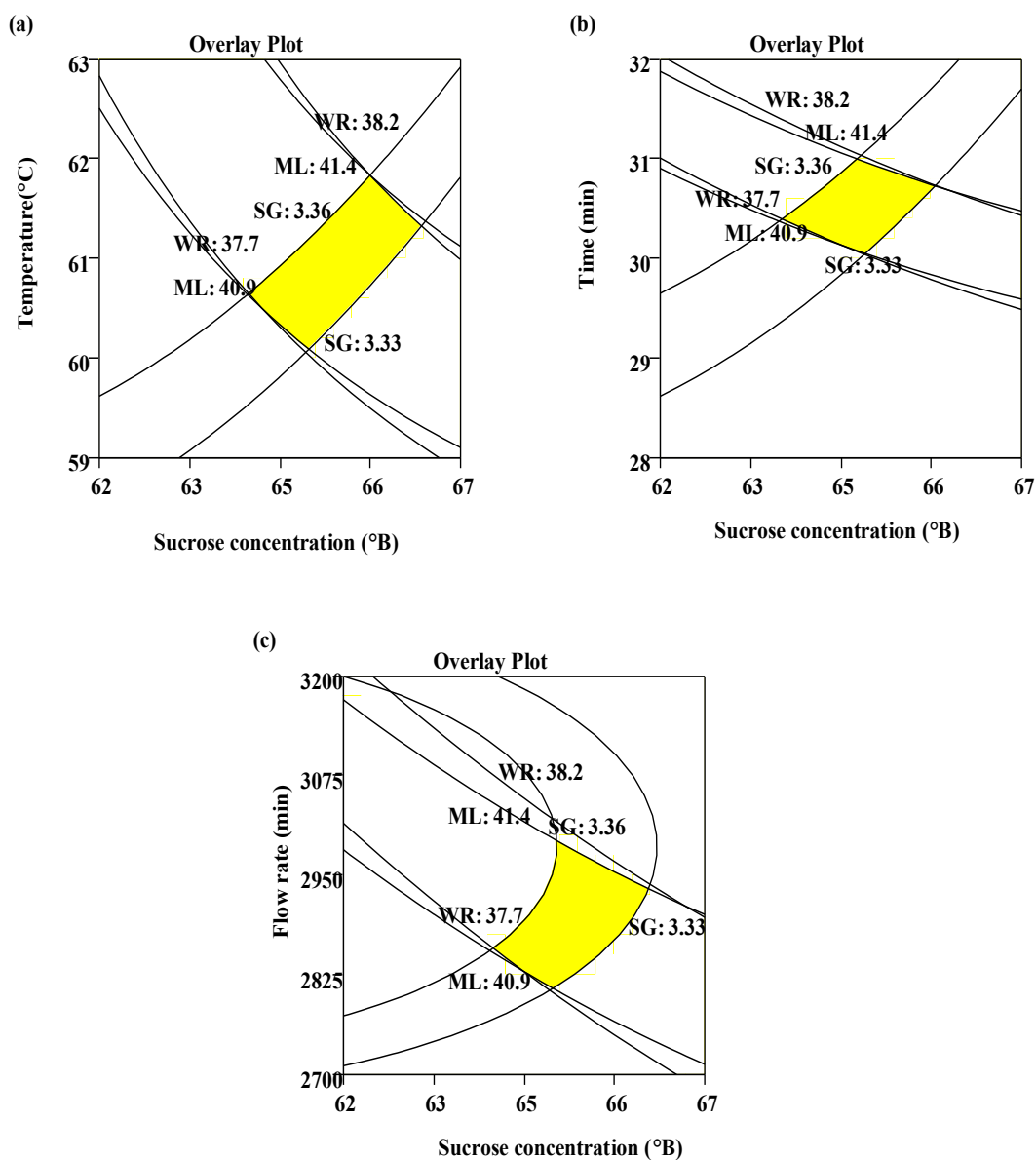


Figure 6.8 The optimum region by overlaying contour plots of the three responses evaluated as a function of (a) sucrose concentration and temperature (at constant Flow rate =2800 ml/min and contact time=30 min); (b) sucrose concentration and contact time (at constant temperature= 60°C and Flow rate=2800 ml/min); (c) sucrose concentration and flow rate (at constant temperature = 60°C and contact time=30 min)

6.4 Conclusions

It was found that the Azuara model adequately describes the equilibrium moisture and solid content and can describe satisfactorily the transient mass transfer kinetics in the osmotic dehydration process of apple cylinder. The diffusion model is used to compute the diffusion coefficients and can be applied for mass transfer prediction. The values of the moisture and solids effective diffusion coefficient were found to be dependent on sucrose concentration, temperature and flow rate of osmotic solution. The results demonstrated that increasing sucrose concentration, temperature and temperature led to higher moisture diffusivity, but with solids diffusivity the temperature effect was not significant. Response surface methodology was effective in optimizing process parameters for the osmotic dehydration of apple. The desirability function method could be effectively used to assess process optimization under different user identified constraints including maximizing moisture loss, weight reduction and/or minimize solids gain. The program can also be used to arrive at target performances or performances at set levels of process variables. It is necessary to notice that the regression equations applied in this study are only useful within the experimental range and should not be extrapolated.

CONNECTIVE STATEMENT TO CHAPTER 7

In the previous several chapters, a new method - microwave osmotic dehydration under continuous flow medium spray (MWODS) condition was conceived, developed, evaluated and optimized. Osmotic dehydration is a process of partial removal of water; therefore, the osmotically treated product need to be finished dried or further processed by other techniques such as freezing, thermal processing etc. In dehydration applications, several techniques have been employed for this second stage drying: air-drying, freeze-drying, vacuum-drying etc. The OD processing conditions often affect the second stage drying performance. This chapter focuses on the evaluation of the second stage air-drying (the simplest of all such methods) of apples as affected by the MWODS pre-treatment conditions. The results were compared to two other methods without the use of MWODS pre-treatment: air-drying on one side and freeze drying on the other which is generally expected to give the least and most desirable results, respectively.

Based on results from Chapter 7, a manuscript has been accepted for publication.

Azarpazhooh, E and Ramaswamy, HS. 2010. Evaluation of factors influencing microwave osmotic dehydration of apples under continuous flow medium spray (MWODS) conditions during second stage of drying. International Food Engineering. MS #1927 (Accepted).

All experiment work and data analysis were carried out by the candidate under the overall supervision of Dr. HS. Ramaswamy.

CHAPTER 7. EVALUATION OF FACTORS INFLUENCING MICROWAVE OSMOTIC DEHYDRATION OF APPLES UNDER CONTINUOUS FLOW MEDIUM SPRAY (MWODS) CONDITIONS DURING SECOND STAGE OF DRYING

Abstract

The effect of microwave-osmotic dehydration pre-treatment under continuous flow medium spray (MWODS) conditions on the second stage air-drying kinetics of apple (*Red Gala*) cylinders was evaluated. MWODS pre-treatment was carried out using a response surface methodology involving 5-levels of sucrose concentration (33-66.8°B), temperature (33-66.8°C) and contact time (5-55 min). Drying time, coefficient of moisture diffusion (D_m) and coefficient of moisture infusion (I_m) were evaluated as responses and the results were compared with their air-dried (AD) (worst scenario) and freeze-dried (FD) (best scenario) counterparts without the osmotic treatments. The diffusion and infusion coefficients were based on the solution of Fick's diffusion model. Empirical models developed for all response variables were significant ($P \leq 0.001$) and the lack of fit was not significant. MWODS pre-treatments significantly affected the D_m values and reduced the air-drying time of apples by about 30-65% in comparison with untreated apple thereby providing opportunity for better energy savings. On the other hand, the values of I_m during the rehydration process were highest for the freeze-dried samples followed by apples air-dried after MWODS treatment, and the least for the untreated air-dried.

7.1 Introduction

Air drying is one of the most common processes of food preservation in which the solid is exposed to a hot stream of air and moisture evaporates. Although air drying can extend the shelf life of products, it is an energy-intensive operation that uses about 15% of all industrial energy (Fernandes et al., 2006; Rodrigues and Fernandes 2007a). Osmotic dehydration can be used as a pre-treatment to reduce the total processing time and air drying time (Fernandes et al., 2006). It is acknowledged to be an energy-efficient method of partial dehydration, since there is no need for a phase change (Bolin et al.,

1983). Since osmotic dehydration is a mild process compared to hot air drying, it has the ability to reduce the overall energy for further drying; however, osmotic dehydration generally does not produce a product of low moisture content that can be considered shelf-stable. Consequently, the osmotically treated product should be further processed; generally by air, freeze-drying, or vacuum-drying methods (Sankat et al., 1996). Air-drying after osmotic dehydration pre-treatment has been proposed for apples by many researchers (Lenart 1996; Simal et al., 1998; Reppa et al., 1999; Sereno et al., 2001a; Nieto et al., 2004). Published results reveal that osmotic dehydration pretreatment implies structural changes in the samples which can influence the mass transport properties of products during the second stage air drying (Lewicki 1998; Mandala et al., 2005). Microwave-osmotic dehydration under continuous flow medium spray (MWODS) has been suggested as a way to accelerate the water loss from fruit, while reducing the solids gain (Azarpazhooh and Ramaswamy, 2010a). A microwave field can enhance the osmotic pressure between the fruit and its surrounding solution; therefore, the driving force for water removal is increased. Water molecules selectively absorb microwave energy resulting in increased moisture out-flux, while simultaneously limiting transfer of solutes from the solution into the food (Li and Ramaswamy, 2006c; Azarpazhooh and Ramaswamy, 2010a). Knowledge of the drying kinetics of osmotically pre-treated foods is essential to the design and optimization of the second stage drying processes. There are several mathematical models that can be used for simulation of the dehydration process. Fick's law of diffusion is the most common one used for finding the moisture diffusivity of the product (Srikiatden and Roberts, 2006). In addition, dehydrated products are normally rehydrated before consumption, and hence the kinetics of rehydration can be used to develop a drying method (Azzouz et al., 2002). The rate of moisture pick-up during rehydration can also provide some information about the storage stability of the product.

The objectives of this research were to evaluate the second stage air-drying mass transfer kinetics of apples after MWODS pre-treatment and compare them with similar air-dried (AD) and freeze-dried (FD) products without the pre-treatment, to evaluate and model the moisture diffusion coefficients during air-drying as a function of MWODS pre-

treatment variables, and to likewise evaluate moisture infusion coefficients during rehydration.

7.2 Materials and Methods

7.2.1 Preparation of samples

Apples (*Red Gala*) were purchased from a local market in Montreal, Canada and stored in a refrigerator at 2-5°C and 95% relative humidity until use (2 to 3 days). Commercial sucrose (Redpath Canada Ltd., Montreal, QC) was used as the osmotic agent. Apples were brought overnight to room temperature before coring and cutting them into cylinders (14 mm height × 14 mm diameter) with a cork borer and a knife. The moisture content of fresh and MWODS-treated as well as dried apples was determined gravimetrically using the AOAC method (AOAC, 2000). The experiments were replicated three times and the average of the moisture ratio at each value was used for drawing the drying curves.

7.2.2 Microwave osmotic dehydration treatment

Batches of apple cylinders that weighed approximately 100 g were tied in a nylon mesh bag to be placed on a perforated platform inside a specially fabricated cylindrical glass chamber, 12.5 cm diameter. A commercial spray device also about 12 cm in diameter (Waterpik CF-151-S, Waterpik Technology Inc., Markham, ON) was attached to the top of the chamber to continuously spray the osmotic medium on the apple samples. The glass chamber assembly with the sample was placed inside a domestic digital microwave oven (Danby DMW1153BL 0.031 m³, 1100 W nominal power at 2450 MHz). The internal dimensions of the oven were 35 × 35 × 21.5 cm (additional details in chapter 3). The osmotic solution was circulated through the chamber as a shower from the top which, after contacting the sample, is sucked from the bottom using a peristaltic pump and returned to the spraying device after allowing to flow through a long coil placed in a temperature controlled water bath for temperature equilibration to the operating level. The microwave oven was operated at full power during the treatment.

After each treatment, the oven was turned off and the sample bag was removed, samples were taken out, lightly rinsed in water to remove the excess sugar solution, drained and then placed on a pre-weighed drying basket.

MWODS pre-treatments were carried out using a central composite rotatable design and a response surface methodology at five levels of: sucrose concentration (33-66.8 °B), medium temperature (33-66.8 °C) and medium contact time (5-55 min) with the flow rate of the osmotic medium maintained at 2800 ml/min (Table 7.1). The ratio of fruit/syrup was 1:30, preventing any significant change of syrup concentration during osmotic drying.

7.2.3 Air-drying procedures

Test samples were dried in a domestic dryer (Equi-Flow Food Dehydrator, Marysville, WA) which was modified to achieve cross-flow dehydration using air at 60°C, 15±1% relative humidity and 0.64±0.02 m/s air flow rate (Nsonzi and Ramaswamy, 1998b). According to some literature, the maximum air temperature for drying fruit with no changes in the fruit quality is 60°C (Demirel and Turhan, 2003; Karim and Hawlader, 2005). The temperature, air velocity and relative humidity of the air flowing over the sample tray were measured using an air velocity/relative humidity/temperature meter (Air Velocity Meters, Velocicalc plus. Model 8360, St. Paul, MN, USA). The dryer was warmed up for 1 h before starting the experiment to achieve stable conditions. Apple test samples were spread uniformly in a single layer on a wire mesh screen tray (20 cm² by 10 cm height) suspended from a digital balance (Haus TS4KD MFD, Haus Corporation Florham Park, NJ). The initial mass of the test sample was kept approximately at 100 g in order to maintain constant air conditions. The moisture loss during drying was continuously monitored and recorded at intervals of 15 min until a moisture content of 25% dry base (db) was attained. The target weight of dried samples was calculated based on the initial mass and moisture content of the test samples. To determine the equilibrium moisture content, a second batch of apple samples

was left in the oven and monitored gravimetrically until three constant consecutive weights were obtained (24-28 h).

7.2.4 Freeze- drying (FD)

The freeze-drying was used to obtained samples for comparative purposes (quality and rehydration characteristics). For freeze drying, a 100 g batch of fresh apple cylinders (14mm diameter and 14mm height) were weighed and placed in a freeze dryer (Thermo Savant, MODULYOD-115, Holbrook, NY, USA) with temperature of -45°C and a vacuum of 100-120 mbar for 20-24 h. Initial and final masses were determined by weighing samples to reach the final moisture of 25% dry basis (db).

7.2.5 Mathematical model

The influence of different osmotic pre-treatment conditions on the second stage of drying was evaluated using mass transfer kinetic parameter, diffusion coefficient, and the drying time required to achieve a final moisture content of 25% (dry basis, db). In order to do this, first transient moisture ratio (MR) was calculated using the following equation:

$$\text{MR} = \frac{X - X_e}{X_0 - X_e} \quad (7.1)$$

MR is the dimensionless moisture ratio; X the moisture content at any time t (kg/kg dry solid); X_0 the initial moisture content (kg/kg dry solid), and X_e the equilibrium moisture content (kg/kg dry solid). The MR was plotted against the air drying time to obtain the drying curves.

7.2.6 Determination of the coefficient of moisture diffusion (D_m)

In order to determine the coefficient of moisture diffusion (D_m) in the apples during air-drying, the drying curve in the form of moisture ratio vs. time was used. A diffusion model based on heat mass transfer analogy (Ramaswamy et al., 1982) was used

for the diffusion controlled mass transfer to estimate the effective moisture diffusivity of dried apple cylinders. The following is the modified formula for the transient mass average moisture content change in a finite cylinder as a function of time (Ramaswamy and Van Nieuwenhuijzen, 2002):

$$\frac{M_e x_e - M_t x_t}{M_e x_e - M_0 x_0} = 0.56 e^{-\frac{8.25}{a^2} D_m t} \quad (7.2)$$

M_0 , M_t and M_e represent the sample mass (kg), x_0 , x_t and x_e the moisture content (g/g) at time zero, t and equilibrium conditions, respectively, a the radius of the sample in m, t the drying time in second and D_m the coefficient of moisture diffusion in m^2/s . In this model, D_m was obtained from plotting the experimental drying data in terms of

$\ln \frac{M_e x_e - M_t x_t}{M_e x_e - M_0 x_0}$ versus drying time which gives the slope:

$$\text{slope} = \frac{8.25}{a^2} \times D_{\text{eff}} \quad (7.3)$$

7.2.7 Rehydration

3 g of dried apple samples (MWODS pre-treated, untreated or freeze-dried) were rehydrated in distilled water maintained at $80 \pm 1.0^\circ\text{C}$ using a temperature controlled water bath (Jambrak et al., 2007). The ratio of the volume of apple cylinder to the rehydration medium (water) was maintained at 1:25. The samples were initially weighed and subjected to rehydration for up to 15 min. Then they were taken out at 1, 3, 5, 7, 9, 12 and 15 min, placed on a filter paper with a slight vacuum for 1 min, and their weights were recorded. Determinations were made in triplicates. The moisture content of the apple after rehydration was determined using the AOAC procedure (AOAC 2000).

7.2.8 Determination of the coefficient of moisture infusion (I_m)

Fick's second law was used to find the coefficient of moisture infusion (I_m), and moisture gain during rehydration was calculated by using an inverse diffusion model. The

two-adjustable-parameter Azuara's model (Azuara et al., 1992) was used (Eq. 7.4) for predicting moisture pick-up after rehydration.

$$MG_t = \frac{S_1(MG_e)}{1 + S_1 t} = \frac{t(MG_e)}{\frac{1}{S_1} + t} \quad (7.4)$$

Equation (7.4) can be linearized as:

$$\frac{t}{MG_t} = \frac{1}{S_1(MG_e)} + \frac{t}{MG_e} \quad (7.5)$$

MG_t is the moisture gain fraction at any time, t , S_1 a constant related to the rate of water infusion from the product, and MG_e the moisture gain fraction at equilibrium. The equilibrium moisture gain, MG_e , can be obtained as the reciprocal slopes of t/MG_t against reciprocal of time. Using the equilibrium moisture loss (MG_e) the coefficient of moisture infusion was obtained from Eq. (7.6) (Ramaswamy and Van Nieuwenhuijzen, 2002).

$$\frac{M_e x_e - M_t x_t}{M_e x_e - M_0 x_0} = 0.56e^{-\frac{8.25}{a^2} I_m t} \quad (7.6)$$

7.2.9 Experimental design and statistical analysis

A response surface methodology (RSM) was used to determine the effect of independent variables (sucrose concentration, temperature, and contact time) on the drying time, the coefficient of moisture diffusion (D_m) and the coefficient of moisture infusion (I_m). For designing the experimental data, a central composite rotatable design (CCRD) including 20 experiments formed by 6 central points and 6 ($\lambda = 1.68$) axial points was employed (Table 7.1). Process variable ranges were established by means of preliminary experiments (Azarpazhooh and Ramaswamy, 2010a). A commercial statistical package, Design-Expert version 6.01 (Statease Inc., Minneapolis, MN) was applied to calculate the RSM. These values were related to the coded variables (x_i , $i = 1, 2$, and 3) by a second order polynomial using the equation below:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{11} X_1^2 + b_{22} X_2^2 + b_{33} X_3^2 + b_{12} X_1X_2 + b_{13} X_1X_3 + b_{23} X_2X_3 \quad (7.7)$$

The coefficients of the polynomial model were represented by b_0 (constant term), b_1 , b_2 and b_3 (linear effects), b_{11} , b_{22} and b_{33} (quadratic effects), and b_{12} , b_{13} and b_{23} (interaction effects). The statistical significance of the terms in the regression equations was examined by analysis of variance (ANOVA) for each response. The adequacy of the model was checked by the coefficient of determination, R^2 , adjusted- R^2 and coefficient variation (CV) (Myers and Montgomery, 2002).

7.3 Results and discussion

7.3.1 Dehydration kinetics

Typical drying curves under selected experimental conditions are shown in Figure 7.1 as transient moisture content vs. time. These conditions were selected from Table 7.1 to demonstrate some general trends with respect to the process variables. Figure 7.1a shows the air drying curves for samples MWODS treated at different sucrose concentrations with the osmotic medium temperature at 50°C and medium contact time of 30 min. Figure 7.1b shows similar trends with respect to different temperatures at the midlevel sucrose concentration (50°B) and contact time of 30 min and Figure 7.1c shows the trends with respect to the different osmotic contact times at 50°B and 50°C. In each case the drying curve for the untreated control is also included for comparison. From each sub-figure (7.1 a, b and c), it can be easily realized that at each level of MWODS treatment, the drying pattern is fairly smooth and somewhat similar, except that the curves progressively shifted downwards at higher levels of each osmotic parameter. The control curve without the MWODS treatment was always at the top of the curves for the MWODS treated samples. This is primarily because the control sample had the highest initial moisture content. The moisture content of the MWODS treated samples progressively decreased as the osmotic treatment severity increased for each osmotic treatment variable, and hence they were initiated at progressively at lower moisture levels (the primary reason for the downward shifting of the curves). These curves also generally

indicated the typical falling rate drying behavior with rate of moisture removal steadily decreasing with time. Such a behavior has been confirmed in food products by several studies (Tan et al., 2001; Jambrak et al., 2007; Gachovska et al., 2008; Doymaz, 2009). This is typical of diffusion controlled moisture transport behavior which would give a semi-logarithmic relationship between residual moisture content and time.

For each of the above treatment conditions (Table 7.1), the time required to reduce the moisture content to the target 25% (db) level was experimentally obtained (from drying curves shown typically in Figure 7.1 and the drying times obtained are summarized in the same table. These drying times were then related to the osmotic treatment variables through the response surface analysis.

Table 7.1 Experimental design of process in coded^a and actual variables and values of experimental data for microwave osmotic dehydration under spray mode

Experiment No	Sucrose concentration (°B)	Temperature (°C)	Contact time (min)	M _i (Kg/Kg dry matter)	M _{eq} (Kg/Kg dry matter)	Drying time (min)	D _m (10 ⁻¹⁰) m ² /s
1	40(-1)	40(-1)	15(-1)	5.16	0.057	345	7.9
2	60(+1)	40(-1)	15(-1)	3.97	0.056	360	8.19
3	40(-1)	60(+1)	15(-1)	4.05	0.06	315	9.5
4	60(+1)	60(+1)	15(-1)	3.89	0.08	285	7.71
5	40(-1)	40(-1)	45(+1)	2.94	0.047	225	11.7
6	60(+1)	40(-1)	45(+1)	2.37	0.06	210	11.6
7	40(-1)	60(+1)	45(+1)	2.6	0.054	255	13.3
8	60(+1)	60(+1)	45(+1)	2.64	0.06	210	13.3
9	33(-1.68)	50(0)	30(0)	3.66	0.072	315	10.3
10	67(+1.68)	50(0)	30(0)	1.09	0.064	180	13.5
11	50(0)	33(-1.68)	30(0)	3.75	0.049	270	10.8
12	50(0)	67(+1.68)	30(0)	1.69	0.043	195	10.8
13	50(0)	50(0)	5(-1.68)	5.13	0.069	440	6.8
14	50(0)	50(0)	55(+1.68)	1.43	0.067	195	10.5
15	50(0)	50(0)	30(0)	2.57	0.054	240	11
16	50(0)	50(0)	30(0)	2.11	0.051	220	12.4
17	50(0)	50(0)	30(0)	2.13	0.054	185	13.5
18	50(0)	50(0)	30(0)	1.18	0.046	240	14.4
19	50(0)	50(0)	30(0)	2.18	0.055	230	13.6
20	50(0)	50(0)	30(0)	2.12	0.059	211	12.4

^a Code 0 is for center point of the parameter range investigated, ±1 for factorial points

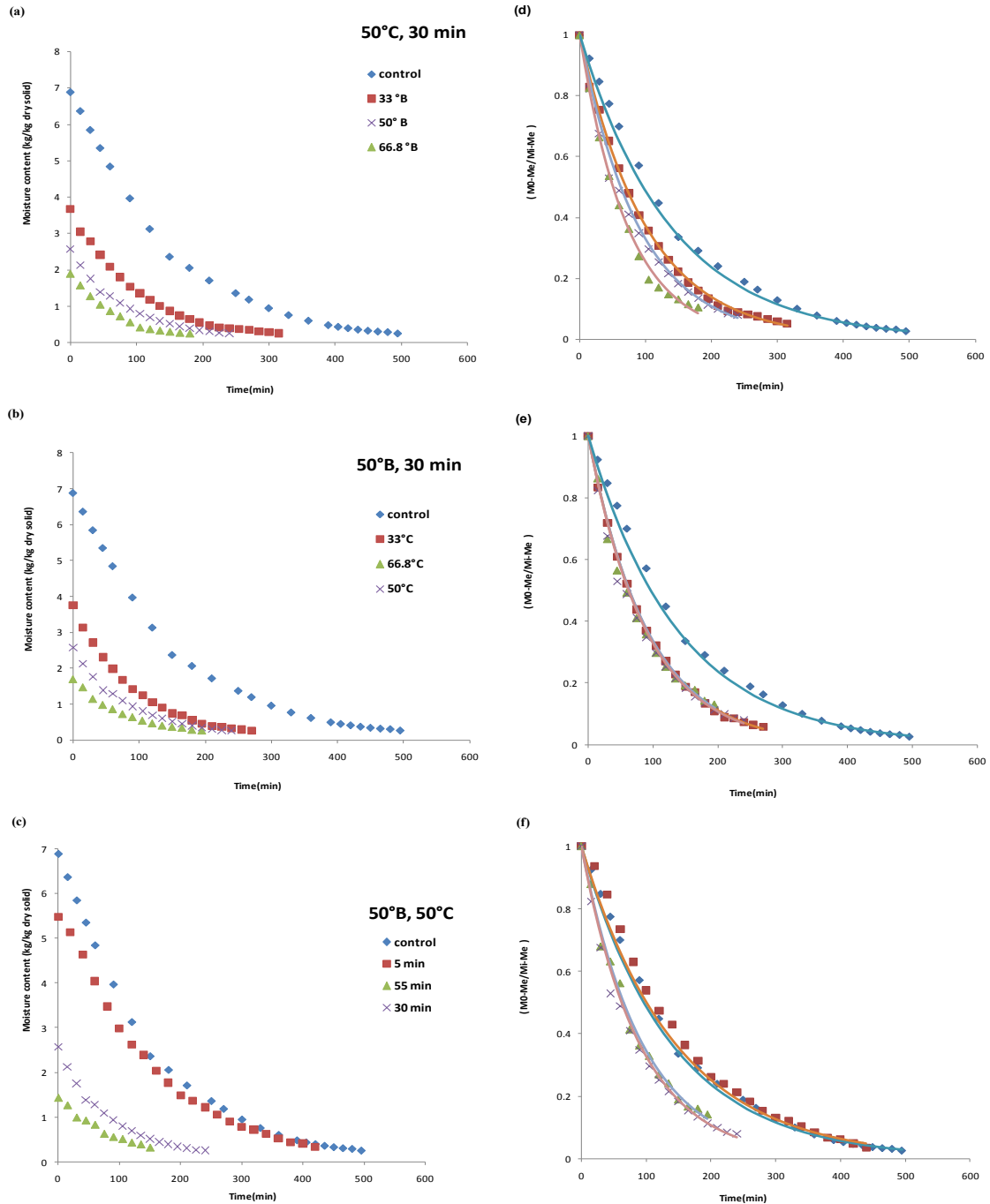


Figure 7.1 Experimental drying curves (a,b,c) (points) and moisture ratio (d,e,f) for with and without (control) MWODS pre-treated apple at (a,d) different sucrose concentration (medium temperature, 50°C, contact time, 30 min); (b,e) different temperatures (sucrose concentration, 50°B, contact time, 30 min); and (c,f) different contact times (sucrose concentration, 50°B, Temperature, 50°C). Lines show model predictions

7.3.2 Effect of osmotic treatment process variables on drying time

From the ANOVA (Table 7.2), it can be observed that the regression model for the drying time as a function of osmotic variables was significant ($P \leq 0.0001$). The P -values indicated that the linear effect of sucrose concentration and contact time, and the quadratic effect of contact time were significant ($P < 0.001$), while interaction effects of independent variables were not significant ($P > 0.05$) with respect to air drying time. Based on the sum of squares, it can be recognized that the osmotic medium contact time had a greater influence than sucrose concentration.

Figure 7.2 shows the three-dimensional response surface generated by the model and shows the influence of two variables at a time. Drying times associated with MWODS pre-treated apples was clearly lower as compared to with the drying time for untreated control (dash area in Figure 7.2). The air drying time decreased with an increase in sucrose concentration, temperature and contact time of MWODS pre-treatment. These are essentially contributed by the lower initial moisture contents associated with the treated samples. The moisture content of MWODS treated samples was 25-84% lower than that of the untreated samples prior to air drying. There was a clear direct relationship between the drying time and initial moisture content (Figure 7.3). There was also considerable spread in the drying time vs. initial moisture content curve (Figure 7.3) to indicate this is only a general trend and the MWODS treatment conditions themselves also have a considerable role in affecting the air drying time. Longer treatment times in higher concentration sucrose solutions at higher temperatures are conducive to higher moisture loss during the osmotic treatment resulting in lower moisture content product for air drying.

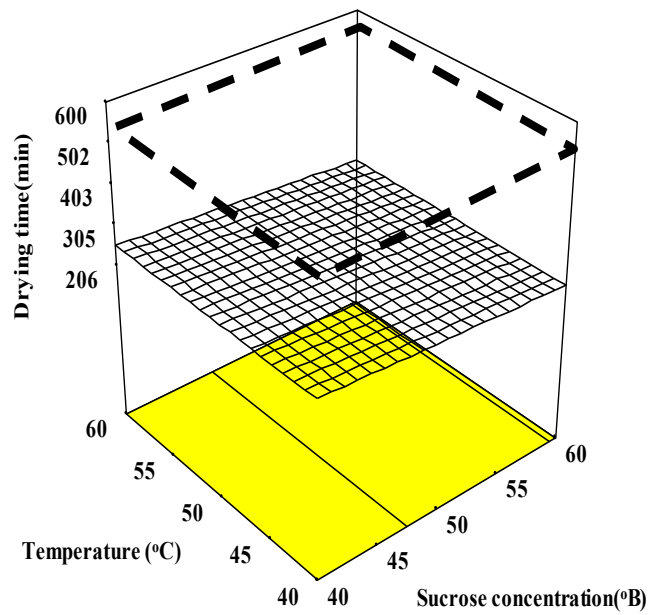
Table 7.2 ANOVA and regression coefficients of the second-order polynomial model for the response variables (actual values).

Source	Drying time min			Coefficient of Moisture Diffusion(10^{-10}) m^2/s			Coefficient of Moisture inffusion (10^{-9}) m^2/s		
	Coefficient	Sum of squares	P- Value	Coefficient	Sum of squares	P- Value	Coefficient	Sum of squares	P- Value
Model	233	72651	< 0.0001***	3.447	74.186	< 0.0001***	-258	696.83	< 0.0001***
Linear									
C	-22.1	6680	0.0196*				5.019	2.59	0.2019
T			NS		1.423	0.3215	5.192	0.68	0.5021
t	-59.8	48880	< 0.0001***	0.454	38.187	< 0.0001***	1.375	1.38	0.3437
Quadratic									
C.C			NS				-0.045	297.71	< 0.0001***
T.T			NS		6.688	0.0421*	-0.044	285.11	< 0.0001***
t.t	34.1	17091	0.0008**	-0.006	30.171	0.0003**	-0.018	225.58	< 0.0001***
Interaction									
C.T			NS			NS	-0.010	8.57	0.0312
C.t			NS			NS			NS
T.t			NS			NS	-0.007	8.36	0.0329
Statistic analysis for the model									
Lack of fit		13700	0.130 NS		13.1	0.585NS		11.775	0.152NS
R-squared		0.820			0.785			0.978	
Adj R-squared		0.787			0.728			0.962	
CV		12.3			10.430			14.718	

C, T and t are sucrose concentration ($^{\circ}B$), process temperature ($^{\circ}C$) and Contact time (min)

*Significant at 0.05 level. **Significant at 0.01 ***Significant at 0.001 level; NS: Non significant

(a)



(b)

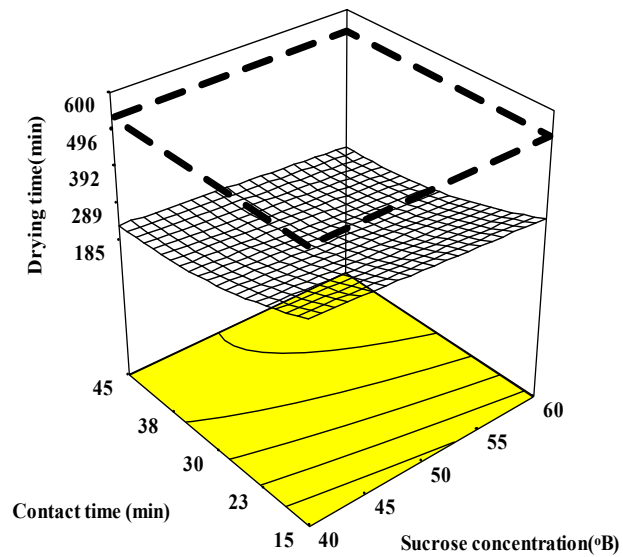


Figure 7.2 Three-dimensional (3D) response surface plots showing the effect of the osmotic pre-treatment on the air drying time: (a) the effect of sucrose concentration and temperature at medium contact time of 30 min; (b) the effect of sucrose concentration and contact time on drying time with sucrose concentration at 50°C. The surface with the dash line is the untreated control

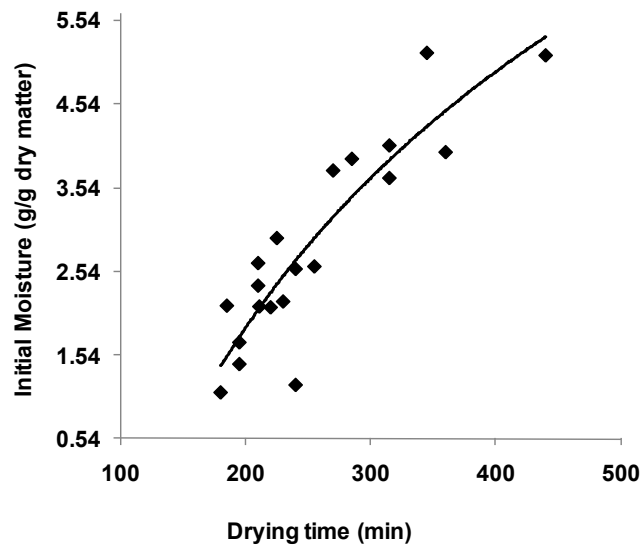


Figure 7.3 The correlation between initial moisture and drying time after MWODS pre-treatment

Figure 7.4 shows the percentage reduction in air drying time in MWODS pre-treated samples as influenced by sucrose concentration and osmotic medium temperature of the MDODS pre-treatment. The pre-treatment of apples shortened air drying time by 30-65% as compared to untreated control. This contributes to a two-fold advantage of MWODS pre-treatment in drying of apples. First, since MWODS treatment reduces the initial moisture content by 25-84%, and hence this amount of moisture does not need to be removed during the air drying (reduced load for the air drier). Second, in the air drying process, the moisture is removed in the form of vapor and hence the latent heat of vaporization needs to be supplied the hot air. Each kilogram of water requires 2250 kJ of heat for vaporization. Unlike in air drying, the osmotic dehydration process does not require the supply of latent heat of vaporization since the moisture is removed in the liquid form. Hence for the amount of moisture removed during the MWODS treatment, no latent heat was required. This represents a significant energy saving. For example, if 50% of the original moisture is removed by the osmotic pre-treatment, on a crude estimate, one could save over 1000 MJ of heat from a ton of fruit. The degree of energy saving depends on the extent of utilization of the MWODS pre-treatment. Further, it is well recognized that the air drying process is not very energy efficient; hence, the longer

the product stays in the drier, the more energy inefficient the system becomes. Since the MWODS pre-treatment results in 30-65% reduction in air drying times, it allows for better energy efficiency of the air dryer due to the lower operational times.

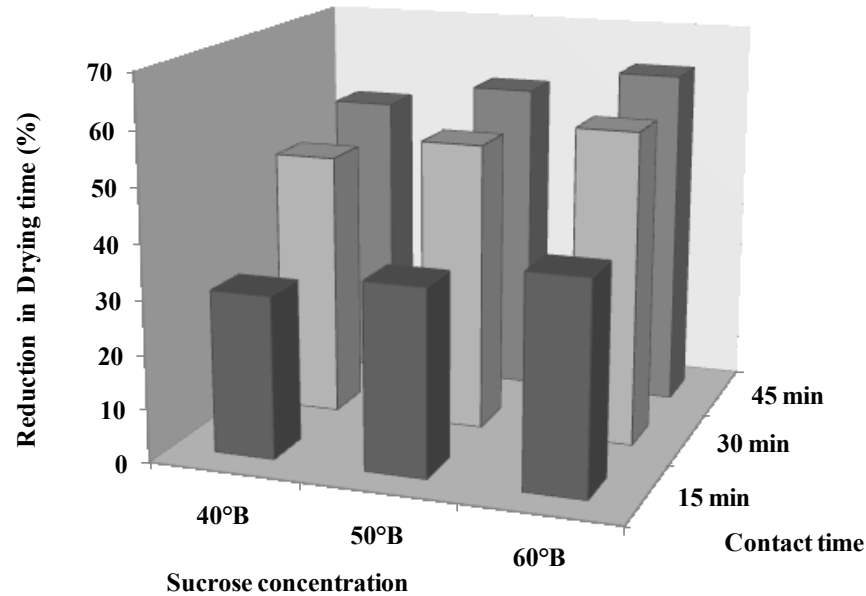


Figure 7.4 The effect of sucrose concentration and contact time at a temperature of 50°C on % reduction in drying time

7.3.3 Mathematical modeling of dehydration kinetics

In order to model drying kinetics, the coefficients of moisture diffusion of apples during air-drying were estimated with the experimental data with and without the MWODS pre-treatment. The coefficient of moisture diffusion was determined from the slopes of the residual moisture ratio vs time curves (Eq. 7.3). The semi-logarithmic plots demonstrated a fairly good fit to experimental data with R^2 higher than 0.97 (data not shown). The computed D_m values of MWODS pre-treated apples are shown in Table 7.1. The coefficient of moisture diffusion from the present study is difficult to compare with other reported literature references due to differences in method, variety, composition and structure (Simal et al., 1997). Karathanos et al. (1995) found the effective diffusivity in the range of 4 to $21 \times 10^{-10} \text{ m}^2/\text{s}$ for apples. Nsonzi and Ramaswamy (1998b) also

observed the same level for blueberries (1 to 2×10^{-10} m²/s). Pavón-Melendez et al. (2002) reported different results for D_m from 2.2×10^{-10} to 9.4×10^{-10} m²/s at 60°C for different fruits and vegetables. The values reported for effective diffusivity in this study were within the general range of 10^{-9} to 10^{-10} m²/s for biological materials; however, D_m values found in this work were higher than those reported in the literature for apples (El-Aouar et al., 2003). Based on the solution of Fick's second law equation for a finite cylinder (Eq. 7.2), moisture loss during convective drying was predicted assuming a uniform initial moisture distribution, constant diffusion coefficient, negligible external resistance shrinkage and negligible temperature gradients during drying. Figure 7.1 also shows (d,e,f) the experimental data (points) on the moisture ratio versus drying time and the model prediction (lines) for the untreated control as well as selected MWODS treated samples at different sucrose concentrations, temperatures and contact times. A good agreement between experimental and predicted values of moisture ratio ($R^2 > 0.98$) was observed.

7.3.4 Polynomial models for moisture diffusivity

The second-order polynomial response surface model (Eq. 7.2) was fitted to the coefficient of moisture diffusion and the sum of squares of the sequential model was analyzed (Table 7.2) The results showed that quadratic term significantly improved the model and it was chosen as an acceptable model of the response variables. Table 7.2 presents the estimated regression coefficients of the quadratic polynomial models for the response variable, together with the corresponding coefficients of determination (R^2). Moreover, the model adequacy was checked by adj- R^2 and coefficient of variation (CV). The lack of fit was not significant, meaning that the models were accurate for predicting the responses (Myers and Montgomery, 2002). Model fitting and ANOVA were validated by analyzing residuals, including the examination of diagnostic plots and calculation of case statistics. In order to check the adequacy of the model, the backward stepwise solution was used to step down the effects that are not significant ($P > 0.05$). The model adequately predicted the response values as R^2 was comparable to Adjusted - R^2 and CV

values were less than 10%. The response surface was generated by keeping one variable at its zero level (center point) and varying the others in their experimental range.

7.3.5 Effect of MWODS treatment on the coefficient of moisture diffusion (D_m)

From the ANOVA (Table 7.2), it can be observed that the regression model for the moisture diffusivity was significant at $P \leq 0.0001$. The P -values indicated that the linear and effect of contact time ($P \leq 0.0001$), and quadratic effect of temperature $P \leq 0.001$ and were significant at $P < 0.05$, while the interactions effects of independent had a non-significant ($P > 0.05$) effect on D_m . The three-dimensional response surface plots illustrate the relationship between independent and dependent variables (Figure 7.5). D_m values increased with an increase in, osmotic temperature and contact time. Rahman and Lamb, (1991); Karathanos et al. (1995) and Simal et al. (1997) reported that osmotic dehydration usually reduces the coefficient of moisture diffusion; however, the D_m reported by Park et al. (2003) was higher in osmotic dehydrated pears than untreated samples when applying a higher air velocity due to the reduction in the effect of shrinkage and surface hardening. As can be seen from the response surface plots (Figure 7.5) and the dashed area, the D_m values of MWODS treated samples were higher than those of untreated sample.

7.3.6 Mathematical modeling of rehydration kinetics

Moisture gain vs time was plotted for MWODS pre-treated, untreated air-dried (AD), and freeze-dried (FD) apples during rehydration. Figure 7.6 (a,b,c) shows a typical rehydration curve. The results show that increasing sucrose concentration, temperature and contact time result in increasing the rehydration ability. It can be seen that the moisture content of dehydrated apples as a function of rehydration time increased exponentially. This exponential increase in moisture content is in agreement with previous research (Jambrak et al., 2007).

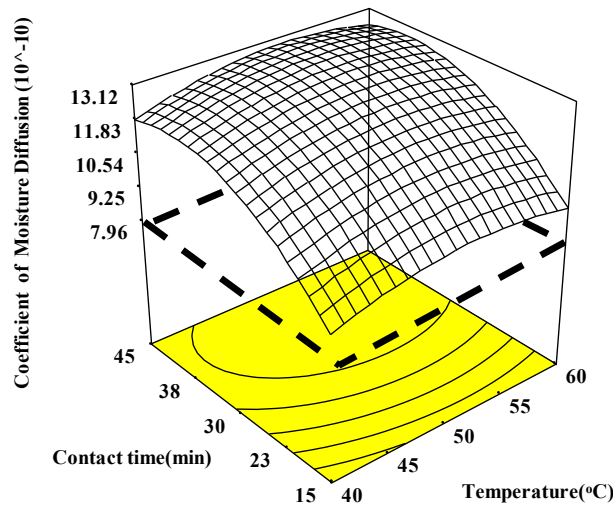


Figure 7.5 Three-dimensional (3D) response surface plots showing the effect of the effect of temperature and contact time on moisture diffusivity (sucrose concentration= 50°C). The surface with the dash line is the untreated control

The results show that rehydration ability was the highest in the FD samples followed by MWODS air dried samples, and air-dried. The FD samples are more porous and the cell walls are more permeable to adsorption of water; therefore, rehydration ability was high. Similar results were obtained by Prothon et al. (2001); Venkatachalapathy and Raghavan (1999); and Nsonzi and Ramaswamy (1998b). The combined process of osmotic dehydration followed by convective air drying has been reported to strongly decrease the values of the mass transfer coefficients during rehydration (Nsonzi and Ramaswamy, 1998a; van Nieuwenhuijzen et al., 2001; Torreggiani, 1995). However, pre-treating with MWODS before the osmo-convection processed had a reversed effect. It is known that application of microwave causes increased permeabilization of the cell membranes which facilitate faster water loss during air drying. In addition, osmotic dehydration as a pre-drying treatment is improved texture of the final product due to prevention of shrinkage. The results of Neumann (1972) and Jayaraman et al., (1990) showed higher RC when food materials were soaked in sucrose solution prior to drying which agrees with the results of this study. Rehydration ability of

air-dried samples was less than the two other methods. This might be due to excessive heat during drying which destroys the osmotic properties of the cell and the cell turgor, thereby affecting the ability of the tissue to absorb and to retain water. In addition case hardening during air drying results in decreasing rehydration capacity.

In order to model the moisture pick-up during rehydration, the two parameter Azuara model was used. The Azuara parameters are summarized in Table 7.3 indicating the demonstrating the associated R^2 to be higher than 0.95. The experimental and Azuara model predicted moisture gain during rehydration are shown in Figure 7.6 which demonstrated a good fit of data with the Azuara model. Further, the curves show the upper and lower limits to be associated with the freeze dried (FD) and air dried (AD) samples with MWODS treated samples falling between these two limits. These results indicate the modification of the structure of the product which has resulted from the ML and SG patterns associated with the MWODS treatment. In order to compare the effect of different process variables, a new parameter, the coefficient of moisture infusion (I_m), was calculated. This is analogous to the D_m values during dehydration. In order to compute these values, it is necessary to have the maximum moisture gain during the rehydration. Since the objective of rehydration is to reconstitute the product to the original moisture content, this was used as the limiting value for the purpose of calculating I_m values. The Values of I_m were obtained from the slopes of moisture gain ratio vs time using the Fickian model Eq. 7.9. These are also summarized in Table 7.3.

7.3.7 Effect of osmotic treatment variables on the coefficient of moisture infusion (I_m)

From the ANOVA (Table 7.2), it can be observed that the regression model for the coefficient of moisture infusion (I_m) was significant at $P \leq 0.001$. The P -values indicated that the quadratic effects of sucrose concentration and contact time were significant ($P < 0.0001$), whereas the interactions of (sucrose concentration and temperature) ($P < 0.05$), and interaction effect of (temperature and contact time) had a significant ($P < 0.05$) effect on I_m . Figure 7.6 shows the combined effects of

concentration, temperature and contact time (taken two at a time with the third being maintained at the central level). All two plots showed somewhat similar results as described earlier with respect to the main variables with only marginal interaction effects. Between sucrose concentration and temperature (Figure 7.7a) and sucrose concentration and contact time (Figure 7.7b), the contact time effect was more prevalent. It can be seen that the values of I_m for FD apples were the highest, followed by MWODS air- dried and AD and apples; it is clear that higher I_m results in higher rehydration ability.

Table 7.3 Azuara and Infusion Parameters at different conditions

Experiment No	Sucrose concentration (°B)	Temperature (°C)	Contact time (min)	Azuara Parameters		R^2	Infusion Parameters	
				M_e (kg/kg dry matter)	S_1 min^{-1}		$I_m \cdot 10^{-9}$ m^2/s	R^2
1	40(-1)	40(-1)	15(-1)	2.99	0.013	0.99	2.83	0.98
2	60(+1)	40(-1)	15(-1)	3.57	0.0119	0.99	3.5	0.93
3	40(-1)	60(+1)	15(-1)	7.10	0.0103	0.99	8.73	0.98
4	60(+1)	60(+1)	15(-1)	4.26	0.0086	0.99	3.85	0.98
5	40(-1)	40(-1)	45(+1)	4.06	0.011	0.99	4.21	0.91
6	60(+1)	40(-1)	45(+1)	6.04	0.00085	0.99	5.37	0.95
7	40(-1)	60(+1)	45(+1)	4.35	0.0129	0.99	4.61	0.94
8	60(+1)	60(+1)	45(+1)	4.49	0.0057	0.96	3.04	0.9
9	33(-1.68)	50(0)	30(0)	4.05	0.008	0.99	3.64	0.97
10	67(+1.68)	50(0)	30(0)	3.12	0.0093	0.99	2.85	0.97
11	50(0)	33(-1.68)	30(0)	3.85	0.011	0.99	3.9	0.99
12	50(0)	67(+1.68)	30(0)	3.19	0.016	0.99	3.14	0.97
13	50(0)	50(0)	5(-1.68)	4.75	0.0104	0.98	5.7	0.91
14	50(0)	50(0)	55(+1.68)	4.46	0.008	0.97	4.12	0.93
15	50(0)	50(0)	30(0)	24.87	0.0074	0.98	17.42	0.95
16	50(0)	50(0)	30(0)	27.01	0.0075	0.98	17.32	0.95
17	50(0)	50(0)	30(0)	26.13	0.0088	0.99	15.33	0.98
18	50(0)	50(0)	30(0)	27.17	0.0079	0.98	16.64	0.95
19	50(0)	50(0)	30(0)	26.10	0.0085	0.96	17.64	0.99
20	50(0)	50(0)	30(0)	27.23	0.0089	0.95	17.33	0.98

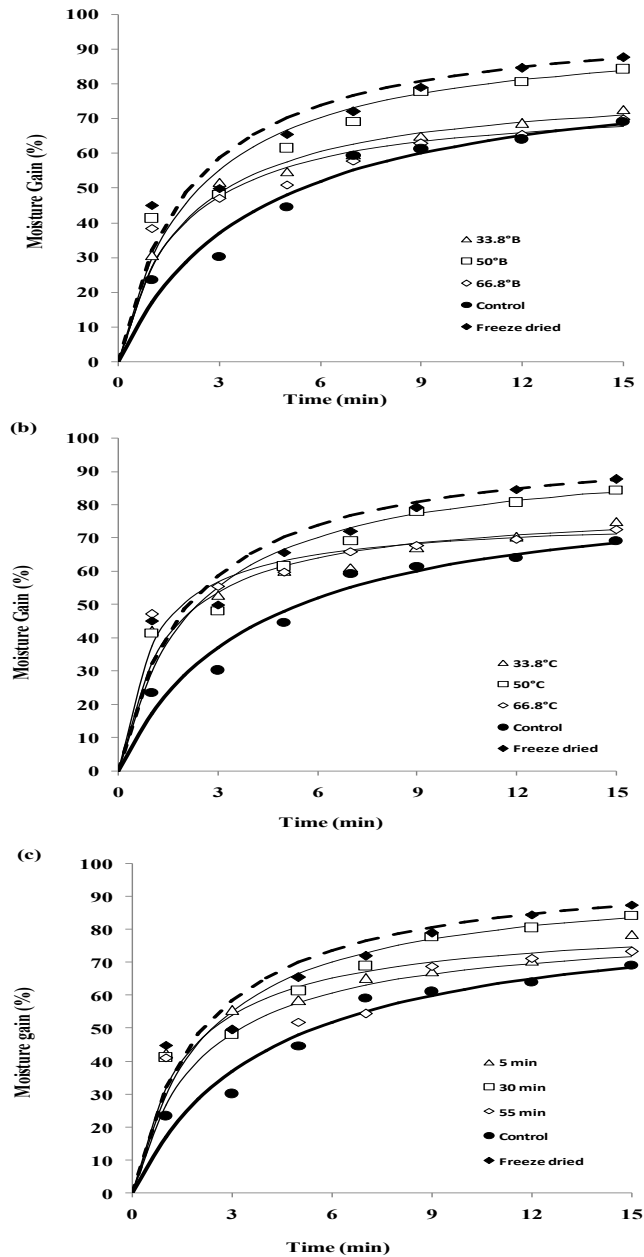
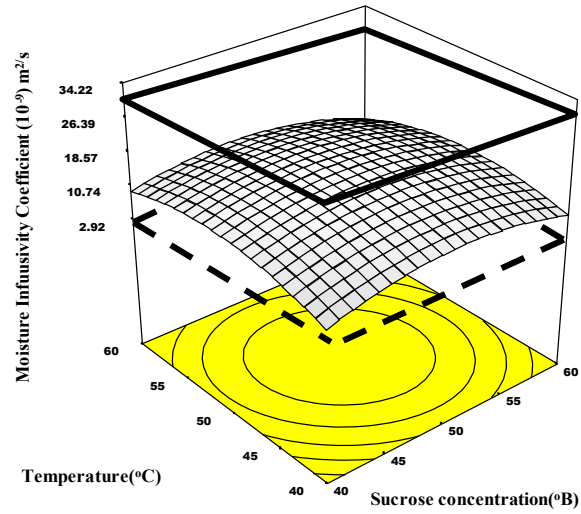


Figure 7.6 Rehydration curves of MWODS pre-treated and untreated apple followed by hot-air drying(dash line) and freeze-drying(solid line) (a) different sucrose concentration (temperature, 50°C, contact time, 30 min); (b) different temperatures (sucrose concentration, 50°B, contact time, 30 min); and (c) different contact times (sucrose concentration, 50°B, temperature, 50°C). Predicting lines are based on Azuara prediction

(a)



(b)

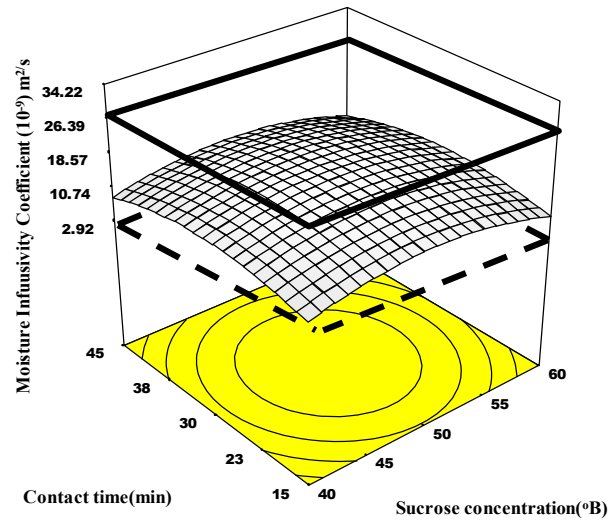


Figure 7.7 Three-dimensional (3D) response surface plots showing the effect of the variable on the response: (a) the effect of sucrose concentration and temperature on the moisture infusivity coefficient (contact time, 30min); (b) the effect of sucrose concentration and contact time on moisture infusivity coefficient (temperature, 50°C). The surface with the dash line (untreated); the surface with solid line (freeze-dried)

7.4 Conclusions

The second stage air-drying kinetics of MWODS treated samples was studied. The effects of sucrose concentration, temperature, and contact time on drying time, moisture diffusivity and moisture infusivity were investigated. The moisture diffusivity increased with increasing contact time and temperatures. The mass transfer kinetics effectively modeled using the Fick's law, and the model parameters were adequately related to the MWODS pre-treatment variables. Further, the moisture infusion processes of the dried samples were evaluated using a simulated rehydration method. The results revealed that coefficient of moisture infusion (I_m) for FD apples were the highest, followed by MWODS air- dried apples and AD; therefore, higher I_m results in higher rehydration ability than air dried samples, not to the extent of reaching that of FD samples which are very porous.

CONNECTIVE STATEMENT TO CHAPTER 8

The primary purpose of osmotic dehydration is to improve the product quality in terms of color, and texture as compared to other methods. The effect of microwave osmotic dehydration pretreatment under continuous flow medium spray (MWODS) condition and followed by the second stage air-drying on the quality parameters of dehydrated apple (*Red Gala*) cylinders was evaluated in this chapter. During air-drying, fruit generally undergo enzymatic and/or non enzymatic browning. The osmotic pretreatment generally has been shown to reduce this browning effect and also to improve the product texture. Generally air drying results in hard texture and freeze-drying results in brittle product, while OD has the potential to produce a more desirable chewy product. In this chapter, the resulting products from the MWODS – air drying combination process were compared to two products from two other methods without the use of MWODS pre-treatment: air-drying on one side and freeze drying on the other which are generally expected to give the least and most desirable results.

Part of the results of this study has been presented at the following conference:

Azarpazhooh, E and Ramaswamy, HS. 2010. Color parameter changes in apple cylinder during microwave osmotic dehydration under continuous flow medium-spray conditions. Annual meeting of Institute of Food Technologists. July 17-20, Chicago, USA. (Poster).

Based on results from Chapter 8, a manuscript has been submitted for publication:

Azarpazhooh, E and Ramaswamy, HS. 2010. Quality evaluation and optimization of microwave osmotic pre-treated apples after the second stage air drying. International Journal of Microwave Science and Technology (Submitted).

All experiment work and data analysis were carried out by the candidate under the overall supervision of Dr. HS. Ramaswamy.

CHAPTER 8. QUALITY EVALUATION AND OPTIMIZATION OF MICROWAVE OSMOTIC PRE-TREATED APPLES FOLLOWING THE SECOND STAGE AIR DRYING

Abstract

Prepared apple (*Red Gala*) slices were subjected to microwave osmotic dehydration treatment under continuous flow medium spray (MWODS) conditions and then finish dried in an air-drier to a final moisture content of 25% (dry basis). The dried samples were evaluated for color parameters (L^* , a^* , b^* values, color intensity (ΔE), chroma and hue angle), textural properties (maximum force (hardness), the slope of the final section of the force-distance curve (rigidity), and the area under the force-distance curve (energy)), and rehydration capacity (RC). The MWODS pre-treatments were based on a central composite rotatable design (CCRD) and a response surface methodology (RSM) using five levels of osmotic variables: sucrose concentration (33.3-66.8°B), temperature (33.3-66.8°C), and contact time (5-55 min) at a constant flow rate of 2800 ml/min. The air drying was carried out at 60°C, 15±1% relative humidity and 0.64±0.02 m/s air velocity. The results were compared to untreated air-dried (AD) (worst case scenario) and freeze -dried (FD) (best case scenario) treated apples without the MWODS treatment.

The results revealed that color parameters were affected regardless of the type of MWODS treatment. Increasing sucrose concentration and temperature caused an increase in color parameters of MWODS air-dried apples and enhanced their quality. Comparison of MWODS air-dried apples with AD show that the (AD) apples were darker in color, whereas those air dried after the MWODS pre-treatment were lighter with higher L^* and b^* values, higher hue and chroma values but lower a^* value and ΔE . Further the color parameters of treated samples were closer or equal to the freeze-dried (FD) apples. The hardness was decreased by increasing the osmotic sucrose concentration of MWODS pre-treatment producing softer dried apples, whereas AD samples were hard and FD apples were brittle. Finally, FD samples yielded a product with higher rehydration capacity followed by MWODS air-dried, and the least for the AD.

Applying the desirability function method, optimum operating conditions were found to be sucrose concentration of 49.6 °B, temperature of 51.9 °C, and contact time of 33.3 min. At this optimum point, L^* value, ΔE , hardness and rehydration capacity (RC) were found to be 82.3, 6.2, 7.1 and 88.5, respectively, with a 0.90 desirability.

8.1 Introduction

Over the last few decades, a heightened interest in improving the marketability of high quality dried food has been evident in all segments of the food process industry. The process of osmotic dehydration appears to be the ideal result of this interest with its promise of producing new minimally processed fruits. Osmotic dehydration is a pre-treatment technique; further processing, such as air, freeze and vacuum drying is necessary to complete the process. It is recommended that the quality (color, texture and rehydration capacity) of air, freeze or vacuum dried fruits and vegetables could be improved by a prior osmotic step (Nsonzi and Ramaswamy, 1998b). Although freeze-drying is considered to be one of the best methods to keep the quality attributes of the materials, it is costly; for that reason, it is sometimes used as a reference method used to compare drying experiments (Nsonzi and Ramaswamy, 1998b). Microwave osmotic dehydration (MWODS) could be used, before air-drying, to accelerate mass transfer and to create more homogeneous concentration profiles in the fruit. In this process, within a 30 min treatment period, the moisture in fruit was reduced by about 50% of its original moisture (Azarpazhooh and Ramaswamy, 2010a). In chapter 5 and 6, MWODS was evaluated and optimized under different osmotic conditions, and the drying rate of apple was predicted; however, in order to design an osmotic pre-treatment process and to determine the stability and acceptability of the final product, knowledge of quality assessment such as color, texture and rehydration capacity is necessary. There have been numerous studies on the evaluation of color change during osmotic dehydration. The color of the products is measured by lightness (L^* value), redness or greenness (a^* value) and yellowness or blueness (b^* value), during or after drying. Falade et al. (2007) has reported that the transparency and color of the fruit may alter favorably due to physical and chemical changes during osmotic dehydration. They evaluated L^* , a^* , b^* values,

color intensity and chroma values of osmo-oven dried watermelon, and reported that color parameters increase with an increase in osmotic solution concentration. Osmotic dehydration improves fruit quality by stabilizing color parameters and inhibiting decolourisation of fruit by enzymatic oxidative browning due to infusion of extensive sugars. In addition, as the water activity of samples is reduced , the non-enzymatic browning reaction is also decreased (Krokida et al., 2000c).

Another important quality characteristic of dried products is texture, which is usually measured by mechanical tests. Puncture force is usually used to measure the textural property of dehydrated products which is the measure of the hardness of the product surface, and presents the extent of case hardening during drying (Lin et al., 1998). Cell turgor is the main factor that contributes to mechanical properties of plant tissue. During osmotic treatment the main changes that affect mechanical behavior of plant tissues are loss of cell turgor, alteration of middle lamella (Alzamora et al.,1996). Differences in mechanical behavior of the dried samples must be related to the differences induced in the composition of the soluble water phase and in the solid matrix during treatments. Contreras et al. (2007) reported that soluble pectin is increased during drying which alters the cell bonding zone resulting in changing the solid matrix consistency. Osmotic dehydrated product has a softer texture due to leaching of calcium into the osmotic solution which in turn results in lowering the concentration of calcium content ions inside the tissue (Prothon et al., 2001). In addition, rehydration capacity is used as a quality index to present the physical and chemical alternations during drying (Moreira et al., 2008). Most dehydrated products are frequently rehydrated prior to consumption like in a yogurt or used as an ingredient in cooking preparations. Rehydration compiles two simultaneous processes: the absorption of water by dried tissue and the leaching of a soluble. Compositional changes during osmotic dehydration may have a negative impact on rehydration capacity where rehydration of osmotically dried fruit is lower than the untreated one (Lewicki, 1998).

The objectives of this work are to study the effect of osmotic drying variables like sucrose concentration, temperature, and contact time on quality parameters (color, texture

and rehydration capacity) during the microwave-osmotic dehydration of apples under continuous flow medium spray conditions (MWODS) followed by air-drying and then comparing the results with air-dried and freeze-dried apples without MWODS treatment; and to find out the optimum conditions for producing MWODS air-dried apples using a central composite rotatable design (CCRD) of experiments and a response surface methodology (RSM) for data analysis.

8.2 Materials and Methods

8.2.1 Materials

Details are presented in chapter 7.

8.2.2 Osmotic dehydration and drying procedure

Details are presented in chapter 7.

8.2.3 Air- drying method

Details are presented in chapter 7.

8.2.4 Freeze- drying

Details are presented in chapter 7.

8.2.5 Color measurement

The color values of air-dried (MWODS treated or untreated) and freeze- dried apples were measured, in L*, a*, b* system, using a tristimulus Minolta Chroma Meter (Minolta Corp., Ramsey, NJ). The instrument was warmed up 20 minutes before experiments, and calibrated with white standard. At least six measurements were individually made on each sample at different locations and the average value was reported. The color value was determined in a three-dimensional color space, with L* (luminosity), a* (green - to red +), and b* (blue - to yellow +) values of the apple samples.

In addition, the total color intensity Eq. (8.1), chroma Eq. (8.2), Hue angle Eq. (8.3) were calculated. Fresh apples were used as the reference for measuring ΔE where subscript “o” refers to the color reading of fresh apples (Maskan, 2001b; Maftoonazad and Ramaswamy, 2008).

$$\Delta E = \sqrt{(L_o^* - L^*)^2 + (a_o^* - a^*)^2 + (b_o^* - b^*)^2} \quad (8.1)$$

$$\text{Chroma} = \sqrt{(a^*)^2 + (b^*)^2} \quad (8.2)$$

$$\text{Hue angle} = \tan^{-1}\left(\frac{b^*}{a^*}\right) \quad (8.3)$$

8.2.6 Mechanical properties measurement

Mechanical properties of MWODS pre-treated and untreated apples followed by air-drying were compared with freeze-dried samples. The texture of dried apple was analyzed using a texture analyzer (TA/XT/PLUS Stable Micro. Systems Ltd., Godalming, UK) by means of a puncture test (2.5 diameter punch), considering a relative deformation of 85 % and a deformation rate of 2 mm/s. Eight replicates were performed for each treatment, and the average was reported. A force–distance curve was recorded by the instrument and three textural attributes including hardness (N), the slope of the final section of the force-distance curve (rigidity) (N/mm), and the absorbed energy as area under the force–distance curve (J) were collected.

8.2.7 Rehydration capacity

Apple cylinders (8 samples) after air- drying and freeze- drying were rehydrated by immersion in excess distilled water at room temperature (20°C for 14 h). After rehydration, samples were placed in filter paper with a slight vacuum for 1 min and then their weights were measured. The rehydration capacity was determined as the weight ratio between the rehydrated sample and the sample before rehydration (g) (Levi et al., 2006).

$$\text{Rehydration Capacity} = \frac{W_r - W_d}{W_d} \quad (8.4)$$

W_r is the weight after rehydration (kg) and W_d is the weight of dried material (kg). Determinations were made in triplicate, and the products were compared with AD and FD apples.

8.2.8 Experimental design for optimization of parameters

Aiming to evaluate the influence of sucrose concentration ($^{\circ}\text{C}$) and solution temperature (T) and contact time (t) on color parameters, texture and rehydration capacity of dried apples, statistical optimization experiments were initially carried out according to a Central Composite Rotatable Design (CCRD) including 20 experiments formed by 6 central points and 6 ($\lambda = 1.68$) axial points (Table 8.1 and 8.2). The independent process variables were sucrose concentration (33.3–66.8 $^{\circ}\text{B}$), temperature (33.3–66.8 $^{\circ}\text{C}$) and contact time (5–55 min) with a constant flow rate of 2800 ml/min. Design Expert Version 6.01 (Statease Inc., Minneapolis, USA) was used for regression and graphical analysis of the data obtained. The following polynomial model was fitted to the data:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{11} X_1^2 + b_{22} X_2^2 + b_{33} X_3^2 + b_{12} X_1X_2 + b_{13} X_1X_3 + b_{23} X_2X_3 \quad (8.5)$$

Here b_0 is the constant regression coefficients of the model; Y represents the experimental response color parameter, texture and rehydration capacity; X_1 , X_2 and X_3 in Eq. (8.5) are sucrose concentration ($^{\circ}\text{B}$), process temperature ($^{\circ}\text{C}$) and contact time respectively. Analysis of variance (ANOVA) for each response was found the significant terms in the model, and for checking the adequacy of the model, the effects that are not significant ($P > 0.05$) may carried out stepping down by using “backward” reduction algorithm and then coefficient of determination (R^2), adjusted- R^2 , and Coefficient variance (CV) were considered (Myers Myers and Montgomery, 2002). Response surface plots were generated. The relationship between independent and dependent variables is illustrated in three-dimensional representations of the response. The response surfaces

were based on the coefficients presented in Tables 8.3 and 8.4. The multiple responses were simultaneously optimized by the desirability function method of the Design Expert software 6.01 (Statease Inc., Minneapolis, USA). For each variable and response, the desired goal was considered (maximized, minimized or within ranges) and the independent variables were kept within range.

8.3 Results and Discussion

8.3.1 Model fitting

The relevant experiment results of different runs evaluated under the different CCRD experimental conditions are tabulated in Table 8.1 and Table 8.2. The second-order polynomial response surface model (Eq. 8.5) was fitted to each of the response variables (Y). The sum of squares of the sequential model was analyzed to find the variation of color and texture, and rehydration capacity. These analyses indicated that adding terms up to quadratic significantly improved the model (data not given). In order to determine the significant effects of process variables on each response, an analysis of variance and the regression equation coefficients of the proposed models for each response were conducted which are shown in Tables 8.3 and 8.4. The ANOVA showed that lack of fit was not significant ($P > 0.05$) for all responses which mean that all models represented the data sufficiently accurately for predicting the relevant responses. The coefficient of determination, R^2 , representing the suitability of fitting the experimental model to the actual data, was found to be higher than 0.85 for all the responses and suitably in agreement with Adj- R^2 . Moreover, the fact that the coefficient of variation (CV) was less than 10 indicated that the variation in the mean value was less; therefore, the response model was satisfactorily developed. Figures 8.1 and 8.2 show the comparison between the experimental values and the model predicted values. The results demonstrate that the polynomial regression models were in good agreement with the experimental results, and the models were able to identify operating conditions.

Table 8.1 Experimental design of process in coded and actual variables and values of experimental data (Color parameters)

Process Conditions	Sucrose concentration (°B)	Temperature (°C)	Contact time (min)	L* value		a* value		b* value		ΔE		Hue angle		Chroma	
1	40(-1)	40(-1)	15(-1)	75.9	± 2.36	9.32	± 1.09	35	± 0.46	13.3	± 0.8	75.1	± 1.85	36.2	± 0.2
2	60(+1)	40(-1)	15(-1)	74.6	± 1.95	9.19	± 0.06	31	± 0.56	12.9	± 1.2	73.3	± 0.39	32	± 0.5
3	40(-1)	60(+1)	15(-1)	76.7	± 1.95	7.82	± 1.59	32	± 1.68	10.7	± 0.3	76.2	± 2	32.8	± 2
4	60(+1)	60(+1)	15(-1)	77.3	± 0.21	7.88	± 0	31	± 1.36	10	± 0.4	75.7	± 0.6	32	± 1.3
5	40(-1)	40(-1)	45(+1)	79.7	± 0.07	7.58	± 0.6	33	± 0.89	8.8	± 0.1	77	± 1.33	33.8	± 0.7
6	60(+1)	40(-1)	45(+1)	78	± 0.33	7.13	± 0.68	31	± 0.77	9.1	± 0.7	77	± 0.88	31.8	± 0.9
7	40(-1)	60(+1)	45(+1)	79.4	± 1.02	6.56	± 0.34	30	± 1.44	7.4	± 1	77.6	± 0.04	30.6	± 1.5
8	60(+1)	60(+1)	45(+1)	79.6	± 1.47	6.3	± 0.8	31	± 11.17	7.4	± 4.1	78.6	± 6.14	32	± 11
9	33(-1.68)	50(0)	30(0)	79.2	± 0.85	7.1	± 0.79	32	± 1.38	8.3	± 0.5	77.3	± 0.83	32.4	± 1.5
10	67(+1.68)	50(0)	30(0)	78.3	± 1.05	6.78	± 1.22	29	± 1.15	8.3	± 0.1	77	± 2.76	30	± 0.8
11	50(0)	33(-1.68)	30(0)	79.3	± 1.74	8.93	± 1.62	33	± 1.02	10	± 0.3	74.9	± 3.06	34.3	± 0.6
12	50(0)	67(+1.68)	30(0)	81.3	± 1.74	6.96	± 0.74	31	± 0.75	6.5	± 0.1	77.3	± 1.61	31.6	± 0.6
13	50(0)	50(0)	5(-1.68)	72	± 0.62	9.1	± 0.75	34	± 0.14	15.7	± 0.7	74.9	± 1.25	34.9	± 0.1
14	50(0)	50(0)	55(+1.68)	77.1	± 0.23	6.31	± 0.16	32	± 1.15	9.8	± 0.5	78.9	± 0.66	32.9	± 1.1
15	50(0)	50(0)	30(0)	81.4	± 0.10	5.46	± 0.23	33	± 0.79	6.7	± 0.6	80.6	± 0.17	33.5	± 0.8
16	50(0)	50(0)	30(0)	82.6	± 0.06	4.35	± 0.99	35	± 1.04	6.8	± 0.6	82.8	± 1.83	34.8	± 0.9
17	50(0)	50(0)	30(0)	81.3	± 0.10	4.35	± 0.53	33	± 0.76	6	± 0.7	82.4	± 0.74	33	± 0.8
18	50(0)	50(0)	30(0)	81	± 1.09	6.49	± 0.4	33	± 0.99	7.3	± 1.2	78.8	± 0.34	33.5	± 1
19	50(0)	50(0)	30(0)	83.6	± 0.52	6.47	± 0.15	33	± 0.96	5.9	± 0.4	78.7	± 0.58	33.1	± 0.9
20	50(0)	50(0)	30(0)	82	± 0.76	5.98	± 0.75	34	± 1.44	7.4	± 0.9	80.1	± 1.63	34.7	± 1.3

Mean ± standard deviation

Table 8.2 Experimental design of process in coded and actual variables and values of experimental data (texture parameters, rehydration capacity)

Process	Sucrose concentration	Temperature	Contact time	Hardness		Rigidity		Energy		Rehydration Capacity	
Conditions	(°B)	(°C)	(min)	(N)		(N/mm)		(N)		(%)	
1	40(-1)	40(-1)	15(-1)	25.3	± 1.15	25.7	± 1.09	30.10	± 1.13	98.3	± 1.07
2	60(+1)	40(-1)	15(-1)	19.5	± 0.77	21.8	± 0.85	39.50	± 1.94	94.6	± 1.39
3	40(-1)	60(+1)	15(-1)	22.5	± 1.26	49.8	± 2.21	10.80	± 1.54	97.8	± 1.4
4	60(+1)	60(+1)	15(-1)	27.3	± 3.75	43.9	± 1.29	62.60	± 3.3	94.9	± 1.34
5	40(-1)	40(-1)	45(+1)	24.5	± 4.43	29.4	± 0.78	30.70	± 0.53	67.4	± 1.27
6	60(+1)	40(-1)	45(+1)	19.5	± 3.36	48.2	± 0.01	8.70	± 1.14	64.8	± 1.34
7	40(-1)	60(+1)	45(+1)	23.4	± 4.12	14.3	± 1.36	29.70	± 1.06	77.4	± 2.17
8	60(+1)	60(+1)	45(+1)	29	± 0.34	31.1	± 0.44	50.30	± 0.9	75.6	± 1.29
9	33(-1.68)	50(0)	30(0)	30	± 0.15	22.2	± 2.11	29.90	± 1.44	70.9	± 0.67
10	67(+1.68)	50(0)	30(0)	29.8	± 1.68	33.1	± 0.42	55.00	± 0.34	66.3	± 0.84
11	50(0)	33(-1.68)	30(0)	13.8	± 1.74	27.4	± 0.88	13.60	± 0.96	78.6	± 1.37
12	50(0)	67(+1.68)	30(0)	19.4	± 1.19	33.2	± 0.59	32.30	± 1.05	87.2	± 0.8
13	50(0)	50(0)	5(-1.68)	21.3	± 1.15	40.2	± 1	32.80	± 0.92	122.3	± 10.3
14	50(0)	50(0)	55(+1.68)	22.1	± 0.41	32.6	± 0.94	22.90	± 1.67	80.1	± 0.87
15	50(0)	50(0)	30(0)	8.79	± 1.23	4.36	± 0.53	4.89	± 1.33	90.4	± 1.17
16	50(0)	50(0)	30(0)	6.89	± 1.25	4.57	± 0.75	5.45	± 1.01	91.2	± 1.07
17	50(0)	50(0)	30(0)	3.3	± 1.26	6.66	± 1.23	3.98	± 0.91	90.6	± 1.27
18	50(0)	50(0)	30(0)	6.78	± 75.56	7.51	± 1.13	4.66	± 0.98	89.9	± 1.45
19	50(0)	50(0)	30(0)	5.89	± 0.25	6.89	± 0.66	5.18	± 0.95	90.5	± 1.18
20	50(0)	50(0)	30(0)	6.56	± 0.1	5.84	± 1.28	6.89	± 0.34	91	± 0.55

Mean ± standard deviation

Table 8.3 Analysis of variance (ANOVA) for the fit of experiment data (Color parameters) to response surface model

Sources	L* value			a* value			b* value			ΔE			Chroma			Hue angle		
	Coefficient	SS	P-value	Coefficient	SS	P-value	Coefficient	SS	P-value	Coefficient	SS	P-value	Coefficient	SS	P-value	Coefficient	SS	P-value
Model	23.9	147	< 0.0001***	48.5	34.3	< 0.0001***	31.525	41.2	< 0.0001***	52.300	127	< 0.0001***	39.1	41.5	< 0.0001***	-1.694	99.5	0.0001***
Linear																		
C	1.09	1.06	0.1912	-0.527	0.13	0.5728	0.389	6.73	0.573	-0.5705	0.034	0.7118	0.304	6.72	0.0003**	1.232	0.25	0.675
T	0.651	5.04	0.01*	-0.932	4.67	0.004**	-0.031	6.60	0.004*	-0.6604	15.3	< 0.0001***	-0.206	9.1	< 0.0001***	1.692	6.83	0.042*
t	0.797	31	< 0.0001***	-0.266	9.41	0.0003**	-0.221	2.49	0.0003***	-0.6884	42.5	< 0.0001***	-0.221	5.07	0.001**	0.436	20.59	0.002**
Quadratic																		
C*C	-0.011	18.2	< 0.0001	0.005	3.86	0.0073**	-0.010	14.7	0.0073*	0.0057	4.61	0.0007**	-0.009	11.89	< 0.0001***	-0.012	22.34	0.001**
T*T	-0.006	5.02	0.0102*	0.0087	10.9	0.0001***	-0.005	3.19	0.0001***	0.0055	4.43	0.0008**	-0.003	1.29	0.0494*	-0.016	37.87	0.0001***
t*t	-0.012	98.5	< 0.0001***	0.0035	8.95	0.0003**				0.0095	66	< 0.0001***				-0.006	25.48	0.0008**
Interaction																		
C*T			NS			NS	0.0086	5.9	0.001**			NS	0.0084	5.7	0.0006**			NS
C*t			NS			NS	0.0039	2.67	0.01*			NS	0.0036	2.33	0.0125*			NS
T*t			NS			NS			NS			NS			NS			NS
Lack of fit		2.6	0.908		0.2	1.00NS		0.200	0.999NS		1.04	0.926NS		0.045	1.00NS		2.29	0.998NS
Statistic analysis for the model																		
R-squared			0.931			0.815			0.914			0.966			0.927			0.851
Adj R-squared			0.923			0.813			0.863			0.961			0.885			0.782
CV			0.94			8.84			1.766			5.45			1.58			1.49

C, T and t are sucrose concentration (°B), temperature (°C), contact time (min). *Significant at 0.05 level. **Significant at 0.01 ***Significant at 0.001 level;. NS: Non significant

Table 8.4 Analysis of variance (ANOVA) for the fit of experiment data (texture parameters, rehydration capacity) to response surface model

Sources	L* value			a* value			b* value			ΔE			Chroma			Hue angle		
	Coefficient	SS	P-value	Coefficient	SS	P-value	Coefficient	SS	P-value	Coefficient	SS	P-value	Coefficient	SS	P-value	Coefficient	SS	P-value
Model	23.9	147	<0.0001***	48.5	34.3	<0.0001***	31.525	41.2	<0.0001***	52.300	127	<0.0001***	39.1	41.5	<0.0001***	-1.694	99.5	0.0001***
Linear																		
C	1.09	1.06	0.1912	-0.527	0.13	0.5728	0.389	6.73	0.573	-0.5705	0.034	0.7118	0.304	6.72	0.0003**	1.232	0.25	0.675
T	0.651	5.04	0.01*	-0.932	4.67	0.004**	-0.031	6.60	0.004*	-0.6604	15.3	<0.0001***	-0.206	9.1	<0.0001***	1.692	6.83	0.042*
t	0.797	31	<0.0001***	-0.266	9.41	0.0003**	-0.221	2.49	0.0003***	-0.6884	42.5	<0.0001***	-0.221	5.07	0.001**	0.436	20.59	0.002**
Quadratic																		
C*C	-0.011	18.2	<0.0001	0.005	3.86	0.0073**	-0.010	14.7	0.0073*	0.0057	4.61	0.0007**	-0.009	11.89	<0.0001***	-0.012	22.34	0.001**
T*T	-0.006	5.02	0.0102*	0.0087	10.9	0.0001***	-0.005	3.19	0.0001***	0.0055	4.43	0.0008**	-0.003	1.29	0.0494*	-0.016	37.87	0.0001***
t*t	-0.012	98.5	<0.0001***	0.0035	8.95	0.0003**				0.0095	66	<0.0001***				-0.006	25.48	0.0008**
Interaction																		
C*T			NS			NS	0.0086	5.9	0.001**			NS	0.0084	5.7	0.0006**			NS
C*t			NS			NS	0.0039	2.67	0.01*			NS	0.0036	2.33	0.0125*			NS
T*t			NS			NS			NS			NS			NS			NS
Lack of fit		2.6	0.908		0.2	1.00NS		0.200	0.999NS		1.04	0.926NS		0.045	1.00NS		2.29	0.998NS
Statistic analysis for the model																		
R-squared			0.931			0.815			0.914			0.966			0.927			0.851
Adj R-squared			0.923			0.813			0.863			0.961			0.885			0.782
CV			0.94			8.84			1.766			5.45			1.58			1.49

C, T and t are sucrose concentration (°B), temperature (°C), contact time (min). *Significant at 0.05 level. **Significant at 0.01 ***Significant at 0.001 level;. NS: Non Significant

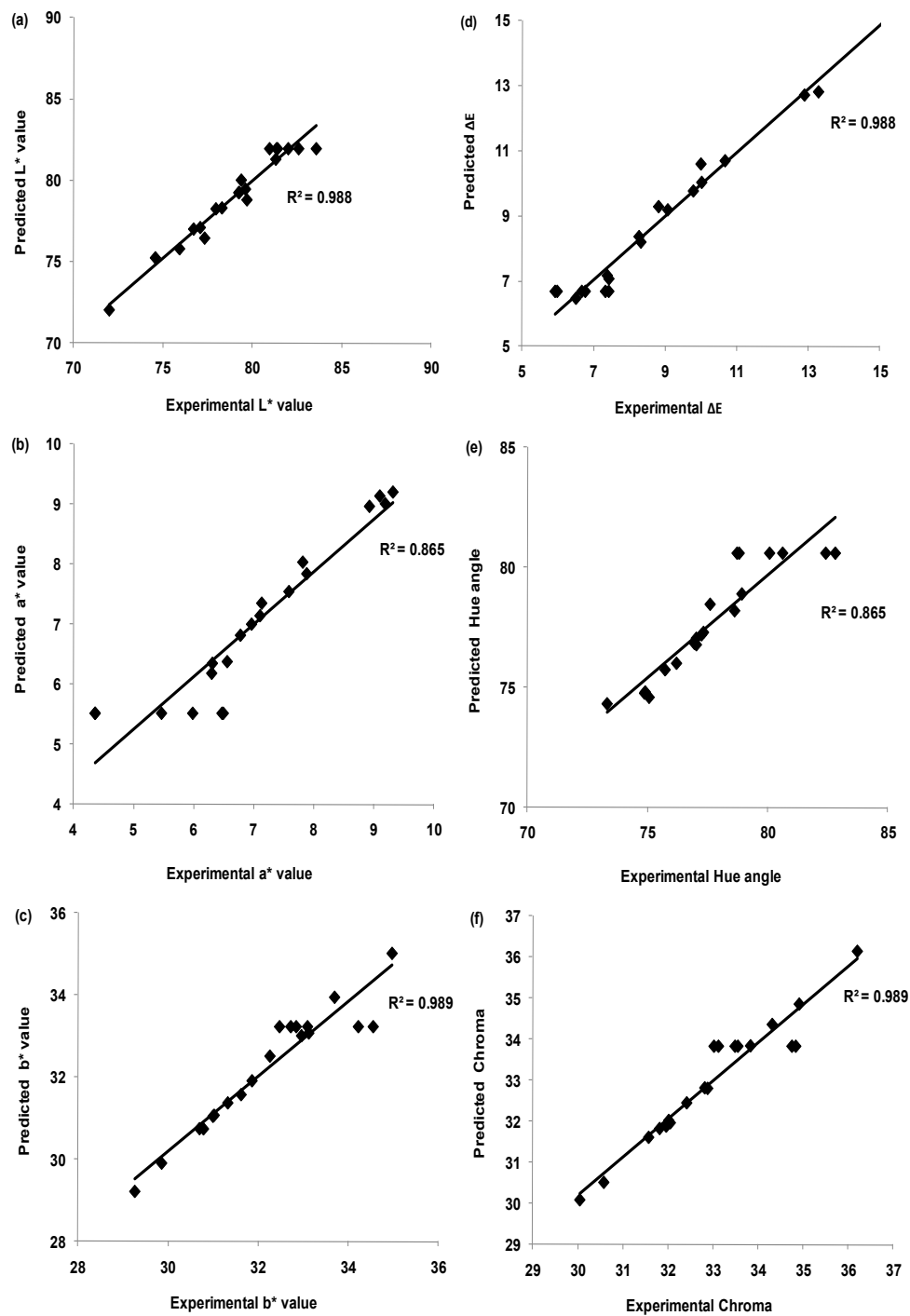


Figure 8.1 Comparison between experimental and predicted values of (a) L^* value (b) a^* value; (c) b^* value, (d) ΔE value; (e) Hue angle; (f) Chroma

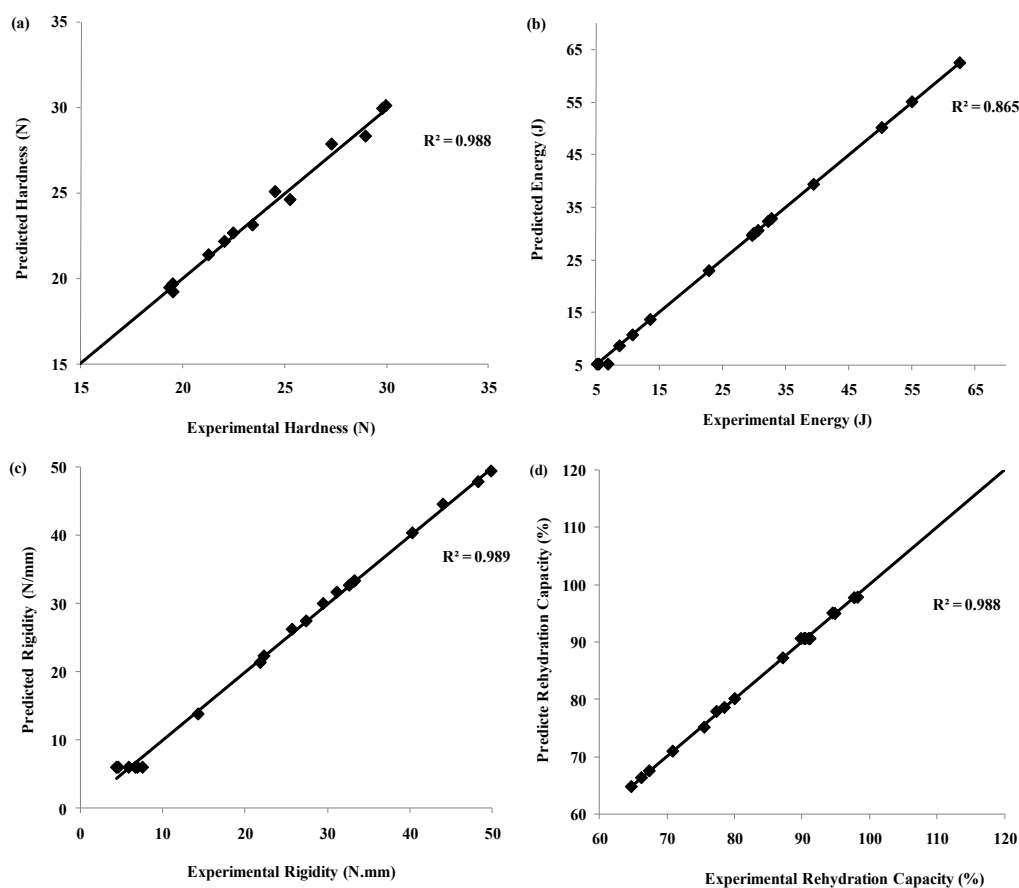


Figure 8.2 Comparison between experimental and predicted values of (a) Hardness (b) Energy; (c) Rigidity, (d) Rehydration Capacity (%)

8.3.2 Effect of process variables on color parameters

8.3.2.1 L^* , a^* , b^* values

The results given in Table 8.3 reveal that L^* , a^* values were significantly ($P < 0.05$) affected by linear effects of temperature and contact time, and all quadratic effects of independent variables, whereas the interaction effects of sucrose concentration, temperature and contact time were not significant at the 5% level. The importance of the independent variables on L^* , a^* values were the same and in the following order: contact time > temperature > sucrose concentration (based on the sum of squares). The b^* value was significantly ($P < 0.05$) affected by linear effects of temperature and contact time

parameters, and all quadratic effects of independent variables and the interaction effects of (sucrose concentration and temperature) and (sucrose concentration and contact time). The importance of the independent variables on b^* value could be ranked in the following order: sucrose concentration > temperature > contact time (based on the sum of squares).

The fitted model for the color parameters responses based on actual values is shown in Table 8.3. Tristimulus L^* values of MWODS air-dried apples is given in Figure 8.3 (a, b). The L^* value indicates the lightness of the sample and it has been used as an indicator of fruit browning. The results show that MWODS air-dried apples had a higher L^* value when compared with AD apples (Krokida et al., 2000b). The effect of contact time was more significant than sucrose concentration and temperature. The results show that increasing contact time results in increasing the lightness of samples, however at longer contact time L^* value was decreased. This might be due to higher infusion of sugar into the fruit resulting in higher L^* value (Pereira et al., 2006). It is also clear that increasing sucrose concentration resulted in increasing L^* value (Pereira et al., 2006) up to a certain extent; however, at higher sucrose concentrations, L^* value decreased due to solutes filling the pores in the fruit tissue. With respect to temperature, a higher process temperature resulted in better color characteristics maintenance of apples compared to a low temperature process which is due to lowering the viscosity of sucrose concentration at higher temperature, therefore enhancing water removal and preventing blocking the pores in the fruit tissue. Osmotically pre-treated samples did not discolor as much as the AD apples and the value for lightness (L^*) is very close to the L^* value of freeze-dried samples. The results show that around the central point, L^* value was overlapped with the value for FD. Freeze-drying seems to prevent color changes, resulting in products with improved color characteristics (Nsonzi and Ramaswamy, 1998b).

The a^* value indicates chromaticity on a green (−) to red (+) axis. Increasing a^* value has been used as an indicator of fruit browning, and higher a^* value shows that the samples are more red. Figure 8.4(a,b) shows that the a^* value decreased with increasing sucrose concentration, temperature and contact time, however, at higher independent

variables a^* value increased, which might be due to solids accumulation during osmotic pre-treatment, and possible membrane swelling/plasticizing effect, which might have increased the cell membrane permeability to sucrose molecules (Li and Ramaswamy, 2006c) consequently increasing the color intensity of the products. Comparison of MWODS air-dried apples with AD and FD show that AD apples had a higher a^* value and were browner, while FD samples had lower a^* value and were very close to the MWODS air-dried apples.

The effect of changing sucrose concentration, temperature and contact time on b^* value is given in Figure 8.5(a, b). The b^* value is an indicator of blue (-) to yellow (+) color. The synergistic effect of an increase in b^* value by the combination of sucrose concentration and temperature is clearly evidenced in this figure. Increasing sucrose concentration and temperature lead to an increase in the b^* value, however at center points b^* value decreased. Increasing contact time results in decreasing b^* value gradually which can be attributed to the increase in solute uptake at higher sucrose concentration and temperature (Heredia et al., 2009). The b^* value in MWODS dried samples did not show significant difference with Ad and FD apples was not higher than AD samples and close to FD apples. Prothon et al. (2001) reported that osmotically pre-treated samples did not brown as much as the untreated samples and the value for b^* values increased slightly.

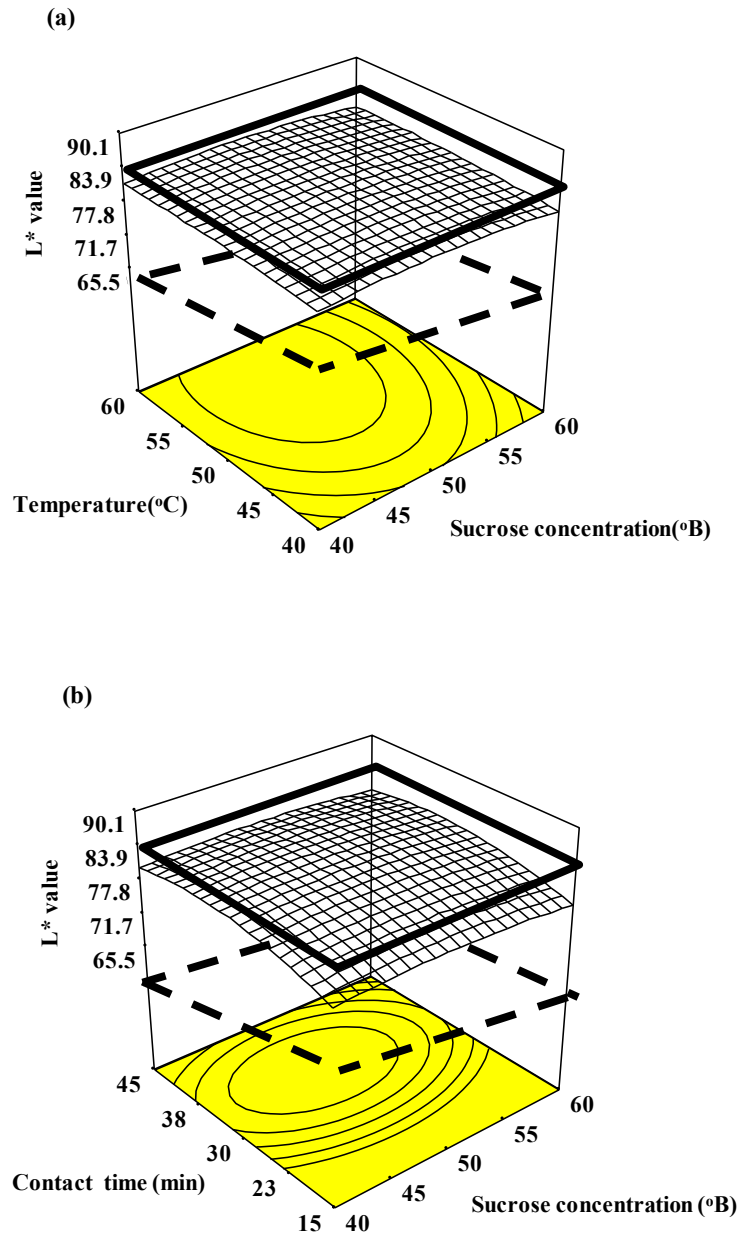


Figure 8.3 Response surface curves for L^* value (a) effect of sucrose concentration and temperature at contact time = 30 min; (b) effect of sucrose concentration and contact time at temperature = 30 °C. The perimeter with the dash line is included for air-dried sample with L^* value of 65.5 and the perimeter with solid line shows the L^* value by the freeze- dried sample (83.9)

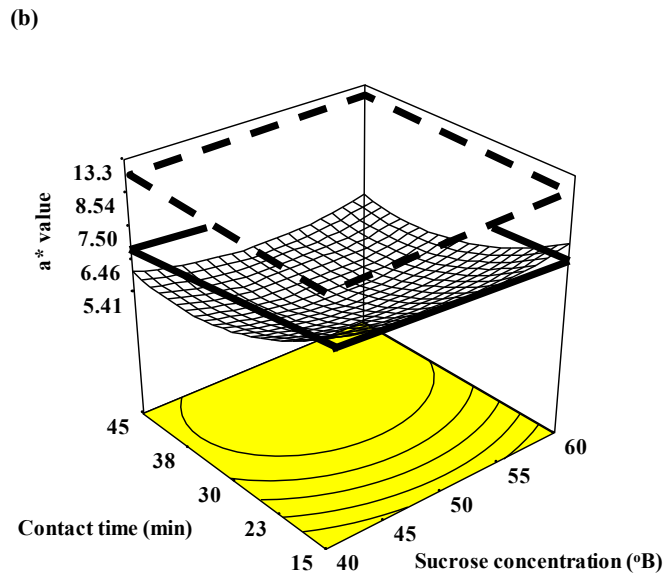
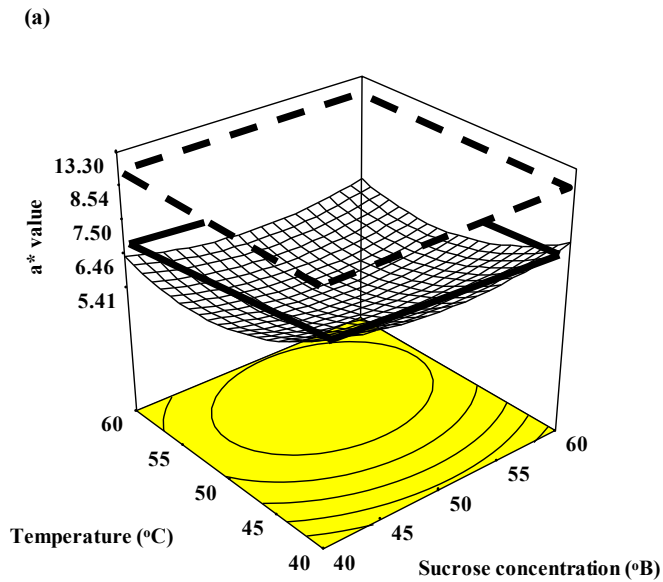
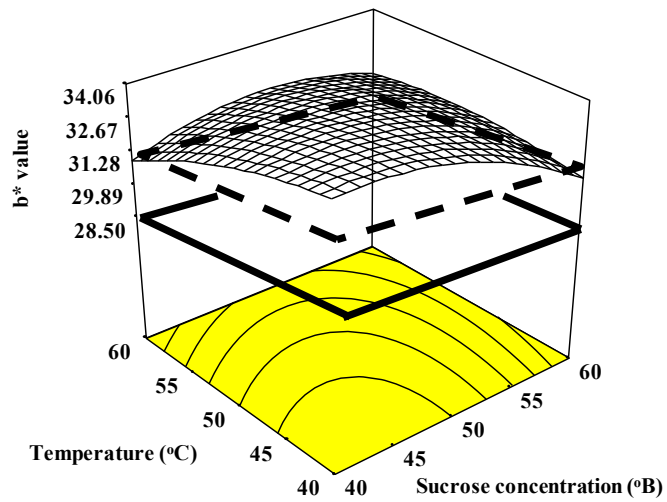


Figure 8.4 Response surface curves for a^* value (a) effect of sucrose concentration and temperature at contact time = 30 min; (b) effect of sucrose concentration and contact time at temperature = 30 $^{\circ}\text{C}$. The perimeter with the dash line is included for air-dried sample with a^* value of 13.3, and the perimeter with solid line shows the a^* Value by the freeze- dried sample (6.6)

(a)



(b)

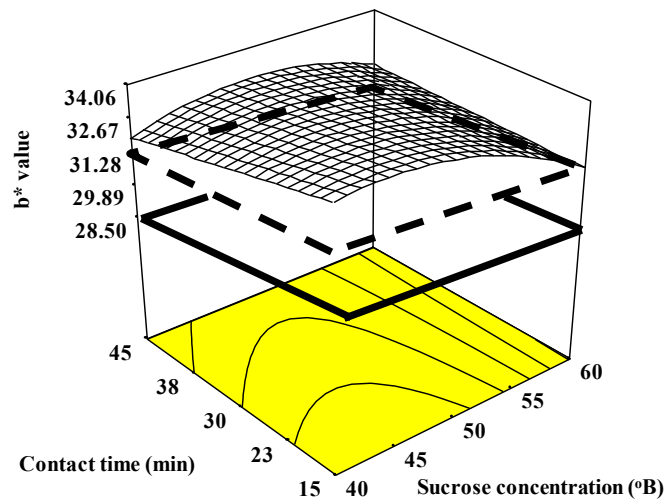


Figure 8.5 Response surface curves for b* value (a) effect of sucrose concentration and temperature at contact time = 30 min; (b) effect of sucrose concentration and contact time at temperature = 30 °C. The perimeter with the dash line is included for air-dried sample with b* value of 31.2, and the perimeter with solid line shows the b* Value by the freeze- dried sample (28.5)

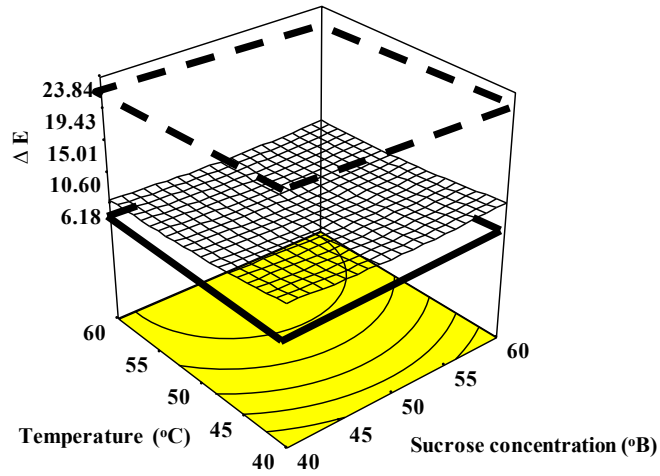
8.3.2.2 ΔE , chroma, hue angle

The results given in Table 8.3 reveal that ΔE , were significantly ($P < 0.0001$) affected by linear effects of temperature and contact time, all quadratic effect of independent variables, whereas the interaction effects of sucrose concentration, temperature and contact time were not significant ($P > 0.05$). The importance of the independent variables on ΔE could be ranked in the following order: contact time > temperature > sucrose concentration (based on the sum of squares).

The chroma value was significantly ($P < 0.05$) affected by all linear and quadratic and interaction effects of (sucrose concentration and temperature) and (sucrose concentration and contact time). The importance of the independent variables on chroma could be ranked in the following order: temperature > sucrose concentration > contact time (based on the sum of squares). The hue angle was significantly ($P < 0.05$) affected by linear effect of temperature and contact time, and all quadratic effects of sucrose concentration, temperature and contact time, whereas the interaction of independent variables were not significant ($P > 0.05$). The importance of the independent variables on hue angle could be ranked in the following order: contact time > temperature > sucrose concentration (based on the sum of squares).

The effect of changing MWODS pre-treatment sucrose concentration and temperature and contact time on the ΔE is given in Figure 8.6 (a,b) and compared with the untreated and freeze-dried samples. ΔE color intensity is the combination of L^* , a^* and b^* values which is extensively applied to present the color variance of foods during processing. Figure 8.6 (a, b) shows that increasing sucrose concentration, temperature and contact time results in decreasing ΔE , whereas at higher level of independent variables b^* value was increased. A comparison with other drying methods revealed that ΔE was lowered in MWODS air-dried apples than in AD ones, while it was very close to FD apples. Falade et al. (2007) reported the same results during drying watermelon.

(a)



(b)

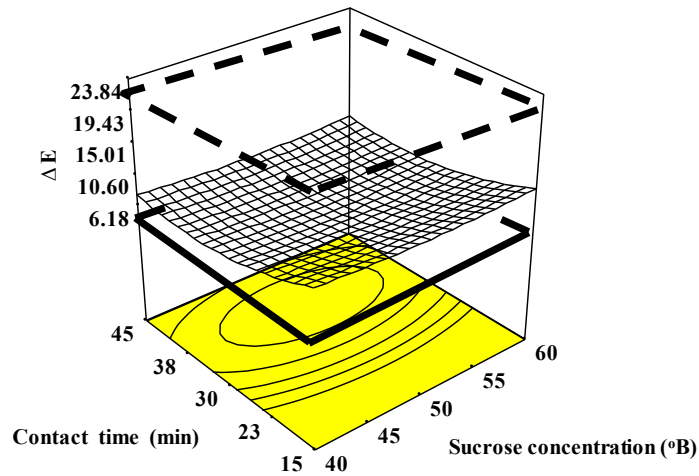
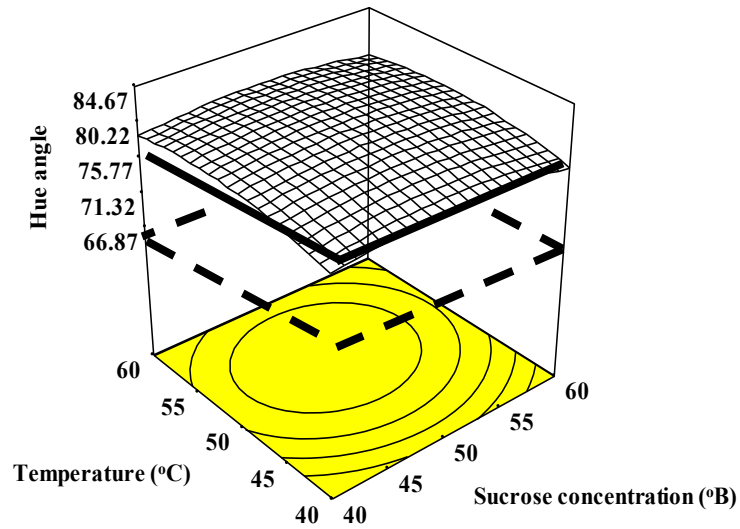


Figure 8.6 Response surface curves for total color difference (ΔE) (a) effect of sucrose concentration and temperature at contact time = 30 min; (b) effect of sucrose concentration and contact time at temperature = 30 °C. The perimeter with the dash line is included for air-dried sample with ΔE of 22.84, and the perimeter with solid line shows the ΔE by the freeze-dried sample (6.64)

Figure 8.7(a,b) presents the effect of sucrose concentration, temperature and contact time on the hue angle. The results revealed that increasing independent variables results in increasing hue angle while at higher level of sucrose concentration, temperature and contact time, hue angle was decreased. Hue angle is the average of red, yellow and blue. If the value of hue angle is higher than 90° , this means that the produce is less yellow and greener. On the other hand, when the hue angle value is less than 90° , this means that the produce is orange-red color (Waliszewski et al., 2002). The results show that MWODS pre-treatment remarkably increased the hue angle which is higher than AD. Comparison of MWODS air-dried with freeze dried samples show that at some points ($60^{\circ}\text{B}/60^{\circ}\text{C}$), the hue angle was even higher than in the freeze- dried sample. Figure 8.8(a,b) presents the effect of sucrose concentration, temperature and contact time on the chroma value which is the degree of color saturation and relates to the strength of the color. A greater chroma value represents a more pure and intense color (Pomeranz and Meloan, 1994; Rodrigues et al., 2003). Chroma value of MWODS pre-treatment apples was increased by increasing sugar concentration (Falade et al., 2007), whereas at higher sucrose concentration, chroma decreased. Increasing temperature and contact time decreased chroma. Moreover, the results showed that values of chroma of MWODS dried samples under different conditions were close to the ones for FD apples, while the difference between AD and MWODS dried apples was obvious. This indicates the stability of the yellow color in apples (Moreno et al., 2000).

(a)



(b)

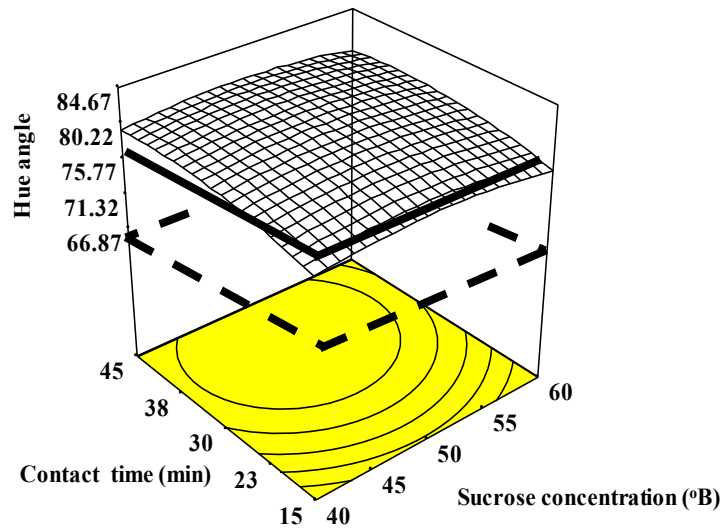
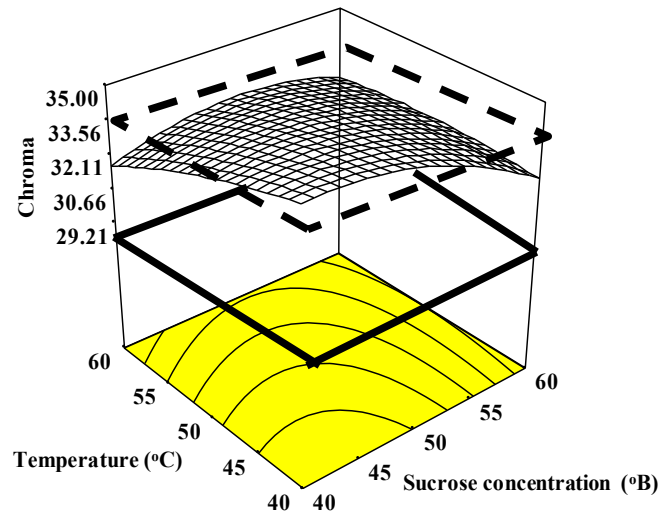


Figure 8.7 Response surface curves for Hue angle (a) effect of sucrose concentration and temperature at contact time = 30 min; (b) effect of sucrose concentration and contact time at temperature = 30 °C. The perimeter with the dash line is included for air-dried sample with Hue angle of 66.87, and the perimeter with solid line shows the ΔE by the freeze- dried sample (78.38)

(a)



(b)

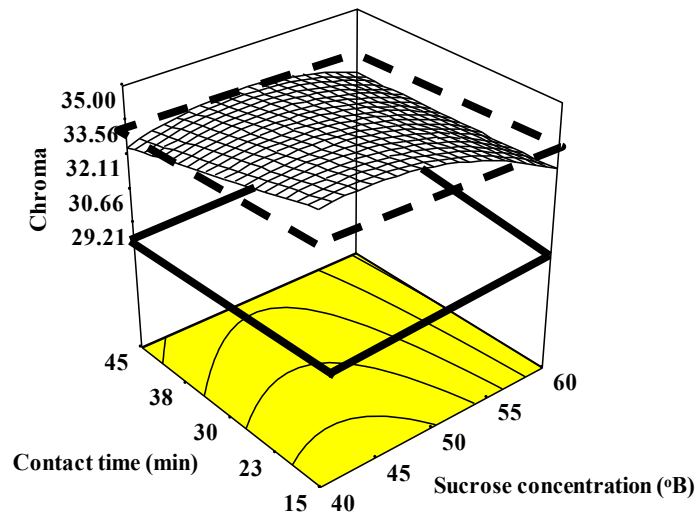


Figure 8.8 Response surface curves for Chroma (a) effect of sucrose concentration and temperature at contact time = 30 min; (b) effect of sucrose concentration and contact time at temperature = 30 °C. The perimeter with the dash line is included for air-dried sample with Chroma of 33.96, and the perimeter with solid line shows the Chroma by the freeze- dried sample (29.21)

8.3.3 Effect of process variables on mechanical responses

8.3.3.1 Hardness

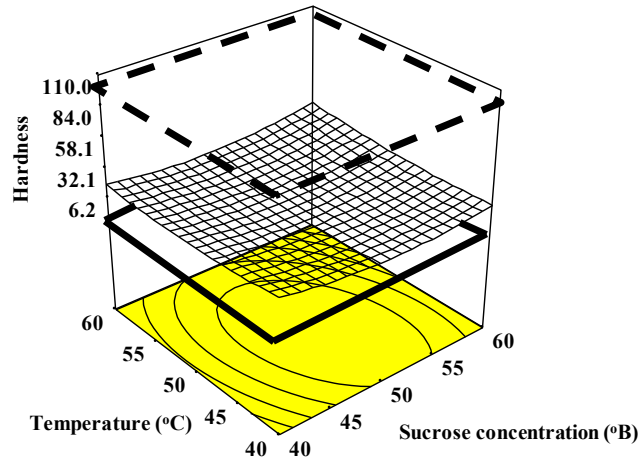
Texture measurements of MWODS air-dried, AD and FD apples have been carried out through puncture force. The mean values of mechanical responses (hardness, energy and rigidity) in MWODS air-dried apples can be observed in Table 8.2. The fitted model for the mechanical responses is shown in Table 8.4. As can be seen in Table 4, hardness was significantly affected by linear effect of temperature and all quadratic effects of sucrose concentration, temperature and contact time, and the interaction effects of (sucrose concentration with temperature) ($P < 0.0001$). The importance of the independent variables on hardness could be ranked in the following order: temperature>contact time>sucrose concentration (based on the sum of squares).

Figure 8.9 (a, b) shows the interaction of (sucrose concentration with temperature) and (sucrose concentration with contact time) on hardness, which is the maximum force in the force-distance curve. From the plot, it can be seen that the hardness of dried apples is decreased by an increase in the sucrose concentration. Textural properties of fruits are closely linked to cellular structure and pectic composition. Adelmo et al., (1993) observed tissue softening during OD of Red Delicious apple cylinders, which was attributed to pectin solubilization and associated cell separation during soaking. At higher sucrose concentration > 50 °B, the hardness of the samples was increased. This could be due to the blocked pores in the fruit tissue, leading to a thicker cell wall, thereby increasing the hardness of the samples (Prothon et al., 2001).

Increasing temperature and contact time showed the same results as sucrose concentration. As can be seen in Figure 8.9b, contact time > 30 min favors hardness; moderation by the prevailing osmotic concentration difference between the fruit and the solution results in increasing the hardness of apples. Notable differences in the hardness of the MWODS-air-dried and AD samples can be observed. A great increase in hardness has been observed in AD samples while MWODS air-dried samples had a lower hardness and were softer than AD apples (Maltini et al., 1993; Mandala et al., 2005). This could be

explained by losing of turgor and ion movement from the cell wall to the surrounding medium (Lewicki 1998; Castelló et al., 2010). The hardness of FD samples was lower than from the osmotic treatment due to more porosity in its texture (Lin et al.,1998). However, the FD samples were brittle.

(a)



(b)

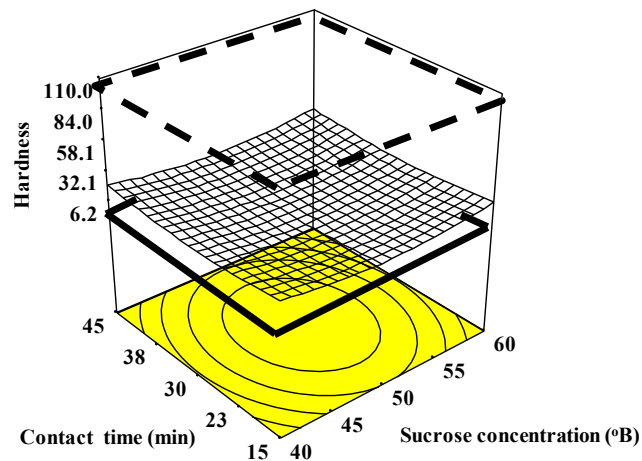


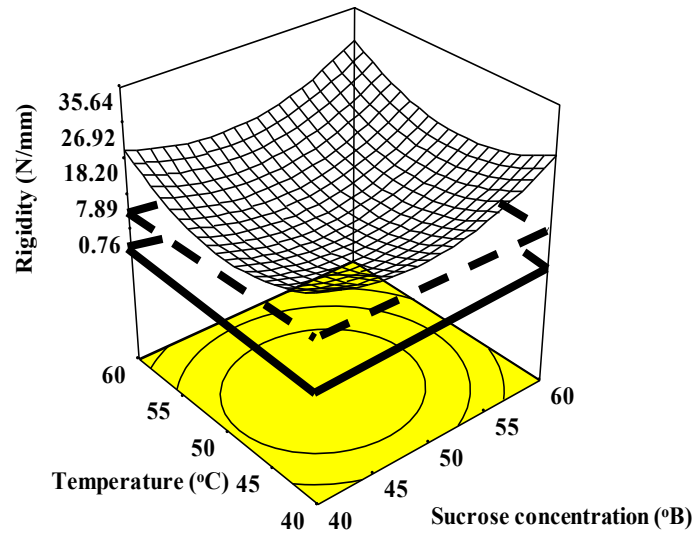
Figure 8.9 Response surface curves for Hardness(N) (a) effect of sucrose concentration and temperature at contact time = 30 min; (b) effect of sucrose concentration and contact time at temperature = 30°C. The perimeter with the dash line is included for air-dried sample with Hardness of 110N, and the perimeter with solid line shows the Hardness by the freeze- dried sample (6.17N)

8.3.3.2 Rigidity

The results given in Table 8.4 show that all linear , quadratic effect of independent variables, and all interaction affects of (sucrose concentration*contact time) and (temperature *contact time) on the rigidity were significant ($P < 0.0001$). The fitted model for the mechanical responses is shown in Table 8.4. The importance of the independent variables on rigidity could be ranked in the following order: contact time > sucrose concentration > temperature (based on the sum of squares).

The variation of rigidity with (sucrose concentration with temperature) and (sucrose concentration with contact time) at constant contact time (30 min) and temperature (50°C); respectively, are presented in Figure 8.10 (a,b). As it shows, increasing sucrose concentration, temperature and contact time results in decreasing the rigidity of apples; however, at higher sucrose concentration and temperature, and contact time the rigidity of apples increased. Comparing MWODS air-dried apples with other methods show that the rigidity of samples in MWODS air-dried apples was higher than in untreated ones which is probably due to solids uptake during the osmotic process; in addition, pectic substances at the middle lamella are redistributed during osmotic dehydration which provides support to plant cells and better structural integrity. However, the loss of turgor pressure in untreated dried samples results in reducing the cell's ability to regain its original form (Khin et al., 2007). The decrease in rigidity in FD apples means that less distance was required to move through a structure of apple, which was due to the porous structure of FD apples, allowing the probe to move the cells easily. Pereira et al. (2006) reported that sucrose concentration treatment in melons preserved the texture characteristics, avoiding severe softening, and that higher sucrose concentration (60°Brix sucrose) resulted in increasing the hardness of dried melon.

(a)



(b)

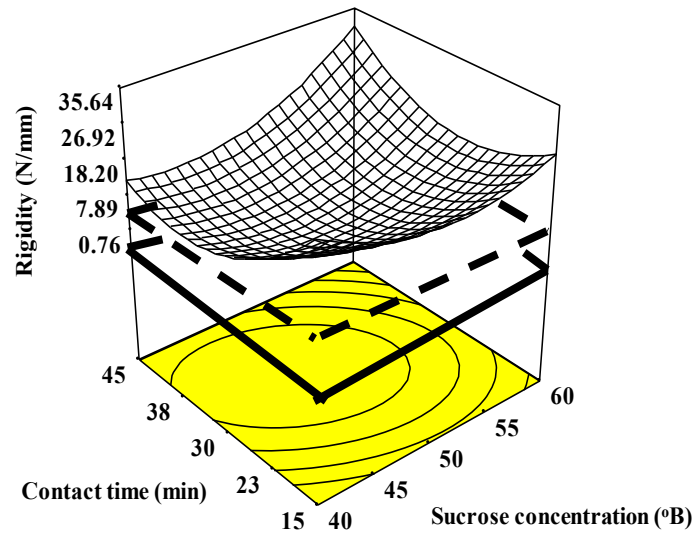


Figure 8.10 Response surface curves for Ridity (N/mm) (a) effect of sucrose concentration and temperature at contact time = 30 min; (b) effect of sucrose concentration and contact time at temperature = 30 °C. The perimeter with the dash line is included for air-dried sample with Ridity of 7.89 (N/mm), and the perimeter with solid line shows the Ridity by the freeze-dried sample (0.76 (N/mm))

8.3.3.3 Absorbed energy during the compression test

The absorbed energy was significantly ($P < 0.0001$) affected by linear, quadratic and interaction effects of sucrose concentration, temperature and contact time. The fitted model for the energy responses is shown in Table 8.4. Based on the sum of squares, the importance of the independent variables on moisture loss could be ranked in the following order: sucrose concentration > temperature > contact time.

The variation of energy with (sucrose concentration and temperature) and (sucrose concentration and contact time) at constant contact time (30 min) and temperature 50°C; respectively, are presented in Figure 8.11 (a, b). As it shows, increasing sucrose concentration, temperature and contact time result in decreasing the energy; however, higher sucrose concentration, temperature, and contact time (at the center point), result in increasing the area under the force-distance curve of the samples. As can be seen the energy response of MWODS air-dried apple was lower than the one of the untreated samples which means that osmotic dehydrated samples were softer. During air drying, the internal structure of the fruit is deformed resulting in formation of crystalline regions in the amorphous polymers due to cross-linking of polymers; water removal added more rigidity to the external layers; as a result, the energy in AD samples increased (Lewicki and Jakubczyk, 2004). The outer layers of untreated air-dried apples become rigid, and considerable mechanical strength is thereby acquired (Lewicki et al., 1997). The energy associated with MWODS samples was higher than FD ones, because the infusion of sugar inside the fruit resulted in increasing the viscous nature of fruit and decreasing its elasticity (Krokida et al., 2000a). The energy of FD samples was lower which is due to the prose sample.

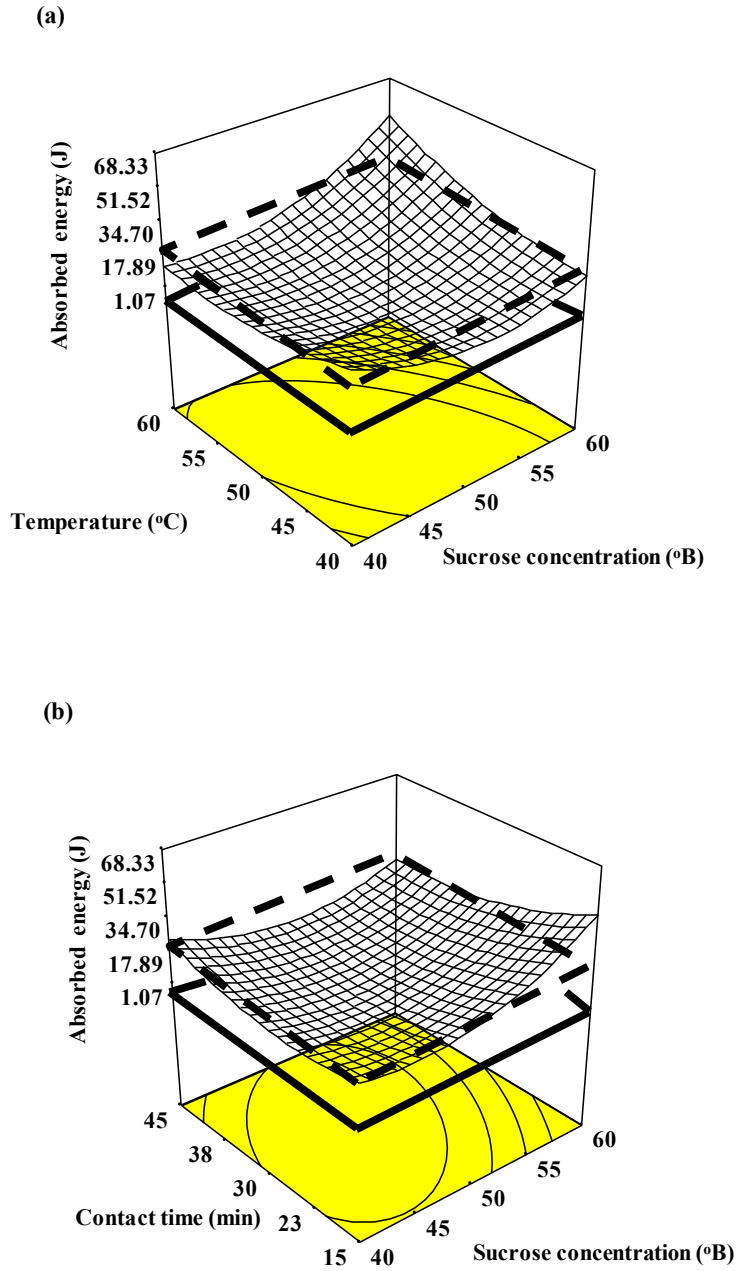


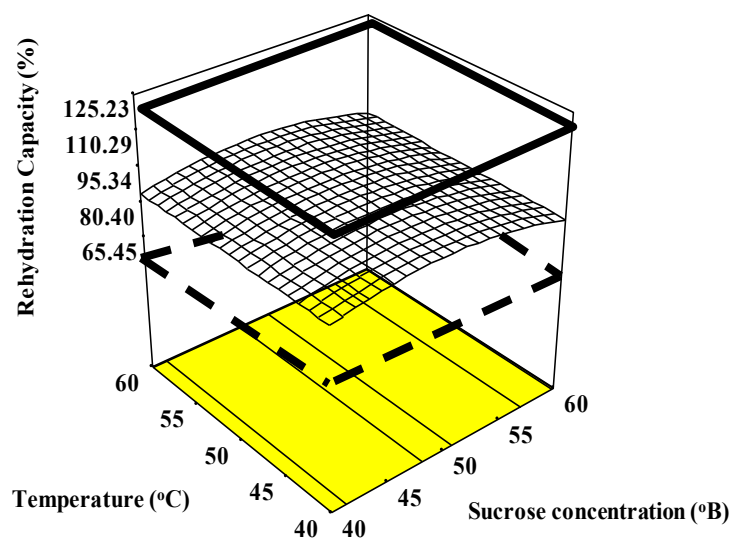
Figure 8.11 Response surface curves for absorbed energy(a) effect of sucrose concentration and temperature at contact time= 30 min; (b) effect of sucrose concentration and contact time at temperature = 30 °C. The perimeter with the dash line is included for air-dried sample with an absorbed energy of 27.8 (J) and the perimeter with solid line shows the absorbed energy by the freeze- dried sample (1.07J)

8.3.4 Effect of process variables on rehydration capacity

Table 8.2 gives the mean value of rehydration capacity (RC) for MWODS air-dried apples. The results given in Table 8.4 show that all linear and quadratic effects of independent variables and interaction effect of (temperature and contact time) had a significant impact on rehydration capacity ($P < 0.0001$). Based on the sum of squares, the importance of the independent variables on moisture loss could be ranked in the following order: contact time > temperature > sucrose concentration.

Figure 8.12 (a, b) shows the response surface plot of RC vs. two independent variables (sucrose concentration and temperature) and (sucrose concentration and contact time), respectively. The results indicate that increasing sucrose concentration and temperature results in increasing rehydration capacity, however at sucrose concentration > 50°B, the RC was reduced. It might be due to accumulation of sucrose molecules along the surface of cytoplasm resulting in formation of a dense superficial layer, which could have actually decreased the water absorption (Nsonzi and Ramaswamy, 1998b). Increasing temperature results in increasing RC, whereas, increasing contact time had a negative effect on RC and results in decreasing RC. Comparing three methods of drying revealed that RC in MWODS air-dried samples was higher than in untreated air-dried and lower than freeze-dried samples. This could be the result of the fact that freeze-dried samples are more porous, and the cell walls are more permeable to adsorption of water; therefore, RC is higher than other methods. Similar results were obtained by (Prothon et al., 2001).

(a)



(b)

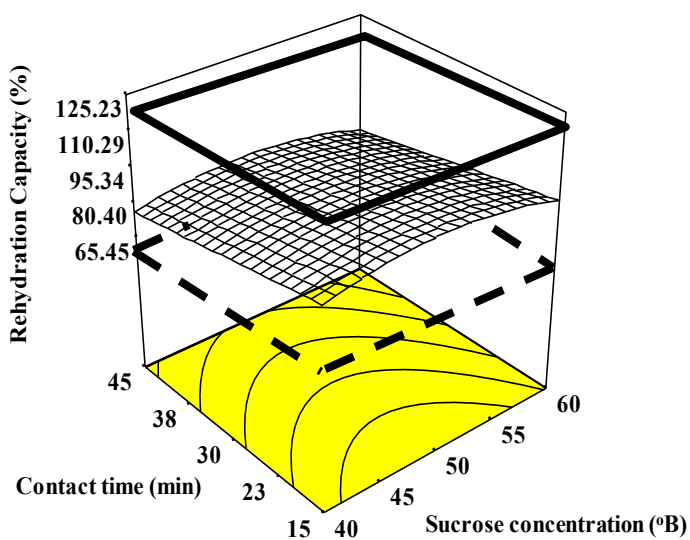


Figure 8.12 Response surface curves for Rehydration Capacity (%) (a) effect of sucrose concentration and temperature at contact time= 30 min; (b) effect of sucrose concentration and contact time at Temperature = 30°C. The perimeter with the dash line is included for air-dried sample with Rehydration capacity of 65.45%, and the perimeter with solid line shows the Rehydration capacity by the freeze- dried sample (120.23%)

8.3.5 Optimization

The optimal conditions for the MWODS air-dried apples were predicted using the optimization function of the Design Expert Software. These are presented in Table 8.5. The optimum condition for MWODS air-dried apples was determined to obtain maximum L* value, minimum ΔE , minimum hardness, and maximum rehydration capacity, while sucrose concentration, temperature and contact time were kept in the range (40-60°B) and (40-60°C), and (15, 45 min) respectively. Among the various optimum conditions, the highest value of L: 82.27, hardness: 7.1 N rehydration capacity: 88.49 , and minimum ΔE : 6.2, were provided by using sucrose concentrations of 49.61°B, a temperature of 51.87 °C and contact time of 33.3 min with the 0.90 desirability.

Table 8.5 Results of optimization by desirability function

Name	Goal	Limit	Limit	Weight	Weight	Importance		
C	is in range	40	60	1	1	3		
T	is in range	40	60	1	1	3		
t	is in range	15	45	1	1	3		
L	maximize	72.02	83.55	1	1	3		
ΔE	minimize	5.93	15.72	1	1	3		
Hardness	minimize	3.30	29.97	1	1	3		
RC	is in range	64.78	122.26	1	1	3		
Solutions								
Number	C	T	t	L	ΔE	Hardness	RC	Desirability
1	49.61	51.87	33.37	82.27	6.20	7.12	88.50	0.904
2	49.62	51.87	33.37	82.27	6.20	7.12	88.50	0.904
3	49.60	51.86	33.36	82.27	6.20	7.12	88.50	0.904
4	49.62	51.84	33.38	82.27	6.20	7.12	88.48	0.904
5	49.62	51.87	33.35	82.27	6.20	7.12	88.51	0.904
6	49.60	51.87	33.35	82.27	6.20	7.12	88.51	0.904
7	49.62	51.86	33.39	82.27	6.20	7.12	88.48	0.904
8	49.62	51.88	33.38	82.27	6.20	7.13	88.49	0.904
9	49.62	51.88	33.38	82.27	6.20	7.13	88.49	0.904
10	49.63	51.85	33.39	82.27	6.20	7.12	88.48	0.904

8.4 Conclusions

In conclusion, a dehydrated product with less color change and a more rigid and softer structure was obtained by the MWODS air-dried apples. The MWODS air-dried samples exhibited higher values for all the color parameters than seen in the un-treated air-dried samples and closed to FD apples. Generally, L^* and b^* value increased in the MWODS air-dried apples while a^* values decreased. Higher color intensity and chroma values were recorded in MWODS air-dried apples than in the air-dried ones. In addition, the texture of MWODS air-dried samples was softer than untreated air-dried ones, while FD apples were more brittle. Finally, the rehydration capacity of MWODS air-dried is higher than the AD and lower than FD apple cylinders.

CHAPTER 9. GENERAL CONCLUSIONS

In this study, the microwave osmotic dehydration under spray mode (MWODS) processing conditions was designed, developed, evaluated and optimized. A second stage air-drying finishing was used to investigate the quality of the final product. The following findings were the specific highlights of the study:

- Microwave osmotic dehydration combination under continuous flow medium spray (MWODS) processing conditions was developed for the first time to improve moisture transfer rate and simultaneously limit the solids gain rate.
- The MWODS process was compared with other existing methods under similar flow conditions [MWOD under immersion, MWODI, conventional osmotic dehydration under spray (CODS) and immersion (CODI) modes] and the MWODS process was demonstrated to offer superior moisture loss rate and reduced solids gain as compared to other techniques.
- In general ML rates were higher in MW mode as compared to conventional modes under both spray and immersion heating conditions
- Further, ML rates were higher in spray mode as compared to immersion mode in both MW and conventional systems.
- The ratio of moisture loss/solids gain (ML/SG) is an important indicator of process efficiency in terms of higher moisture removal relative to solids uptake and is generally taken as an index of producing better quality OD products. The MWODS process gave consistently higher ML/SG ratio as compared with the other methods.
- To compare the effectiveness of different osmotic drying conditions, one more parameter was used that gives the cumulative time effect of the drying process. This was defined as the dehydration time (t_w , t_m and t_s) needed to reach moisture loss, solids gain, or weight reduction to specified target levels. The t_m and t_w values were considerably shorter and t_s longer with the MWODS as compared to other methods.

- Moisture diffusivity (D_m) was higher and solids diffusivity (D_s) was lower with MWODS as compared to other methods
- The two-parameter Azuara model was demonstrated to be adequate to describe the transient mass transfer kinetics, and useful in computing the equilibrium point for the moisture loss and solids gain based on the short duration osmotic treatments, rather than waiting for the real equilibration to be achieved.
- Fick's second law is generally used to model the mass transfer during osmotic dehydration. Fick's equation of unsteady state diffusion was used to calculate the mass diffusion coefficients representing moisture loss (D_m) and solid gain (D_s) during osmotic dehydration process under continuous flow conditions.
- The D_m values were higher and D_s values were lower with MWODS as compared to the other methods. D_m and D_s were dependent on temperature and concentration of the osmotic solution.
- Both Azuara and Fickian diffusion models were shown to be adequate in describing the mass transfer kinetics during the MWODS. The Azuara model was a better predictor than the diffusion model. In order to use the diffusion model with better predictions, it was necessary to add the intercept parameters making the diffusion model also a two-parameter model similar to Azuara model.
- Half-drying time under different conditions is an effective measure of the rate of drying. The half-drying times were reciprocally related to the diffusion coefficients.
- A CCRD model combined with RSM was used for more detailed evaluation of mass transfer kinetics of apples under a wide range of MWODS processing conditions. The mass transfer kinetics under MWODS processing conditions demonstrated trends similar to those associated with classical OD process. The kinetic parameters - ML, SG and WR – were related to process variables through RSM analysis and response surface plots were generated to show the trends in their variations.
- Response surface methodology was also used to optimize the MOWDS process parameters based on target constraints like maximizing ML, minimizing SG, etc.

and the range the osmotic dehydration processing conditions for optimal processes were developed.

- Air drying was used as the second stage drying for reducing the moisture content of the osmotically treated products to achieve shelf-stability. The air-drying kinetic parameters were related to MWODS pre-treatments in order to optimize the overall process. The moisture diffusivity during air drying was higher in the MWODS pre-treated samples than in those without pre-treatment. Thus it was possible to reduce the air drying times through the MWODS treatment.
- The color parameters [Lightness (L^*), redness (a^*), yellowness (b^*), color intensity (ΔE), chroma and hue angle] of MWODS pretreated and subsequently air dried products were evaluated. These parameters were influenced by the osmotic treatment variables like osmotic solution (sucrose) concentration and temperature. Air-dried (AD) apple cylinders were darker, whereas MWODS air-dried samples were lighter with higher L^* and b^* values, lower a^* value, and higher hue and chroma values and lower (ΔE).
- Color parameters results showed that the MWODS treated products were close or equal to the freeze-dried (FD) apples.
- During osmotic dehydration, apple cylinders lost water and gained sucrose, resulting in changes to the texture of the product. The maximum force (hardness) was decreased by increasing the osmotic sucrose concentration of MWODS pre-treatment producing softer (chewy) dried apples, whereas air-dried samples were hard and FD apples were brittle.
- Rehydration capacity was lower for MWODS than freeze-dried, whereas it was higher than for air-dried samples.

RECOMMENDATIONS FOR FUTURE RESEARCH

This research work has demonstrated not only several important findings but also showed some areas of interests for future development, which could be summarized as follows:

- Scaling up of operation from the bench top system to pilot scale
- Design for continuous movement of the product in addition to the osmotic medium
- Investigating microwave osmotic dehydration equilibrium kinetic study; combining with histological anatomy and microscopy analysis techniques;
- Osmotic dehydration solution management and microbiological study;
- Investigating the effete of microwave osmotic dehydration on product sensory quality and shelf life.
- Development of innovative dehydration process to infuse and trap bioactive compounds in the sample for nutritional value addition;

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