UNIVERSITI PUTRA MALAYSIA

EFFECTS OF THERMAL AND NONTHERMAL TREATMENTS ON KINETICS OF MASS TRANSFER AND SELECTED QUALITY ATTRIBUTES DURING OSMOTIC DEHYDRATION OF SEEDLESS GUAVA (PSIDIUM GUAJAVA L.)

ALI GANJLOO

FSTM 2011 3

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By

ALI GANJLOO

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirement for the Degree of Doctor of Philosophy

April 2011

It seems to me that all sciences are vain and full of error that are not born of experience, mother of all certainty, and are not tested by experience, that is to say, that do not at the origin, middle or end pass through any of the five senses.

Leonardo da Vinci 1452-1519

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

EFFECTS OF THERMAL AND NONTHERMAL TREATMENTS ON KINETICS OF MASS TRANSFER AND SELECTED QUALITY ATTRIBUTES DURING OSMOTIC DEHYDRATION OF SEEDLESS GUAVA (*PSIDIUM GUAJAVA* L.)

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April 2011

Chairman: Professor Russly Abdul Rahman, PhD

Faculty: Food Science and Technology

This study evaluated the effect of some process parameters and selected enhancement techniques on the kinetics of mass transfer as well as selected quality attributes for osmotic dehydration of seedless guava. Experimental results revealed that higher values of solution concentration and temperature resulted in higher flows of water (39%) and solids (8%) through the seedless guava. In all cases, Peleg equation adequately (R^2>0.92) described the kinetics of mass transfer during osmotic dehydration. Total color difference increased up to 21% while hardness decreased around 32% with increase process variables. A zero order kinetic model was fitted to the experimental data adequately for quality parameters (R^2>0.88). The osmotic dehydration process was optimized for maximum water loss, weight reduction and minimum solute gain through response surface methodology. Results suggested optimum processing conditions of 30% w/w sucrose concentration at 33 ºC after 179 min would result in 0.15 gg^{-1} weight reduction, 0.2 gg^{-1} water loss

and 0.03 gg^{-1} solid gain. Results showed that at the studied range of process parameters, the values of mass transfer terms were not in accordance with an efficient osmotic dehydration process in which 40–60% water loss and <10% solid gain are expected (Eren and Kaymak-Ertekin, 2007). In order to improve the rate of mass transfer a number of enhancement methods such as hot water blanching, thermosonication, ultrasound and centrifugal force were applied. The effect of hot water pretreatment at the temperature range of 80-95°C was evaluated and compared with osmotic dehydration at optimum condition. It improves the kinetics of mass transfer in terms of weight reduction, solid gain, water loss and normalized moisture content up to 15-30%, 2-6%, 16-46% and 7-20% at the temperature range of 80-90 °C, respectively. Traditional blanching lead to 17% increase and 54% decrease in total color difference and hardness values, respectively. For the first time, the simultaneous application of heat and ultrasonic wave (thermosonication) was investigated in order to reduce the intensity of heat treatments which can impair sensorial and nutritional properties of foods. Thermosonication at 90 ºC at different amplitude levels (25-75%) lead to the enhancement of mass transfer of water (up to 4%) and solid (up to 1%) during osmotic dehydration without significant ($p>0.05$) changes of optical and textural properties in comparison with traditional blanching. Finally, the influence of ultrasonic wave and centrifugal force as nonthermal treatment on osmotic dehydration process was investigated to overcome the drawbacks of thermal treatment. Application of ultrasonic treatment compared with osmotic dehydration at optimized condition enhanced water loss and solid gain up to 23% and 3.3%, respectively. It improved “L” value without any significant effect ($p>0.05$) on “a” and “b” values whereas hardness value was significantly ($p<0.05$) affected. Centrifugal force treatment increased water loss around 34%, however,

retarded solid gain. The combination of centrifugal force with osmotic dehydration leads to decrease in hardness of samples (5%) whereas there is no significant ($p<0.05$) effect on color of samples.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENGARUH- PENGARUH PERAWATAN TERMA DAN NONTHERMAL PADA KINETIKA TRANSFER MASSA DAN ATRIBUT KUALITAS PILIHAN SELAMA DEHIDRASI OSMOTIK JAMBU BIJI TANPA BIJI (*PSIDIUM GUJAVA* L.)

Oleh

ALI GANJLOO

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Kajian ini menilai kesan beberapa parameter proses teknik maju terpilih keatas kinetik pemindahan jisim serta beberapa atribut kualiti terpilih bagi penyahidratan osmotik buah jambu batu tanpa biji. Keputusan kajian menunjukkan bahawa nilai yang lebih tinggi bagi konsentrasi larutan dan suhu menyebabkan aliran yang lebih tinggi bagi air dan pepejal melalui jambu tanpa biji. Dalam semua kes, persamaan Peleg adalah mencukupi (R^2>0.92) untuk menggambarkan kinetik pemindahan jisim semasa penyahidratan osmotik. Perbezaan warna total meningkat manakala kekerasan menurun apabila pembolehubah proses meningkat. Satu model kinetik orde sifar dimasukkan dengan data dari eksperimen dan menunjukkan keputusan yang mencukupi bagi parameter kualiti (R^2>0.88). Proses penyahidratan osmotik telah dioptimalkan untuk kehilangan air maksimum, penurunan berat dan penambahan larutan minimum melalui metodologi respon permukaan. Keputusan kajian menunjukkan keadaan pemprosesan optimum adalah pada 30% w/w

konsentrasi sukrosa, 33 ºC suhu selepas 179 minit proses akan menghasilkan 0.15 gg^{-1} pengurangan berat, 0.2 gg^{-1} kehilangan air dan 0.03 gg^{-1} penambahan gula. Keputusan kajian menunjukkan bahawa pada julat parameter proses, nilai terma-terma pemindahan jisim adalah tidak bersesuaian dengan proses penyahidratan osmotik cekap di mana kehilangan air pada kadar 40-60% dan <10% peningkatan pepejal diharapkan dapat di capai (Eren and Kaymak-Ertekin, 2007). Untuk meningkatkan pemindahan jisim beberapa kaedah peningkatan seperti penceluran air panas, thermosonikasi, ultrabunyi dan daya emparan digunakan. Kesan prarawatan air panas pada julat suhu 80-95°C dinilai. Ini telah meningkatkan kinetik pemindahan jisim pada julat suhu 80-90°C. Penceluran tradisional menyebabkan kenaikan 17% dan penurunan 54% dalam perbezaan warna total dan nilai-nilai kekerasan, masing-masing. Oleh kerana haba boleh merosakkan sifat pancaindera dan nutrisi makanan, terdapat minat dalam mencari teknologi baru yang mampu mengurangkan kesan rawatan panas. Dari fakta ini, untuk pertama kalinya, aplikasi serentak haba dan gelombang ultrasonik pada pelbagai peringkat intensiti diselidiki. Thermosonikasi mengarah kepada peningkatan pemindahan jisim semasa penyahidratan osmotik tanpa perubahan signifikan ($p>0.05$) sifat optik dan tekstur dibandingkan dengan penceluran tradisional. Seterusnya, pengaruh keamatan ultrasonik dan daya emparan sebagai rawatan bukan termal semasa proses penyahidratan osmotik diselidiki. Rawatan ultrasonik secara signifikan meningkatkan kinetik pemindahan jisim berbanding dengan sampel yang tidak dirawat. Ia meningkat kan nilai-L tanpa memberi kesan yang signifikan ($p>0.05$) pada nilai-a dan nilai-b tetapi nilai kekerasan secara signifikan ($p<0.05$) terjejas. Daya emparan meningkatkan kehilangan air, tetapi, menurunkan peningkatan pepejal oleh produk. Kombinasi daya emparan dengan penyahidratan osmotik menyebabkan

penurunan kekerasan sampel sedangkan ia tidak mempunyai pengaruh signifikan pada warna sampel. Dapat disimpulkan bahawa penyahidratan osmotik emparan adalah sesuai jika kehilangan air dapat ditingkatkan dan peningkatan gula dapat dihadkan.

AKNOWLEDGEMENTS

I pray to Almighty ALLAH who give me the thoughts, the will, and guided me to complete this work. I pray that ALLAH will bless this work and make it useful for mankind, and that He will forgive us.

My sincere and the deepest gratitude to Professor Dr Russly Abdul Rahman, the chairman of my supervisory committee for his guidance, encouragement, patience and continuous follow up during the course of this study. My appreciation and gratitude is also extended to members of my supervisory committee, Professor Dr Jamilah Bakar and Professor Dr Azizah Osman for their advices, punctuates comments and supports.

My gratitude is also due to all the staff of the Department of Food Technology, and Faculty of Food Science and Technology, UPM for their cooperation. I would like to acknowledge the financial support received from the RMC, the Universiti Putra Malaysia for this project.

I would also like to give my special thanks to my wife, Mandana Bimakr who brightens my life with her support, encouragement, sacrifice and patience.

Finally, I must express my deepest gratitude to my parents continuously encouraged me and presented me the most beautiful World.

I certify that a Thesis Examination Committee has met on 29 April 2011 to conduct the final examination of Ali Ganjloo on his thesis entitled "Effects of Thermal and Nonthermal Treatments on Kinetics of Mass Transfer and Selected Quality Attributes during Osmotic Dehydration of Seedless Guava (*Psidium Guajava* L.)" in accordance with the universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U. (A) 106] 15 March 1998. The committee recommends that the student be awarded the Doctor of Philosophy.

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DECLARATION

I declare that the thesis is my original work except for quotations and citations, which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institutions.

_____

ALI GANJLOO

Date: 29-April-2011

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LIST OF SYMBOLS AND ABBREVIATIONS

List of Symbols		Unit
% w/w	Weight per weight percent	
ANOVA	Analysis of Variance	
AOAC	Analysis Official Committee	
min	Minute	
NMC	Normalized Moisture Content	%
WL	Water Loss	g/g
WR	Weight Reduction	g/g
SG	Solid Gain	g/g
g	Gram	
s	Second	
h	Hour	
K_1	Peleg Rate Constant	min g/g^{-1}
K_2	Peleg Capacity Constant	$(g/g)^{-1}$
CRD	Complete Randomized Design	
R^2	Correlation Coefficient	
χ^2	Chi-square	
RMSE	Root Mean Square Error	
E	Mean Relative Deviation Modulus	
CCD	Central Composite Design	
RSM	Response Surface Methodology	
p	Significance Difference	
WL/SG	Water Loss to Solid Gain Ratio	

K_0	Frequency Factor	min^{-1}
E_a	Activation Energy	$kJmol^{-1}$
R	Universal Gas Constant	8.314J/mol K
T	Absolute Temperature	ºK or ºC
C	Sucrose Concentration	% w/w
S.E.	Standard Error	
β	Regression Coefficient	
L	Lightness	
a	Redness and Greenness	
b	Yellowness and Blueness	
ΔE	Total Color Difference	
mm	Millimetre	
k	Rate Constant	min^{-1} or s^{-1}
SD	Standard Deviation	
μm	Micrometer	
mL	Millilitre	
w/v	Weight per Volume	
RPM	Rotation per Minute	
C	Moisture content/mean solid content after time t	
θ	Time	
z	Thickness	
D	Diffusion coefficient for moisture in solids	m^2/s
m	Moisture content	
s	Solid content	

F_{ow}	Fourier number for moisture diffusion = D_{ew} t/l^2
F_{os}	Fourier number for solid diffusion= D_{es} t/l^2
D_{ew}	Effective diffusion coefficients for water
D_{es}	Effective diffusion coefficients for solute
l	Half thickness of the infinite slab
q_n	non-zero positive roots of the equation tan q_n= -αq_n
α	Ratio of the volume of solution to that of each piece
M_r	Moisture ratio
S_r	Solute ratio
C_n	2α (1+α)/(1+α+$\alpha^2 q_n{}^2$)
$a\alpha_n's$	Roots of the equation $J_0(a\alpha_n) = 0$
C_{pn}	Equal to 2α (1+α)/(1+α+α^2 $q_{pn}{}^2$)
q_{pn}	non-zero positive roots of the equation tan q_{pn} =αq_{pn}
C_{cn}	Equal to 4α(1+α)/(4+4α+α^2 $q_{pn}{}^2$)
q_{cn}	non-zero positive roots of the equation α$q_{cn}$$J_0$($q_{cn}$)+2 J_1(q_{cn})=0
□	open void fraction
D	Diffusivity of the substance
τ	Tortuosity factor which corrects for the path longer than the diffusion distance
w	Water loss by food at any time t

w_e	Equilibrium water loss	
w_m	Maximum water that can diffused out but remains in the food at time t	
S_e	Equilibrium solid gain	
m_t and m_0	Final (time t) and initial sample mass	
x_w and x_{w0}	Final (time t) and initial moisture contents	
x_{sst} and x_{ss0}	Final (time t) and initial salt contents in the sample	
$2r$	Diameter of Finite Cylinder	
$2l$	Height of finite cylinder	
m_w	Mass of water	g
m_s	Mass of salt	g
0	Initial	
∞	Equilibrium	

CHAPTER I

GENERAL INTRODUCTION

The beginning of food preservation by dehydration is not clear. Historically, our ancestors dried foods by trial and error. In the 18th century, first dehydration was done for vegetables. Thereafter, war scenarıos around the world lead to development of drying industry because of the weight and space saving capabilities of the process. In the United States, by the end of the18th century, artificial dryers were developed to replace sun drying. Before the Second World War, drum drying and spray drying was developed to produce milk and eggs products. Drying or dehydration is a water removal process using heat to slow down the rate of metabolic activity of spoilage microorganisms and occurrence of chemical reactions. In addition, drying of foods reduce difficulties of handling, storing, packaging and transporting. In traditional drying methods, high temperatures and long drying times lead to water removal which causes serious reduction in nutritive value, damaging physico-chemical and organoleptical properties of the final products (Lenart, 1996; Telis-Romero *et al*., 2004). Demand for natural, healthy and tasty processed foods increases all over the world and so there is growing attention in improvement of the quality of dehydrated products. Recently, research was widely carried out to explore that osmotic dehydration can be an alternative technology individually or in combination with other techniques to obtain products as well as fresh foods (Torreggiani, 1993; Corzo and Bracho, 2005; Tocci and Mascheroni, 2007; Changrue *et al*., 2008; Lombard *et al*., 2008). This technique has been widely used in food preservation due to its many advantages over the traditional drying methods. The advantages of osmotic

dehydration include retention of sensorial and nutritional properties of dehydrated product (Telis-Romero *et al*., 2004), minimize physico-chemical changes during storage (Koc *et al*., 2008), protect the thermo sensitive compounds such as vitamins and flavors (Tocci and Mascheroni, 2007), reduce energy consumption in subsequent drying process and prolonging shelf-life due to increase in sugar/acid ratio of final product (Lombard *et al*., 2008). So far, too little attention has been focused on osmotic dehydration of guava fruit, *Psidium guajava* L., which is an important origin of minerals and vitamins to the populations of tropical climate. On the other hand, investigation on factors affecting osmotic dehydration of particular food stuff supplies valuable information on those parameters and their levels. Furthermore, study on kinetics of mass transfer as well as quality attributes would augment knowledge on osmotic process. Transportation of water and solutes during osmotic dehydration takes place across semi-permeable cell membranes. Thus, mass transfer rate is commonly low which gives the required motivation for searching new techniques to accelerate the mass transfer rate. During osmotic treatment application of various thermal methods such as steam, hot water and microwave blanching increase the kinetic of mass transfer.

A great amount of research deals with finding new methodologies which would be able to decrease the degree of the heat treatments, since application of heat can spoil many organoleptical and nutritional properties of foods. The use of ultrasound combined with heat generates novel and useful methodologies which benfits different processes including reduced processing times and increased efficiency has been reported in several studies (Cruz *et al*., 2006). In the literature, no information

is available about using thermosonication for improving efficiency of the osmotic dehydration process.

Recently, there are a great number of studies on nonthermal techniques for food processing and preservation due to growing consumer demands for processed fresh-like food with high nutritional and sensorial qualities. These techniques include subjecting the food to pulsed electric field (Amami *et al*., 2007), hydrostatic pressure (Oey *et al*., 2008) and gamma irradiation (Rastogi *et al*., 2006) before osmotic dehydration and application of ultrasonic waves (Stojanovic and Silva, 2007; Fernandes and Rodrigues, 2008), partial vacuum (Deng and Zhao, 2008) and centrifugal force (Amami *et al*., 2007) during or prior to osmosis operation.

Therefore, the specific objectives of the thesis are:

1. To evaluate the effects of process variables (concentration, temperature and immersion time) on osmotic dehydration kinetics of seedless guava. Peleg equation was applied to analyze osmotic dehydration kinetics; and optimiziation of osmosis process using response surface methodology.
2. To investigate the influence of process variables on quality attributes (color and texture) of seedless guava and determine the kinetics of quality attributes changes using mathematical models during the process.
3. To determine the effects of traditional thermal pretreatment (hot water blanching) and thermosonication on osmotic dehydration kinetics and quality attributes of seedless guava.
4. To investigate the influence of ultrasound and centrifugal force treatments on osmotic dehydration kinetics and quality parameters of seedless guava.

CHAPTER II

LITERATURE REVIEW

2.1 Guavas

Guava, *Psidium guajava* L., belongs to the *Myrtaceae*, believed to originate in the Caribbean and commonly has known either as guava or *jambu batu* in Malaysia. The shape of guavas are in the range of oblong to pear but because of out-crossing, some elongate to round variants exist. It has a light yellow, pink blushed skin with white, red or salmon-colored flesh. Guavas emit a strong, sweet, pungent fragrance with flavor ranges from strawberry to lemon (Morton, 1978; Chen *et al*., 2006). The fruit weighs 400-600 g on the average, but can reach a weight of 1 kg. Freshly harvested mature or ripe guava fruit is very popular in Malaysia. Guavas like other tropical fruits continue to ripen after harvest and should not be refrigerated unless overripe.

2.1.1 Varieties

The guava bears fruit all year round in Malaysia. The commercial varieties grown in Malaysia usually have light-green skin when ripe, though there are varieties with yellow skins and are smooth with very faint grooves radiating from the stalk end. Guavas are generally sweet which the flesh may be white, light-yellow, pink or salmon; with textures ranging from crunchy to pulpy (Morton, 1987; Chen *et al*., 2006). Table 2.1 summerizes the characteristics of guava fruit.

2.1.2 Some Physicochemical Properties of Guava

Guava consists of 90.91% water and 10% solids. Their density is 1050 kg/m^3, specific heat is 3.97 kJ/kgºC and thermal conductivity is 0.56 W/mºC (Shamsudin *et al*., 2005). Table 2.2 presents physico-chemical properties of seedless guava fruit.

2.1.3 Nutritional Content of Guava

Guava is a great fruit because it has great amount of nutrients such as vitamins (C, A and B) and good sources of soluble fiber and nicotinic acid. Calcium is typically not found in high amounts in many fruits though it is available in guava fruit. They are very good for the immune system and are beneficial in reducing low-density lipoprotein and protecting the heart. Several studies revealed that higher consumption of fruits and vegetables which are rich in vitamin C, carotenoids and dietary fiber lead to lower risk of cancer among people. Table 2.3 shows nutritional values of guava fruit.

Table 2.1. Summary of Guava Fruit Characteristics

Variety	Fruit characters				
	Shape	Size	Color	TSS	Texture & taste
Kampuchea (GU8)	Oblong to pear shape, elongate to round	400-600g, up to 1 kg	Skin: light yellow to light green. Flesh: white	6.5to7º Brix	Crunchy and sweet
Taiwan	Pear-shape	300-500g, up to 1 kg	Flesh: white	6.8º Brix	Crunchy and sweet-acid
Gloom Toon Klau (GU9)	Rotund to oblate	400-500g	Flesh: white	8-10º Brix	Crunchy and sweet
Gloom Sali (GU10)	Globose	200-400g	Skin: light green Flesh: white	6-7º Brix	Crunchy and sweet
Thai Seedless	Round and asymmetrical	150-300g	Skin: green Flesh: white		Crunchy and sweet
Bangkok Apple	Oblate to globe, misshapen and asymmetrical	100-250g	Skin: light green Flesh: white with faint pink tinge	10-11º Brix	Crunchy and sweet
Beaumont	Oblong	230-250g	Flesh: pink	9.9ºBrix Brix	Soft, mild acid
Burma Red	Globe	150-200g	Flesh: pink	-	Sweet-acid

(Sources: Morton, 1987; Popenoe, 1974).

Table 2.2. Physico-chemical Properties of Guava

Property	Experimental value (25 ºC)
pH	4.15 ± 0.13
Water activity	1.00 ± 0.05
Glucose (%)	1.38 ± 0.15
Fructose (%)	1.66 ± 0.11
Sucrose (%)	0.74 ± 0.01

(Source: Shamsudin *et al.,* 2005)

Table 2.3. Nutritional Values of Guava Fruit

Nutrient	Mean value (%)
Protein	0.54±0.05
Fat	0.24±0.05
Carbohydrates	3.02±0.25
Fiber	10.65±0.02
Calcium	3.3
Phosphorous	7
Iron	2
Vitamin A	21
B1 (Thiamine)	7
B2 (Riboflavin)	4
B3 (Niacin)	9
Vitamin C (Ascorbic acid)	122 mg

(Sources: Shamsudin *et al.,* 2005; Kaur *et al*., 2009; Correa *et al*., 2010)

2.2 Dehydration and Drying Process

According to Krokida and Marinos-Kouris (2003) the main objective of dehydration and drying is lowering water activity enough to prevent growth of microorganisms and slow down the rate of biological reactions by removal of water from food materials. The mechanisms of drying consist of water movement under capillary forces, diffusion of liquid and surface water vaporization using heat that affects physico-chemical attributes and consequently will change the shape, crispiness,

aroma, hardness, flavor and nutritional value of the fresh foods. Although the terms of drying and dehydration are often interchangeable, it may be necessary to distinguish between the two as "drying" refers to the removal of moisture due to simultaneous mass and heat transfer (i.e. thermal drying). Drying as such, refers primarily to the removal of moisture in the vapor phase, whereas dehydration is a more encompassing term and includes methods of moisture removal which can be done without heat (e.g. compression, reverse osmosis, filtration, etc.).

According to Jayaraman and Das Gupta (1992) drying process of foods categorized into three technologies: Solar and air drying in batch or continuous mode and sub atmospheric drying such as vacuum and freeze dryer. Low temperature/low energy processes such as osmotic dehydration have recently received more attention.

The main drawback in fruit drying is damage to the sensory characteristics and loss of nutritional components due to long periods of drying at relatively high temperatures (Mousa and Farid, 2002). These include the loss of aroma volatiles, oxidation of pigments and vitamins and case-hardening of certain products. Case-hardening is a common defect of dried fruits and is caused by fast drying compared to the product□s moisture diffusion rate. Under these conditions, outer layers tend to overdry and inhibit moisture diffusion, leaving the interior wet.

A variety of novel methods have recently been investigated for drying fruits and vegetables. Among them are vacuum, microwave drying and osmotic dehydration. Recently, application of osmotic dehydration has gained more attention to produce intermediate moisture (IMF) products and reduce the consumption of energy and

heat damage when applied prior to traditional drying process (Jayaraman and Das Gupta, 1992).

2.2 .1 Principle of Osmotic Dehydration

Osmosis is the movement of water through a semi-permeable membrane of fruit or vegetables which occurs when fruits or vegetables are submerged in hypertonic solution. The osmotic pressure difference between cells and surrounding concentrated solution supported the driving force for diffusion of water (Talens *et al*., 2003; El-Aouar *et al*., 2006; Rizzolo *et al*., 2006; Rodrigues and Fernandes, 2007). Osmotic dehydration involves three simultaneous counter-current fluxes of mass transfer (Figure 2.1). The first two are a major flux of water from fruit or vegetable cells to hypertonic solution and flux of solids from the solution into the fruit or vegetable cells (Torregiani and Bertolo, 2001; Osorio *et al*., 2007). Neglectable amount of natural solutes like sugars, salts, minerals and organic acids can leach from the cells into the osmotic solution. This phenomen revealed that membrane which is responsible for osmotic transport is not perfectly selective (Lazarides *et al*., 1997; Matusek *et al*., 2008). According to Bildweel (1979) and Garcia *et al*. (2007) osmosis process characterizes by transfer of more water than solute due to limitation of the solute transfer because of different permeability of cellular membranes.

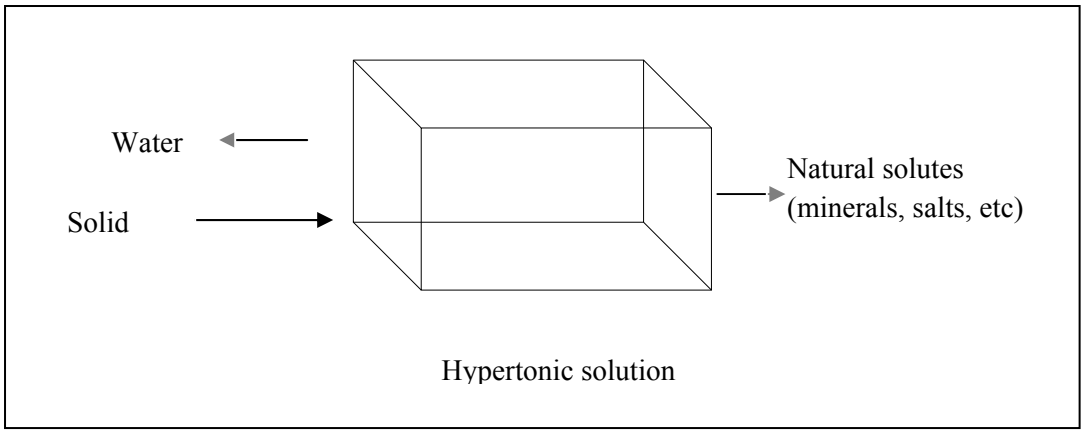


Figure 2.1. Mass Transfer during Osmotic Dehydration

2.2.2 Mechanism of Water Transport in Plant Tissue

The cell is the smallest biological unit with the characteristics of living matter, connected to each other by the middle lamella (Figure 2.2). Tonoplast or vacuolar membrane which separates the vacuolar contents from the cytoplasm and, the plasmalemma or plasma membrane which separates the protoplasm from the primary cell wall are two major membranes of the plant cells. In addition, protoplasmic connections, or plasmodesmata, connect the protoplasm of adjacent cells which this continuous network of protoplasm is called symplasm. The fraction of the cell outside the protoplasm forms another continuous network which known is as apoplasm (McWilliams and Paine, 1977; Noggle and Fritz, 1983; Mayor *et al*., 2007).

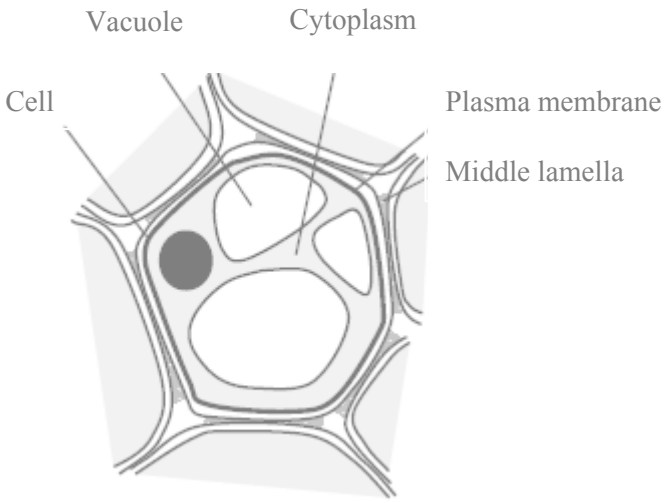


Figure 2.2. The Plant Cell (Mayor *et al*., 2008)

The classic theory of water transportation in plants assumed the water movement from the vacuole of a cell to the neighboring vacuole because of the driving force has been established by the difference in chemical potential of water. Apoplasmic and sympalsic are two ways of water transportation in a plant tissue (Figure 2.3).

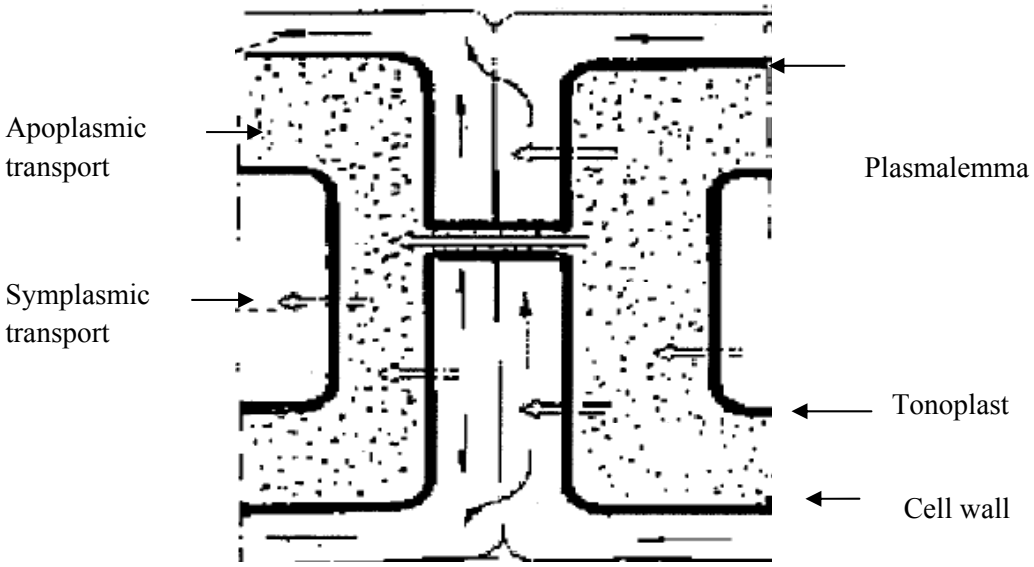


Figure 2.3. Apoplasmic and Symplasmic Transport of Water (Li, 2005)

The nature of protoplasm is generally studied to determine how different substances can penetrate into the living cells. The fact that the protoplast of a plant cell is highly permeable to certain substances, but only slightly permeable to others was established. Sugars, such as glucose or sucrose could slowly pass through the protoplasmic membranes. These hypertonic solutions cause plasmolysis due to the greater total concentration it has than that in the cell sap when the cell is in zero turgor (Noggle and Fritz, 1983).

According to Nobel (1983), the cell wall and the plasma membrane are permeable to water and certain solutes. Cells will be filled with water under pressure as long as there is a high internal concentration of solutes. This capacity of the cell is measured by its cell potential. The difference between the magnitudes of cell turgor pressure and osmotic potential is the potential of water. Net osmosis occurs if the potential of water inside the cell is not equal to that in the extracellular fluid outside: the process takes place from a high water potential section to lower water potential area at a rate proportional to their difference.

The lipid layer of the plasma membrane provides the greatest resistance to osmosis. Thus, its permeability determines the rate of osmosis into or out of the cell as a response to a change in potential of water. Ferrier and Dainty (1977) reported that the main resistance to water flow into the cell is the cell membrane rather than the apoplast; but in some cases, the resistance of the apoplast and its water capacity may contribute significantly to the water potential equilibrium time constant of the tissue. The apoplast is that area in the cell wall and intercellular spaces where water and solutes could move around.

2.2.3 Changes of Physico-chemical and Structural Properties of Plant Tissue During Osmotic Dehydration

Physical, chemical and structural changes of the plant tissue take place due to heat and mass transfer during dehydration process. Since these changes are associated with quality attributes of food (Perera, 2005) and design of equipment (Rao and Quintero, 2005) the knowledge on them seem to be necessary.

***Physical Changes*:** It is becoming increasingly difficult to ignore the physical changes of fruits and vegetables because of their relationship with quality (color and texture) attributes.

Transparency and color of fruits and vegetables may change considerably during osmotic dehydration process due to the following facts:

- Development of browning along with loss or degradation of fruit pigments (Waliszewski, Garcia *et al*., 2001);
- Increase in concentration of pigment which could elevate absorption of light;
- Enhancement of surface reflection which leads to increment of refractive index in liquid phase of product tissue;
- Gas phase exchange near the surface of tissue because of hydrodynamic mechanisms (HDM) action (Martinez-Monzo *et al*., 2001; Talens *et al*., 2002).

All in all, browning along with loss or degradation of fruit pigments seems to be the most drastic facts which influence the acceptability of product, since it expresses changes in the product hue characteristics (Chiralt and Talens, 2005). Several studies have revealed that these effects can be minimized using sugars due to its protective

role of on chlorophylls and anthocyanins and low temperatures (Torregiani and Bertolo, 2001).

Cell turgor, cell wall resistance to compression, density of cell packaging and some factors such as shape, size, strain rate and temperature are mainly influenced on the mechanical attributes of plant tissue (Chiralt *et al*., 2001; Torres *et al*., 2005).

In a published paper by Chiralt *et al*. (2001), they claimed that mechanical behaviors of plant tissues during osmotic dehydration process are affected due to cell turgor loss, cell wall resistance and middle lamella modificaion and changes in sample size and shape. Therefore, failure mode changes, reduction of stress–strain relationship and elevation of visco-elastic ratio are the most important changes in mechanical response which could be occured by osmotic tretments.

Different conditions in osmosis process can induce different mechanical behavior to a various degree which leads to affect mechanical response of product in a different way (Chiralt *et al*., 2001; Torres *et al*., 2005). Chiralt and Talens (2005) have been reported physical alteration in cells organisation like cell debonding due to sample deformation during pulsed vacuum osmtic dehydration (PVOD). They also reported that long duration of osmotic treatment resulted in side effects on textural properties of kiwifruit while the mechanical behavior of strawberry samples was totally different. Mutanda *et al*. (1998) observed different cellular changes in osmotic treatments performed with 35 and 65 ºBrix in which for samples treated with 65 ºBrix cell debonding and cell wall alteration are the most apparent alteration.

Chemical Changes: Various chemical and compositional changes due to mass transportion during osmotic dehydration have been reported. According to Chiralt and Talens (2005) and Peiro *et al*. (2006), alterations in enzyme system of cells and their activities, volatile constituents and changes in pectic fractions, and flow of minerals and vitamins as micronutrients to the solution which induced by osmotic stress are main factors affecting these changes. The required enzymes for the metabolic paths in mango slices activated during osmotic dehydration due to different alterations in the physiology of fruit slices (Tovar *et al*., 2001). It has been reported that activity of enzymes such as polygalacturonase (PG) and peroxidase (POD) increased during osmotic process of cucumber using polyethylene glycol solution. These enzymes activities lead to alteration in pectin methylation degree and mechanical properties of plant tissue (Sajnin *et al*., 2003). Torreggiani *et al*. (1998) pointed out that osmotic treatments of strawberry and kiwi tissues provoked alteration in pectic fractions which involved in textural modifications. A number of chemical alterations which are related to respiration paths changes such as volatile profile changes (Escriche *et al*., 2000; Chiralt *et al*., 2001; Talens *et al*., 2002; Talens *et al*., 2003) and the development of chemicals including ethanol and acetaldehyde (Tovar *et al*., 2001) initiated by application of osmotic treatment. Pfannhauser (1988) carried out an investigation to figure out osmotic dehydration effects on volatile profile behavior of a kiwifruit. There was an increase in some esters while a decrease in aldehydes was observed.

In another study, Torres *et al*. (2006) concluded that osmotic treatments promoted changes in the volatile profile of a mango. They observed a reduction in the 3-carene and nonanal concentration when mango samples were treated using 30 °Brix,

especially in pulsed vaccum osmotic dehydration (PVOD) processed samples while more concentrated sucrose solution (65 °Brix) promoted more volatile compounds losses. Rizzolo *et al*. (2006) pointed out that the type of osmotic agent, solution concentration and immersion time affected the profiles of strawberry□s volatile. They concluded that osmotic treatment using sorbitol caused significant decrease in octyl and ethyl acetates, ethyl and methyl butanoates, and methyl hexanoate while ethyl 2-methylpropanoate and (E)-2-hexenyl acetate reached to maximum concentration changes after 2h of immersion time. On the other hand, using sucrose as osmotic agent lead to maximum concentration change of ethyl propanoate while a rapid decrease in ethyl butanoate were observed after 2h of the treatment.

Cellular Structure Changes: It is important to investigate the microstructural changes during dehydration due to the fact that the most of the observed changes at a macroscopic level are caused by changes occurred at microstructural/cellular level. A fresh plant tissue is composed by connected cells by the middle lamella (Figure 2.4). Elastic behaviour of plant tissue is supported by turgor pressure of the cells (Aguilera *et al*., 1998). Rigidity and strength of the tissue provides by cellulose of the cell wall, whereas its plasticity supports by pectin and hemicellulose of the middle lamella (Lewicki and Pawlak, 2003). During osmotic dehydration process, water will leave the cell by osmosis. Therefore, different phenomena can be observed during dehydration such as plasmolysis (Figure 2.4b) (Mauro, Tavares and Menegalli 2002), cell debonding, or detachment of the middle lamella (Figure 2.4c) (Lewicki and Porzecka-Pawlak, 2005) and cell rupture (Figure 2.4d) (Lewicki and Pawlak, 2003).

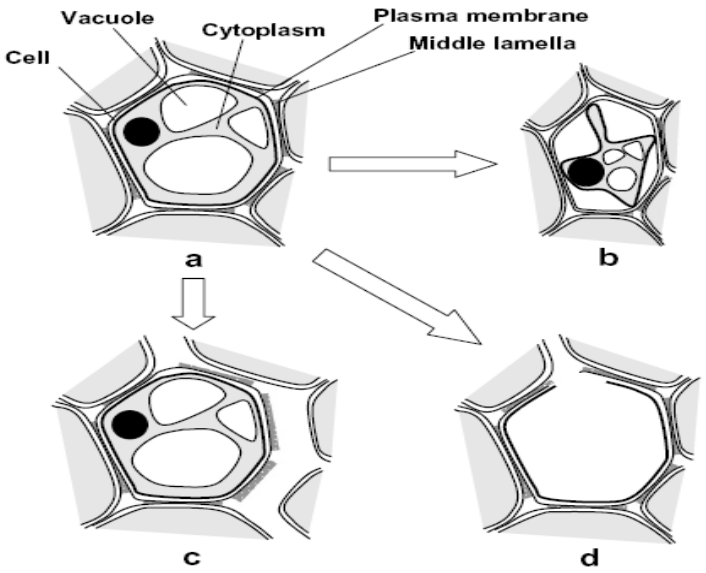


Figure 2.4. Changes of a Plant Cell at Microstructural Level during Dehydration: (a) Fresh Cell; (b) Plasmolysis; (c) Cell Debonding and (d) Cell Rupture (Mayor *et al.*, 2008)

Lewicki and Porzecka-Pawlak (2005) draws our attention to shrinkage, loss in the turgor pressure and cells deformation accompanied with plasmolysis affecting the mechanical attributes during osmotic dehydration of apple. They also mentioned that the denaturation or degradation of the middle lamella components and micro-stresses in plant tissue which induced by water removal lead to cell debonding (ruture) or the detachment of the middle lamella. Lewicki and Pawlak (2003) pointed out that cavities in different size and shape are formed due to the cell rupture which increases the porosity of the product.

Barat and Fito (2001) observed the suction of hypertonic solution into intercellular spaces of apple tissue during osmotic dehydration which cause cells shrinkage, deformation of the cell matrix and increase in size of the intercellular spaces. In the study on osmotic dehydration under atmospheric and sub-atmospheric pressure, Barat *et al*. (1998) identified different changes in both cells and intercellular spaces.

In osmotic dehydration under atmospheric pressure, intercellular spaces become larger cells tend to cylindrical shape. Additionally, irregular shape of cell shrinkage has occured without plasma membrane detachment. From another point of view, application of sub-atmospheric pressure leads to filling the empty space with hypertonic solution as a result of plasma membrane separation from the cell wall.

2.2.4 Factors Influencing Mass Transfer during Osmotic Dehydration

In the past three decades, factors which influence osmotic dehydration process were studied extensively. Raw material characteristics, type and concentration of osmotic agent, shape and geometry of sample, temperature, immersion time, solution to fruit ratio and possible enhancing method such as high pressure, blanching, use of vacuum pressure, microwave power, centrifugal force, ultrasonic waves, high electric field pulse are the main parameters influence on the mass transfer rate during the process (Torreggiani, 1995; Scalzo *et al*., 2001; Sereno *et al*., 2001; Matusek *et al*., 2008).

2.2.4.1 Raw Material Characteristics

Among different factors, characteristics of raw material have a profound rule on mass transfer during osmotic dehydration. The great variability between different fruits is mainly due to initial soluble and insoluble solid content, compactness of tissue, presence of pectin and other cellulosic components, intercellular spaces and enzyme activity (Forni *et al*., 1986; Scalzo *et al*., 2001).

Chilat and Talens (2005) pointed out that cell size, tortuosity, porosity and permeability of cell membrane determine the mass transport in the material. Yamaki and Ino (1992) conducted a survey on mature and immature apples demonstrated that plasma membrane and tonoplast are permeable to sugars. They noted that maturation leads to increase permeability to sugars in which the permeability of tonoplasis much greater than plasma membrane.

2.2.4.2 Osmotic Agent Type and Its Concentration

The selection of a particular osmotic agent and its concentration depends on several factors. The organoleptic characteristics, solubility, cost, molecular mass and lowering capacity of the compound on water activity are the most important factors (Qi *et al*., 1998; Zhao and Xie, 2004). Sugars (sucrose) and salts (sodium chloride) are the two most common solute used for osmotic dehydration, which their advantages already reported by several researchers (Lenart and Flink, 1984).

Several studies have revealed that osmotic agent type and its concentration significantly affected water loss, solids gain, weight reduction and water activity during the process (El-Aouar *et al*., 2006; Kolawole *et al*., 2007). Matusek *et al*. (2008) used fructo-oligosaccharides (FOS) and sucrose as osmotic agents for apple cubes and compared their effects on osmotic dehydration process. They reported that osmotic behavior of fructo-oligosaccharides differs from sucrose due to the higher molecular size which could decrease the rate of solute diffusion.

Glucose as osmotic agent is utilized for several fruits instead of sucrose in order to obtain higher amounts of solid gain and water loss (Salvatori and Alzamora, 2000; Taiwo, Eshtiaghi *et al*., 2003; Nieto *et al*., 2004). Torreggiani (1993) and Mandala (2005) reported that low molecular weight sugar such as glucose, due to the high rate of penetration can increase the absorption of sugar during dehydration process. Since low mass sugar used as osmotic agent the main effect of process instead of dehydration is a solid enrichment. In agreement with Torreggiani (1993), Argaiz *et al*. (1994) found that at same moisture content glucose has more intensive effect on reduction of water activity than sucrose and maltodextrines.

In summary, sucrose can be recommended as one of the best osmotic agents due to its low cost, molecular weight and size, effectiveness, convenience and desired flavor (Lenart, 1996; Taiwo *et al*., 2003), especially when the osmotic dehydration is used as pretreatment before other traditional drying process. Sucrose layer formation on the surface of dehydrated sample is a barrier to contact with oxygen (Lenart, 1996), thereby the oxidative reactions could prevented (Barbosa-Canovas and Vega-Mercado, 2000; Garcia *et al*., 2007).

2.2.4.3 Temperature

Temperature is an important variable influencing osmotic dehydration process. The effects of temperature during dehydration are: (1) diffusion rates enhancment; (2) disruption of the cellular material integrity in critical values (Thebud and Santarius, 1982; Allali *et al*., 2009). Mass transportation kinetics during osmosis process depends on temperature which higher temperatures generally lead to rapid loss of

water through swelling and plasticizing of cell membranes (Lazarides *et al*., 1995; Rastogi *et al*., 2005) and in highly concentrated solution, the rate of diffusion for both solute and water increase through decreasing viscosity of the osmotic solution (Kaymak-Ertekin and Sultanoglu, 2000; Telis *et al*., 2003).

The effect of temperature on mass transfer kinetics during osmotic dehydration of foods has been widely investigated by several authors (Islam, 1982; Lenart and Flink, 1984; Lazarides *et al*., 1995; Chenlo *et al*., 2006; El-Aouar *et al*., 2006; Panades and Fito 2006; Singh *et al*., 2007). They concluded that the kinetics of mass transfer increases with the temperature during osmotic dehydration process. El-Aouar *et al*. (2006) suggested that as uptaking of low amount of solid (<10%) is favored, it can be obtained by lowest medium temperature and contact time while the concentration is set at highest level.

Worthy of note, there is a temperature limit, perhaps 50-60°C, which the cell membrane of the plant tissue is damaged (Marcotte, 1988; Rastogi and Raghavarao, 1996). Ponting *et al*. (1966) and Videv *et al*. (1990) revealed that temperatures above 50 °C caused internal browning and loss of fruity flavor in apple slices.

2.2.4.4 Solution to Sample Ratio

On a lab and industrial scale, the low solution to sample ratio is preferred to limit plant size and the costs of solution regeneration (Marouze *et al*., 2001). Numerous studies are performed using a high solution to sample ratio in the range of 10:1-30:1 to keep solution composition variation at the lowest level making easier

interpretation and modeling of the process. Several studies have claimed that rate of mass transfer during the process is directly related to the proportion of sample to solution (Rastogi, 1995; Pokharkar, 1998; Sunjka and Raghavan, 2004; İspir and Toğrul, 2009). As expected, higher ratio offered a higher solid gain and water loss.

2.2.4.5 Immersion Time

Immersion time seems to affect the mass transfer rate and behavior of membranes during osmotic dehydration procces. It was reported that the most important changes in solids and water in treated samples takes place during the first three hours of the process (Alves *et al*., 2005; Mayor *et al*., 2006).

In the beginning of the process, the large osmotic driving force between sample and solution leads to the high rate of mass transfer (Raoult-Walk, 1992; Lazarides *et al*., 1995). From another point of view, accumulation of solids on the product surface due to rapid mass transfer in the beginning of the process block water removal and solid absorption (Singh *et al*., 2007). This finding supports earlier results of Hawkes and Flink (1978) and Lenart and Flink (1984) for osmotically dehydrated apple and potato. Baroni and Hubinger (1997) concluded that solutes are free to penetrate into all parts of the cell as time passes due to the fact that the cell membranes loss their effective barrier during the process.

2.3 Applications and Constraints of Osmotic Dehydration

A large and growing body of literature has investigated the various application of the osmotic technique in food processing (Andreotti *et al*., 1985; Crivelli *et al*., 1989; Moutounet *et al*., 1991; Alvarez *et al*., 1995; Alzamora *et al*., 1997; Sormani *et al*., 1999; Maestrell *et al*., 2001; Mandala *et al*., 2005; Dermesonlouoglou *et al*., 2008; Moreira *et al*., 2010; Ramallo and Mascheroni, 2010). Osmotic dehydration gains more popularity to produce dehydrated fruits, vegetables and candy due to expensive cost of energy globally.

Despite its application, it is necessary to review the advantages of this process. Advantages of osmotic dehydration are as follows:

- Removal of water efficiently from food products without a phase change (Uddin *et al*., 2004);
- Reduction in amount of required energy for further drying processes (Lazarides and Mavroudis, 1995);
- To Overcome some of side effects of heat on semi-permeable characteristics of cell membranes and quality (color and texture) of food products (Torreggiani and Bertolo, 2001);
- Restraining of enzymatic reactions (Krokida *et al*., 2001).

Certain draw backs and difficulties of osmotic dehydration which need to be studied in order to improve its efficiency are listed below.

- The higher solution viscosity, the higher resistance to mass transfer;
- Limitation in high level of agiatation for highly concentrated solution due to labile nature of fruits and vegetables;

- Product floating due to high density difference between samples and solution requiring an additional mechanical means to keep it immersed in the solution;
- High cost of osmotic solution especially for the industrial adoption of the process;
- The concentration/recycling process of spent osmotic solution by evaporation and /or addition of solute which still is in the form of patents;
- Fortifcation of the diluted solution with flavor and color of food to minimize product quality degradiation due to recycling of the osmotic solution;
- Leaching of soluble solids and extensive solids gain which lead to modification of original composition are other major draw backs of osmotic dehydration process (Lazarides and Mavroudis, 1995) due to its negative effect on nutritional and organoleptical properties (Dixon and Jen, 1977);
- The high amount of solute gain causes additional resistance to water transfer during complementary drying process (Wang and Sastry, 2000);
- The osmotic treatment is time-consuming (Rastogi *et al*., 2000).

2.4 Modeling Mass Transfer during Osmotic Dehydration Process

The importance of the process modelling was found on the need to the correct design of the operation, reduce the experimental work and control of dehydrated food composition. This assumption that mass transfer can be described by Fick's second law is the basis of the most developed models for the osmotic dehydration process (Hawkes and Flink, 1978; Magee *et al*., 1983; Beristain *et al*., 1990; Rahman and Lamb, 1991; Biswal and Bozorgmehr, 1992; Lazarides and Maroudi, 1996). The Eq.

(1.1) presents the Fick's second law for diffusion at unidirectional unsteady state and negligible interactions of the other components on the diffusion of the solute.

$$\frac{\partial C_i}{\partial \theta} = \frac{\partial}{\partial z} D_i \frac{\partial^2 C_i}{\partial z^2} \qquad (1.1)$$

Coefficients of effective diffusion for solid uptake and water loss individually or simultaneously could be estimated using the Eq (1.1). Crank (1975) presented the calculation of diffusion coefficients for different geometry of food materials which is based on constant concentration of the external solution or fixed volume of solution. Furthermore, the diffusion coefficient must be constant and, the surface resistance compared with internal resistance of the solid to diffusion is assumed to be negligible.

2.4.1 Infinite Flat Plate

The following assumptions and boundary conditions are related to infinite plate geometry,

$$C = C_0 \; at \; t = 0 \quad -1 < x < +1$$

$$and \; C = C_1 \; at \; t > 0 \; x = 1$$

The following equations are the solutions of Eq. (1.1) for diffused moisture (M_r) and solid ratio (S_r) (Crank, 1975):

For unlimited volume of hypertonic solution which well-agitated

$$M_r = \frac{(m_t - m_\infty)}{(m_0 - m_\infty)} = \frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{(2n+1)2}exp\left[-\left[n+\frac{1}{2}\right]2\pi^2 F_{ow}\right] \quad (1.2)$$

and

$$S_r = \frac{(s_t - s_\infty)}{(s_0 - s_\infty)} = \frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{(2n+1)2}exp\left[-\left[n+\frac{1}{2}\right]2\pi^2 F_{os}\right] \quad (1.3)$$

For limited volume of hypertonic solution which well-agitated

$$M_r = \frac{(m_t - m_\infty)}{(m_0 - m_\infty)} = \sum_{n=1}^{\infty}\frac{2\alpha(1+\alpha)}{1+\alpha+\alpha^2 q_n^2}\, exp[-q_n^2.F_{ow}] \quad (1.4)$$

$$S_r = \frac{(s_t - s_\infty)}{(s_0 - s_\infty)} = \sum_{n=1}^{\infty}\frac{2\alpha(1+\alpha)}{1+\alpha+\alpha^2 q_n^2}\, exp[-q_n^2.F_{os}] \quad (1.5)$$

For small values of t

$$M_r = \frac{(m_t - m_\infty)}{(m_0 - m_\infty)} = 1 - 4\left(\frac{D_e\, t}{\pi\, l^2}\right)^{1/2} \quad (1.6)$$

This condition is valid over a long immersion time (Azuara *et al*., 1992; Rastogi and Raghavarao, 1994).

2.4.2 Rectangular Parallelepiped

The following equations are obtained by solution of Eq. (1.1) for the transfer of water and solute in rectangular parallelepiped, respectively (Crank, 1975):

$$M_r = \frac{(m_t - m_\infty)}{(m_0 - m_\infty)} = \sum_{n=1}^{\infty} C_n exp\left[-D_{ew} t q_n \left[\frac{1}{a^2} + \frac{1}{b^2} + \frac{1}{c^2}\right]\right] \quad (1.7)$$

and

$$S_r = \frac{(s_t - s_\infty)}{(s_0 - s_\infty)} = \sum_{n=1}^{\infty} C_n exp\left[-D_{es} t q_n \left[\frac{1}{a^2} + \frac{1}{b^2} + \frac{1}{c^2}\right]\right] \quad (1.8)$$

If Fourier number is > 0.1 only the first term in Eq. (1.7) and Eq. (1.8) is significant and other terms can be omitted Therefore, Eq. (1.7) and Eq. (1.8) reduce to Eq. (1.9) and Eq. (1.10), respectively (Rastogi and Niranjan, 1998):

$$-ln\left[\frac{M_r}{C_1}\right] = q_1 \, D_{ew} t \left[\frac{1}{a^2} + \frac{1}{b^2} + \frac{1}{c^2}\right] \quad (1.9)$$

and

$$-ln\left[\frac{S_r}{C_1}\right] = q_1 \, D_{es} t \left[\frac{1}{a^2} + \frac{1}{b^2} + \frac{1}{c^2}\right] \quad (1.10)$$

2.4.3 Infinite Cylinder

$$\frac{M_t}{M_\infty} = 1 - \sum_{n-1}^{\infty} \frac{4}{(a\alpha_n)2} exp[-F_0(a\alpha_n)^2] \quad (1.11)$$

From Eq. (1.11) the Fourier number can be calculated for different values of diffused moisture ratio. Plotting Fourier number against immersion time yielded the slope which will be the effective diffusion coefficient (D_e) (Rastogi *et al*., 1997).

2.4.4 Finite Cylinder

Solution of Eq. (1.1) for diffusion from a finite cylinder rendered the following equations (Crank, 1975; Rastogi *et al*., 1999):

$$M_r = \frac{(m_t - m_\infty)}{(m_0 - m_\infty)} = \sum_{n=1}^{\infty} C_{pn} C_{cn} exp\left[-D_{ew} t\left[\frac{q_{pn}^2}{l^2} + \frac{q_{cn}^2}{r^2}\right]\right] = \sum_{n=1}^{\infty} C_{pn}\, C_{cn}\, exp\left[-D_{ew} t \frac{q_{cn}^2}{A^2}\right] \quad (1.12)$$

and

$$M_r = \frac{(s_t - s_\infty)}{(s_0 - s_\infty)} = \sum_{n=1}^{\infty} C_{pn} C_{cn} exp\left[-D_{es} t\left[\frac{q_{pn}^2}{l^2} + \frac{q_{cn}^2}{r^2}\right]\right] = \sum_{n=1}^{\infty} C_{pn}\, C_{cn}\, exp\left[-D_{es} t \frac{q_{cn}^2}{A^2}\right] \quad (1.13)$$

In Eq. (1.12) and Eq. (1.13) parameter A is defined as

$$\frac{1}{A^2} = \frac{1}{r^2}\left[1 + \left(\frac{r}{l}\right)^2 \left(\frac{q_{pn}}{q_{cn}}\right)^2\right] \quad (1.14)$$

For an infinite cylinder l >> r and the above equation will be A= r (Rastogi *et al*., 1999).

If the Fourier number > 0.1, Eq. (1.12) and Eq. (1.13) reduce to the first term (McCabe, Smith and Harriot 1993):

$$-ln\left[\frac{M_r}{C_{p1}C_{c1}}\right] = \left[D_{ew}t\left[\frac{q_{p1}^2}{l^2} + \frac{q_{c1}^2}{r^2}\right]\right] \quad (1.15)$$

and

$$-ln\left[\frac{S_r}{C_{p1}C_{c1}}\right] = \left[D_{es}t\left[\frac{q_{p1}^2}{l^2} + \frac{q_{c1}^2}{r^2}\right]\right] \quad (1.16)$$

So far, fickian unsteady state diffusion models presented above have been deemed to be the most suitable models to predict coefficients of diffusion during osmotic treatment. However, the shape of material is the most important factor which influence on selection of the suitable model to predict coefficients of water and solids diffusion (Rastogi *et al*., 2002).

Nevertheless, most biological materials are porous or have interconnected voids, which affect diffusion. For diffusion in porous solids, the effective diffusivity is lower than the diffusion coefficient in non-porous solids. Thus, diffusivity of a species in solution is related to the effective diffusivity, D_{eff} in the porous solid using a correction factor, which contains the open void fraction and the tortuosity as given in (Geankoplis, 1993).

$$D_{eff} = \frac{\varepsilon}{\tau} D \quad (1.17)$$

From practical point of view, in food industry, approaches based on Fick's second law are not very useful because of their complexity and unrealistic assumptions (i.e. mass transfer resistance in the solution could not be negligible for high viscosity

hypertonic solution). Thus, several simpler semi-empirical approaches which concerning parameters with physical meaning are advised to model the kinetics of mass transfers during osmotic dehydration. Therefore, several simpler semi-empirical equations focusing on factors with physical meaning are suggested which allow the modelling of mass transfers kinetics during osmosis process (Hawkes and Flink, 1978; Peleg, 1988).

Hawkes and Flink (1978) used a simplified form of the equation of unsteady state Fickian diffusion to model drying kinetics of apple slices in sucrose solutions with different concentrations. The obtained slope of the curve which was generated using plotting the normalized solids content of the apples slices against the square root of time defines as the coefficient of mass transfer. This procedure is applicable to estimate the coefficient of diffusion in semi-infinite medium if unsteady state Fickian diffusion is assumed.

An empirical approach was suggested by Peleg (1988) for modelling water absorption characteristics of foodstuffs. Several researchers used Peleg equation to explain sorption processes in different foods (Maharaj and Sankat, 2000; Sanjuan *et al*., 2001; Seyhan-Gurtas *et al*., 2001). A number of studies have been investigated on water desorption and solid absorption simultaneously for osmotically dehydrated papaya, sardine sheets, apricot, pumpkin and guava using Peleg model (Palou *et al*., 1994; Corzo and Bracho, 2006; Khoyi and Hesari, 2007; Kowalska *et al*., 2008; Corrêa *et al*., 2010).

Biswal *et al*. (1991) used a rate parameter to model osmotic dehydration of green bean as a function of concentration and temperature of osmotic solution. The parameter was calculated from the slope of the straight line obtained from green bean water loss and solids gain vs. square root of time. A similar empirical model was used by Shi *et al*. (1995) for studying the effect of vacuum pressure on osmotic dehydration rate of foods; and by Salvatori and Alzamora (2000) for process variables effects on apple slices.

A model was developed by Hough *et al*. (1993) to predict the kinetics of mass transfer in terms of solute gain and water loss for apple slices in different concentrations of sucrose solution. The model, however, was modified to account for tissue shrinkage during dehydration. The model adequately predicted the percentage of solute gain, water loss, lowering in water activity and shrinkage during the process.

More complex models involving irreversible process thermodynamics have also been developed to integrate the contribution of each component i.e. osmotic solution and water to the osmotic dehydration process, as well as to account for the natural tissue action e.g. shrinkage that occurs during osmotic dehydration (Toupin, 1986; Marcotte, 1988). A model was developed by Toupin (1986) based on irreversible process thermodynamics for predicting mass transfer in plant materials which the diffusion of the impermeable and permeable species in the tissue, and tissue shrinkage was taken into account. The model included terms to describe the cell and tissue properties e.g. permeability of the cell wall and the cell volume, void fraction, fraction of the area of the plasmalemma occupied by the plasmodesmata, critical cell

volume. In the model, many of the parameters were estimated and adjusted because of lack of data. The model was also used to measure cell volume changes during osmotic dehydration.

Marcotte (1988) developed a microscopic model for osmotically dehydration of potato in different concentrations of sucrose solution. The bulk diffusion within the extracellular space was described using relations associated with the extended form of Fick's second law. The trans-membrane and the symplastic transport mechanisms, which were considered to be the major transport mechanisms with respect to osmotic dehydration, were modelled based on the theory of irreversible thermodynamic. As with Toupin (1986), the model included terms for the diameter of the cell volume, total diameter of the cell, tortuosity, and area of exchange of the plasmalemma, among others. The model was used to predict total cell volumes, cell volume and extracellular volume of the potato. A comparison of the calculated cell volume and the experimental volumes showed that the model was able to describe the mass transport phenomena of potato tissue undergoing osmotic dehydration in sucrose solution.

Azuara *et al*. (1992) suggested a model based on mass balance to overcome the limitations of Fick's model and prediction of the kinetics of solid gain and water loss during the process. The model was developed to have the flexibility to facilitate its application to different geometric shapes. It was tested using published data on apple (Azuara *et al*., 1998; Kaymak-Ertekin and Sultanoglu, 2000), carrot (Amami *et al*., 2007; Singh *et al*., 2007), cuts of chicken breast (Schmidt *et al*., 2009) and apricot (İspir and Toğrul, 2009) and, was used to predict the values of water loss and solid

gain beyond the range actually studied. Correlation coefficients obtained for all cases studied were more than 0.95. Then the model was rewritten according to one-dimensional diffusion through a thin slab in order to calculate the apparent diffusion coefficients for each condition (Azuara *et al*., 1992):

$$w = w_e - w_m \quad (1.18)$$

The relationship between w and w_m can be written as Eq. (1.19), since water loss depends on time at constant concentration and temperature of osmotic solution.

$$w_m = \frac{w}{k} = \frac{w}{k_w \times t} \quad (1.19)$$

By assuming the linear (zero-order) relation between K, K_w and t, Eq. (1.20) and Eq. (1.21) can obtained from Eq. (1.18) and Eq. (1.19), respectively.

$$\frac{t}{w} = \frac{1}{k_w \times w_e} + \frac{t}{w_e} \quad (1.20)$$

$$\frac{t}{s} = \frac{1}{K_s \times S_e} + \frac{t}{S_e} \quad (1.21)$$

Water loss and solute gain were calculated according to the following equations:

$$w = \frac{(m_t \times x_w) - (m_0 \times x_{w0})}{m_0} \quad (1.22)$$

$$s = \frac{(m_t\, x_{sst}) - (m_0\, x_{ss0})}{m_0} \quad (1.23)$$

Raoult-Wack *et al*. (1994) suggested bi-compartment model (bi-compartmental model) using agar gel cubes to describe sucrose layer formation on the surface of material (Figure 2.5). The model was representative of the model food cube with two concentric cubic compartments. The water and solutes transfer was then considered to occur between the inner and outer compartment of the model food, as well as between the outer compartment of the model food and the osmotic solution. The authors reported that information about the inner concentration in each compartment would be useful in optimization of osmotic dehydration and the subsequent drying processes. Furthermore, by considering the internal movement of water and solid within the material, the model would accurately predict the influences of any changes in the external conditions (temperature, concentration of osmotic solution) that occurred during osmotic dehydration.

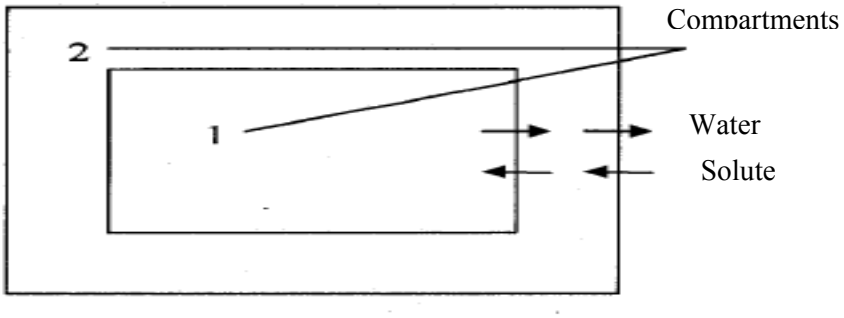


Figure 2.5. Schematic Diagram of Water and Solute Transfer in the Compartment Model (Raoult-Wack *et al*., 1994)

Since the studies on modelling of mass transfer have been focused on simplified semi-empirical models, the mechanisms of water and solids migration are not totally understood (Yao and Le Maguer, 1994).

It is necessary to investigate the equilibrium state for a clearer insight about mechanisms of mass transportation involved in this process (Barat *et al*., 1998). Based on the end-point criterion knowledge, more theoretical models allowing calculation of the process parameters were developed (Lenart and Flink, 1984). Osmotic dehydration kinetics at equilibrium state for apricot (Toğrul and İspir, 2008), potato (Biswal and Bozorgmehr, 1991), fish tilapia (Medina-Vivanco *et al*., 1998), apple (Mosalve-Gonzalez *et al*., 1993; Panagiotou *et al*., 1998), banana and kiwi fruit (Panagiotou *et al*., 1998) have been investigated.

Zugarramurdi and Lupin (1980) proposed a mathematical model, with an exponential approach to the equilibrium value to describe behavior of fish salting process.This model was used by Escriche *et al*. (1998), Corzo and Bracho (2005) to investigate on the osmotic dehydration under vacuum pressure of codfish and sardine sheets.

$$\frac{dX_{\mathrm{i}}}{dt} = ki(X_i^* - X_{\mathrm{i}}) \qquad (1.24)$$

Where

$$X_{\mathrm{i}} = \frac{\text{Mass of } i \text{ at a given time}}{m_t - m_{\mathrm{w}} - m_{\mathrm{s}}} \qquad (1.25)$$

$$X_i^* = \frac{\text{Mass of } i \text{ at equilibrium}}{m_t - m_{\mathrm{w}} - m_{\mathrm{s}}} \qquad (1.26)$$

K_i= Specific rate constant for the variation of *i* (1.27)

Eq. (1.24) can be easily integrated with the following initial condition (t = 0):

$$X_{\mathrm{i}}(0) = X_i^0 \qquad (1.28)$$

$$Xi = X_i^0 \left(e^{-k_i^t}\right) + X_i^* \left(1 - e^{-k_i^t}\right) \qquad (1.29)$$

The Eq. (1.30) shows the releation between the value at time ($t + \Delta t$) and the value at time (t):

$$X_i\,(t + \Delta t) = X_i^*\,(1 - e^{-k_i \Delta t}) + (e^{-k_i \Delta t}) X_i(t) \qquad (1.30)$$

The equilibrium water content (X_{we}) and solid content (X_{se}) expressed as total mass basis were estimated by the following equations:

$$X_{we} = \frac{X_w^0}{1 + X_w^0 + X_s^0} \qquad (1.31)$$

$$X_{se} = \frac{X_s^0}{1 + X_w^0 + X_s^0} \qquad (1.32)$$

In 1996, Parjoko *et al*. suggested different equations to predict the equilibrium water (X_{we}) and solid (X_{sa}) contents:

$$X_{we} = \frac{X_{w0} - w_e}{1 - w_e + s_e} \quad (1.33)$$

$$X_{se} = \frac{X_{ss0} + s_e}{1 - w_e + s_e} \quad (1.34)$$

From a practical point of view, other approaches which are useful to describe mass transfer at cellular level are more complex due to considering diffusion through

membranes of different permeability and transport in intercellular spaces (Chausi *et al*., 2001; Gekas *et al*., 2002).

In summary, Fick's second law gives a high correlation between the experimental and estimated values of kinetics terms of mass transfer (Conway, 1983; Beristain *et al*., 1990). Fick's second law and the analytical solutions given in Crank (1975) have found general acceptance and wide usage in modeling the kinetics of mass transfer during osmotic treatment of different foods with different shapes (Hawkes and Flink, 1978; Conway, 1983; Beristain *et al*., 1990; Biswal and Bozorgmehr, 1991; Yang and Le Maguer, 1992; Mauro, 1995; Lazarides and Maroudis, 1996). This is in spite of the limitations of the applications of the law to osmotic dehydration which include (Raoult-Wack *et al*., 1994):

- The simultaneous mass transfer of moisture out of the sample and solids into the sample reduces to the transfer of either moisture or solids. The probable interaction between the counter-current flow of moisture and solids is therefore not accounted for.
- The possible action of natural cell membranes in the dehydration process is not accommodated.

2.5 Method of Mass Transfer Enhancement

Osmotic dehydration is a low efficient and time-consuming process due to the fact that osmotic pressure is a sole driving force for mass transfer as well as offered dominant resistance by semi-permeable cell membranes to the process. In recent years, various thermal and nonthermal techniques have been widely investigated to

increase the rate of mass transfer. These techniques include heat treatment (blanching) (Del Valle *et al*., 1998; Moreno *et al*., 2000; Escobar *et al*., 2007; Kowalska *et al*., 2008), high intensity electrical field pulses (Bazhal and Vorobiev, 2000; Ade-Omowaye *et al*., 2001; Bouzrara and Vorobiev, 2003; Lebovka *et al*., 2004; Amami *et al*., 2007), ultra high hydrostatic pressure (Eshtiaghi *et al*., 1994; Rastogi *et al*., 2000; Tedjo *et al*., 2002) and ultrasound (Rodrigues and Fernandes, 2007) prior to osmotic dehydration or applying partial vacuum (Rastogi *et al*., 2002; Deng and Zhao, 2008; Moraga *et al*., 2009), centrifugal force (Azuara *et al*., 1996; Amami *et al*., 2007) and microwave power during osmotic treatment (Li and Ramaswamy, 2006; Azarpazhooh and Ramaswamy, 2010).

2.5.1 Application of Blanching Prior to Osmotic Dehydration

Traditionally, blanching carried out by a short time immersion of fruit or vegetable in hot water or steam (Kidmose, 1999; Severini *et al*., 2005; Gowen *et al*., 2007). The heat treatment of blanching before further process such as freezing, canning or dehydration is a necessary step in order to inactivate enzymes responsible for quality changes that occur during distribution and storage. Blanching has some additional advantages like destroying microorganisms, elimination of off-flavor and stabilization of color while thermal treatment can also have negative effects on sensorial (excessive loss of texture and unwanted changes of color) and nutritional quality attributes (Cruz *et al*., 2007).

In fruit and vegetables, the cell membranes and the cell walls are barriers to mass transportation. Therefore, treatments affecting both cell wall structure and membrane

permeability prior to the osmotic treatment should be understood (Mavroudis *et al*., 2004; Escobar *et al*., 2007).

Several studies have found that mass transfer rates increase by application of blanching treatment before osmotic dehydration. They noted steam and microwave blanching before osmotic dehydration lead to increase solid gain because of cell decompartmentation (Alzamora *et al*., 1997; Moreno *et al*., 2000). In 2007, Escobar *et al*. evaluated the effect of blanching on the rates of mass transfer for osmotically dehydrated carrots. They concluded that the blanching pretreatment causes cells death which leads to increase the coefficients of effective diffusion for sucrose as well as water. In agreement with Escobar *et al*. (2007), Matusek *et al*. (2007) reported that blanching process makes the semi-permeable membrane more permeable.

2.5.2 Application of Vacuum Pressure during Osmotic Dehydration

Several studies revealed the higher mass transfer rate under vacuum pressure compared with atmospheric pressure (Fito *et al*., 1992; Fito and Pastor, 1993; Fito and Chiralt, 2000; Paes *et al*., 2007; Deng and Zhao, 2008; Corrêa *et al*., 2010). Fito (1994) pointed out that capillary phenomena occur at low pressure through the hydrodynamic mechanism (HDM).

Shi *et al*. (1995) conducted study to determine the effect of vacuum pressure on mass transfer rate of apricots, strawberries and pineapples concluded that vacuum treatments increased the rate of water transfer without any significant effect on solid

gain. Beside that they mentioned, high porous fruits more affected by vacuum treatment in terms of water losses. Chirlat *et al*. (1999) and Betoret (2002) suggested the vacuum impregnation compared with the process under atmospheric pressure as an operation which increases process yield due to reduction in mass loss and allow us to improves the organoleptical properties and enrich products with minerals, nutrients and vitamins.

In recent years, a new method has been suggested to perform osmotic dehydration at sub-atmospheric pressure. Pulsed vacuum osmotic dehydration (PVOD) comprises applying vacuum treatment for a short period of time at the beginning of process and performing rest of the operation at atmospheric pressure which has advantageous effects on the kinetics of mass transfer and quality attributes of treated materials and also lead to reduce the costs of energy (Fito and Chiralt, 2000; Moreno, 2004; Corrêa *et al*., 2010).

2.5.3 Application of Ultrasonic Waves during Osmotic Dehydration

Application of high power ultrasound in the food industry is still a novel technology. Ultrasonic waves can cause a rapid series of alternative compressions and rarefactions, in kind of a sponge when it is squeezed and released repeatedly (Stojanovic and Silva, 2006). Ultrasonic wave deform porous solid materials, such as fruits which lead to microscopic channels creation and furthure resulted in diffusion boundary layer reduction and increase the mass transfer rate in the fruit (Tarleton and Wakeman, 1998; Fuente-Blanco *et al*., 2006; Fernandes *et al*., 2008). Also, acoustic streaming which caused by the oscillatory motion of an ultrasound wave

would result in enhancing mass transfer (Fernandes *et al*., 2008). Ultrasonic waves produce cavitation consists of the bubbles formation in the liquid medium, which can explosively collapse and generate localized pressure fluctuations. This phenomenon is useful to eliminate strongly attached moisture due to increased diffusion rate during osmotic treatments and accelerates gas removal from the cell matrix (Florous and Liang, 1994). It has been reported that rates of ultrasonic action depending on their frequency (Raoult-Wack *et al*., 1994).

Numerous studies have revealed that different effects of high power ultrasound can increase the rate of mass transfer of apples (Simal, 1998; Carcel *et al*., 2007), blueberries (Stojanovic and Silva, 2006), melon and pineapple (Fernandes *et al*., 2008; Fernandes *et al*., 2008) and peppers (Gabaldon-Leyva *et al*., 2007). Deng and Zhao (2008) reported that combination of osmotic treatment with ultrasound is preferable to remove water and retaining color of apples. However, this combination caused more solid gain, lower rehydration capability and serious structure collapse. Lower moisture content, less penetration of calcium ions and rehydration rate could be obtained using ultrasounic technique compared with pulsed vacuum treatment.

Fernandes *et al*. (2008) observed significant changes on melon cell matrix during osmotic dehydration including a gradual disruption of cells together with loss of shape of cell walls (after 30 min) and a significant breakdown of the tissue (from 1h onwards). They concluded that ultrasound waves alter melon cell in different way from osmotic dehydration in which no cell breakdown was observed. Additionally, formation of microscopic channels in the cell matrix is responsible for acceleration of water diffusivity. Fernandes *et al*. (2008) concluded that ultrasound and osmotic

dehydration lead to cell structure changes in pineapple. Improvment of water diffusivity and consequently shorter drying time achieved as a result of microscopic channels formation in the cell matrix.

2.5.4 Application of High Hydrostatic Pressure (HHP)

Rastogi and Niranjan (1998) and Tedjo *et al*. (2002) demonstrated the potential of HHP to increase solid gain and water loss due to damage of cell wall during HHP treatment. During HHP processing the following changes can take place simultaneously:

(1) Disruption of cell membrane and wall (Michel and Autio, 2001; Van Buggenhout *et al*., 2005);

(2) Enzyme catalyzed conversion processes (Verlent *et al*., 2004; Verlent *et al*., 2005);

(3) Chemical reactions (Nguyen *et al*., 2006; Oey *et al*., 2006);

(4) Inactivation, denaturation and formation of enzyme, protein and gel, respectively (Ludikhuyze *et al*., 2003; Van der Plancken *et al*., 2005).

In many cases, application of HHP treatment leads to more intensive green color of vegetables as a result of cell disruption and chlorophyll leakage into the intercellular space during HHP processing (Krebbers *et al*., 2002). However, it has been demonstrated that simultaneous activity of pectin methyl esterase (PME) and polygalacturonase (PG) during HHP treatment leads to lose of plant tissue firmness (Basak and Ramaswamy, 1998).

2.5.5 Application of Supercritical Fluid Prior to Osmotic Dehydration

Recently, there has been a great number of studies on supercritical carbon dioxide (SC-CO_2) fluid for the processing of biological materials such as separation technique, microorganisms inactivation and reduction of enzyme activity (Tedjo *et al*., 2000). Lack of information exists on application of SC-CO_2 as a pretreatment prior to osmotic dehydration in order to increase the rate of mass transfer. The investigation of Tedjo *et al*. (2002) on mango is the sole study in this topic. The result of mentioned study showed that solid gain increased during osmotic dehydration while water loss decreased due to diffusion of gas into the sample. Rastogi and Ranghavarao (1994) indicated that solid gain may not necessarily be a function of permeabilized cells alone but may also depend on the type of chemical and structure changes caused by the pretreatment.

2.5.6 Application of Pulsed Electric Field (PEF)

Several studies have revealed that application of PEF intensify juice extraction (Bouzrara and Vorobiev 2003; Sensory and Sastry 2004), diffusion (El-belghiti and Vorobiev, 2004; Fincan *et al*., 2004), osmotic dehydration (Ade-Omowaye *et al*., 2003; Amami *et al*., 2006; Amami *et al*., 2007) and drying (Ade-Omowaye *et al*., 2001; Lebovka *et al*., 2004).

The use of PEF consists of an external electric field application which causes a critical electrical potential throughout the cell membrane. The interest in application of PEF technique is due to the nonthermal electropermeabilisation of cell membranes

which enhances the mass transfer phenomena (Lebovka *et al*., 2001; Elez-Martinez *et al*., 2005; Amami *et al*., 2007). Knorr and Angersbach (1998) reported that field strength, impulse energy and pulse number influence the efficiency of PEF treatment. Ade-Omowaye *et al*. (2003) concluded that high number of pulse used in PEF was not significantly affected the rate of mass transfer however, application of 20 pulses at a given field strength appears adequate for optimal mass transfer.

Tedjo *et al*. (2002) pointed out that using PEF enchanced water loss during osmotic treatment while reducing solid uptake which caused the smallest changes in the taste of final product. They also reported that color of PEF treated mango sample is acceptable as well as fresh fruit.

2.5.7 Application of Centrifugal Force during Osmotic Dehydration

The effect of centrifugal force combined with osmotic dehydration for potato, apple and carrot was studied by Azuara *et al*. (1996) and Amami, *et al*. (2007). It was concluded that water loss increased using centrifuge force which in contrast considerably decelerated the solid gain. Unfortunately, the mentioned researches are the sole studies in the field of centrifugal osmotic dehydration. It would be interesting to assess the effects of rotational speed, temperature, concentration and type of osmotic solution, as well as shape of samples.

CHAPTER III

OSMOTIC DEHYDRATION OF SEEDLESS GUAVA (*Psidium Guajava* L): PROCESS MODELLING AND OPTIMIZATION

3.1 Introduction

Application of preservation methods for guava fruit which is highly perishable is necessary to improve its shelf-life. Osmotic dehydration, due to its potential to keep sensorial and nutritional properties similar to the fresh fruits, and enrich products with some compounds like functional foods seems to be a promising alternative method for food preservation (Prothon *et al*., 2001; García-Martínez *et al*., 2002). This technique enables removal of water partially based on immersing cells content into a hypertonic solution. During the process, two major simultaneous counter current types of mass transfer take place: diffusion of water from the cell matrix to hypertonic solution followed by gain of solutes from the hypertonic medium into the cell pores (Rastogi and Raghavarao, 1997; Madamba, 2003). Neglectable amount of natural solutes such as sugars, salts, organic acids and minerals leach into the hypertonic solution due to the fact that cellular membrane is not perfectly selective (Sablani *et al*., 2004). The mass transfer rate depends on parameters like temperature, immersion time, solution concentration, geometry and size, ratio of solution to sample and type of pretreatment applied (Rastogi *et al*., 2002; Tortoe, Orchard and Beezer, 2007; Panades *et al*., 2008). Worthy of note, these parameters can only be acted upon over a limited range which outside of this range may have negative effect on quality attributes. However, it might increase the rate of mass transfer (Eren and Kaymak-Ertekin, 2007). In 1973, Ponting pointed out that the

taste and composition of the dehydrated product can be modified by solid gain in osmotic treatment. According to Lenart and Grodecka 1989, blocking surface layers of cell tissue by solid gain causes an additional resistance to mass transfer resulting in lower rates of dehydration. Therefore, it is interesting to identify the optimum osmotic treatment conditions to obtain the highest water loss and the lowest solid gain. Response surface methodology is used in industrial investigations due to its potiential to study the effect of multiple independent variables on one or more dependent parameters (Corzo and Gomez, 2004). Based on our knowledge there is no available report on optimisation of osmotic dehydration process of seedless guava.

On the other hand, study on the osmotic dehydration kinetics seems to be necessary to design efficient operation and control the dehydrated material composition adequately. For modelling the mass transfer phenomena during the osmosis process, different approaches based on Fick's second law have been reported (Lenart and Flink, 1984; Chausi *et al*., 2001). Those approches are impractical due to their complexity and unrealistic assumptions. Thus, several simpler semi-empirical approaches which concerning parameters with physical meaning are advised to model the kinetics of mass transfers during osmotic dehydration (Hawkes and Flink, 1978; Beristain *et al*., 1992). Several studies investigating feasibility of Peleg equation have been performed on osmotic dehydration of different fruits and vegetables (Palou *et al*., 1994; Khin *et al*., 2006; Azoubel and Murr, 2004; Khoyi and Hesari, 2007; Atarés *et al*., 2008). Up-to-date little information is available in literature on osmotic dehydration of guava (Panades *et al*., 2006; Panades *et al*., 2008; Corrêa *et al*., 2010). Besides that, a lack of information exists about feasibility

of Peleg model for osmotic dehydration of seedless guava. In order to provide a better understanding and have a clearer insight, the kinetics of mass transfer during osmotic dehydration of seedless guava in different sucrose concentrations and temperatures was studied by using Peleg model. Therefore, the main goals of the current chapter were to investigate effects of solution concentration, temperature and immersion time on the kinetics of mass transfer during osmotic dehydration of seedless guava and optimize the osmotic conditions through response surface methodology. This was performed by: (1) studying the kinetics of mass transfer under different experimental conditions (2) examining the predictive capacity of Peleg equation to the experimental data (3) quantify the equilibrium content of the kinetics parameters at different concentrations and temperatures (4) identify of optimum processing conditions for a high weight reduction and water loss at minimal solid gain.

3.2 Materials and Methods

3.2.1 Sample Preparation

Fresh seedless guavas (*Psidium guajava* L.) were obtained from a local market (Serdang, Malaysia) daily. Fruits were chosen at commercial maturity according to their similarity of color, size, absence of surface defects and ripening grade (around 8 ºBrix). Fruits were washed, peeled and cut into 20±2 mm cubes manually using very sharp stainless steel knife, and gently blotted with tissue paper to eliminate the excess of surface humidity before each experiment. Care was exercised to select only cubes that have the same size to minimize the effect of sample size on the

experimental data. The dimensions of fruit cubes were measured by Mitutoyo digital caliper (±0.02 mm) (Mitutoyo, Waterbury, CT, USA).

3.2.2 Osmotic Dehydration Procedure

Osmotic solution was prepared by mixing commercial grade sucrose with required amount of distilled water. The solution concentrations were 30, 40 and 50% w/w. The solution concentration throughout each experiment was monitored by refractometer (Atago-Master-20 M, Japan). In higher concentration (>50%) due to increase viscosity of the osmotic medium achieving satisfactory mass transfer characteristics is difficult; lower concentration is also inappropriate because of the reduction in driving force for water removal. Experiments were performed at three temperature level (30, 40 and 50 °C) using a circulating water bath (Memmert, WNE14. Memmert GmbH Co. KG, Germany) maintained at the studied temperatures (±0.5°C). The temperature of the water bath and osmotic medium was verified with a digital thermometer (Ellab CTD-85, Ellab, Denmark) and a thermocouple (1.2 mm needle diameter constantan type T). Higher temperatures lead to adverse effects on tissue, color and aroma of product. In contrast, lower temperatures prevent satisfactory mass transfer characteristics due to reduction of the osmotic medium viscosity. The sucrose solution to sample ratio was always 10:1 w/w to avoid significant dilution of the medium by water removal. The cubes were put in 2L beakers and introduced in the osmotic solution. The beakers were covered with a sheet of aluminium film to prevent evaporation of syrup during the process. After dehydration, the seedless guava cubes removed from the osmotic solution, rinsed quickly with distilled water (below 30s) to remove the solution

adhered to the surface and carefully blotted with tissue paper to eliminate the excess surface water. Sampling was performed in time intervals of 15, 30, 45, 60, 90, 120, 150, 180 and 240 min of osmotic dehydration. No shaking was used in any of the experiments. All experiments were performed in triplicate.

3.2.3 Analytical Determinations

In order to determine mass change, weights for seedless guava cubes were measured before and after treatment when the samples reached room temperature (25 ºC) using an analytical Mettler Toledo balance (±0.0001 g) (Mettler Toledo GmbH, Greinfensee, Switzerland). Moisture and solid contents were determined by the gravimetric method in oven (Heraeus Vacutherm VT6025, Germany) at 105 °C until a constant weight (24 h) was reached (AOAC, 1990). All measurements were carried out in triplicate.

3.2.4 Determination of Mass Transfer Kinetic Parameters

Weight reduction (WR); solid gain (SG); water loss (WL); and normalized moisture content (NMC) at different times, t, in agreement with the following expressions have been determined (Lerici *et al*., 1985; Panagiotou *et al*., 1999):

$$WR = \frac{M_0 - M}{M_0}, \quad (3.1)$$

$$SG = \frac{m - m_0}{M_0}, \quad (3.2)$$

$$WL = \frac{(M_0 - m_0) - (M - m)}{M_0}, \quad (3.3)$$

$$\mathrm{NMC} = \frac{1 - \frac{\mathrm{m}}{\mathrm{M}}}{1 - \frac{\mathrm{m_0}}{\mathrm{M_0}}} = \frac{\mathrm{X}}{\mathrm{X_0}} \quad (3.4)$$

Where M_0 is initial mass of fresh sample (g), M is the mass of sample after dehydration (g), m_0 is the initial mass of the solids in sample (g), m is mass of the solids in sample after dehydration (g), X_0 is initial moisture content (%) and X is moisture content of sample after dehydration (%).

3.2.5 Mass Transfer Model

The adaptation of Peleg equation for the present study is given by Eq. (3.5) (Corzo and Bracho, 2006).

$$X = X_0 \pm \frac{t}{K_1 + K_2 t} \quad (3.5)$$

Where X is dependent variable at time t, X_0 is initial dependent variable, K_1 is the Peleg rate constant, and K_2 is the Peleg capacity constant. The Peleg rate constant K_1 relates to dehydration rate at the very beginning, $t = t_0$

$$\frac{dX}{dt} = \pm \frac{1}{K_1} \quad (3.6)$$

The Peleg capacity constant K_2 relates to minimum attainable dependent variable. As $t \rightarrow \infty$, Eq. (3.7) gives the relation between equilibrium content (X_e) and K_2

$$X_e = X_0 \pm \frac{1}{K_2} \quad (3.7)$$

Our assumption was that the Peleg model would predict kinetics of osmotic dehydration process including equilibrium content.

Dependency of the Peleg rate constant to temperature is represented by the linearized Arrhenius equation (Sopade *et al*., 1990):

$$ln(K_1) = ln(K_0) - \frac{E_a}{RT} \quad (3.8)$$

Where K_1 is the Peleg rate constant (min g/g^{-1}), K_0 is the frequency factor (min^{-1}), E_a is the activation energy (kJmol^{-1}), R the universal gas constant (8.314 J/mol K) and T is the absolute temperature (K).

3.2.6 Experimental Designs and Statistical Analysis

3.2.6.1 The Kinetics Study

The experimental design applied was a 3 × 3 × 9 factorial design in a frame of Complete Randomized Design (CRD), corresponding to the solution concentration of sucrose (30, 40 and 50% w/w), temperature (30, 40 and 50 ºC) and immersion time (15, 30, 45, 60, 90, 120, 150, 180 and 240 min). The results were statistically analyzed by analysis of variance (ANOVA) and mean differences were compared using Tukey test at 95% of confidence level using the Minitab Release 14.0 (Minitab Inc. State College, PA, USA). Non-Linear regression using Levenberg-Marquardt

method was used for fitting database to Peleg model using the STATISTICA 6.0 software (StatSoft, Inc., USA). The quality of the fit between the experimental and predicted data was determined according to values of correlation coefficient (R^2), chi-square (χ^2), the root mean square error (RMSE), and the mean relative deviation modulus (E) (Deng and Zhao, 2008). These criterions can be calculated as follows:

$$\chi 2 = \sum_{i=1}^{n} \frac{\left(V_{exp} - V_{pre}\right)^2}{V_{pre}} \qquad (3.9)$$

$$RMSE = \frac{1}{n}\left[\sum_{i=1}^{n}\left(V_{exp} - V_{pre}\right)^2\right]^{0.5} \qquad (3.10)$$

$$E(\%) = \frac{100}{n}\sum_{i=1}^{n} \frac{\left|V_{exp} - V_{pre}\right|}{V_{exp}} \qquad (3.11)$$

where V_{exp} and V_{pre} are the experimental and predicted values, respectively, n is the number of experimental data. A model is considered acceptable if *E* value is less than 10% (Deng and Zhao, 2008). Therefore, the best model was chosen as one with the highest coefficient of correlation (R^2), the least $\chi 2$, RMSE, and E values.

3.2.6.2 The Optimization Study

Optimization of the osmotic dehydration process with three independent process variables at five levels namely osmotic solution concentration (30–50% w/w, x_1), temperature (30 - 50 ºC, x_2) and immersion time (15 - 240 min, x_3) was carried out using response surface methodology (RSM). The levels of different process variable for osmotic dehydration process are shown in Table 3.1. A Central Composite Design (CCD) was used for designing the experimental data. This generated 20

treatments with six replications at the center point to estimate the repeatability of the method (Montgomery, 2001). In order to minimize the effect of unexplained variability induced by extraneous factors on the observed response, the order of the experiments were randomized (Liyana-Pathirana and Shahidi, 2005). For predicting the responses as function of independent variables, a second-order polynomial equation was fitted to data:

$$Y_i = \beta_0+\beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 \qquad (3.12)$$

Where Y_i is predicted responses (i.e. WR, SG and WL), β_n are constant regression coefficients; x_1, x_2 and x_3 are the independent variables. The significant terms ($p < 0.05$) in the model were found by ANOVA for each response. The model adequacies were checked by model analysis, lack-of-fit test, coefficient of determination R^2 and adjusted-R^2 (Myers and Montgomery, 1995; Weng *et al*., 2001). In order to obtain the final reduced model, the statistically non-significant ($p > 0.05$) terms were dropped from the initial models and the experimental data was refitted only to significant ($p < 0.05$) parameters. It should be noted that some variables were kept in the reduced model despite non-significance if a quadratic or interaction term containing this variable was significant ($p < 0.05$) (Mirhosseini *et al*., 2009).

The graphical and numerical optimization procedures were carried out to determine the optimum level of main process variables leading to maximum WR, WL and minimum SG. For graphical optimization procedure, based on final reduced models, three dimensional response surface was plotted to visualize the relationship between the significant ($p < 0.05$) interaction effects of factors and response variables. The three dimensional plots were drawn to show how each response variable related to

two continuous design variables by keeping one variable constant at the center point and varying the other two variables within the experimental range.

Table 3.1. The Levels of Different Process Variables in Coded and Uncoded Form for Osmotic Dehydration of Seedless Guava

Coded values	Uncoded values		
	Concentration, x_1 (%w/w)	Temperature, x_2 (ºC)	Time, x_3 (min)
-1.63	33.87	33.87	58.60
-1.00	30.00	30.00	15.00
0	40.00	40.00	127.50
+1.00	50.00	50.00	240.00
+1.63	46.12	46.12	196.39

Numerical optimization was also carried out using response optimizer to predict the exact optimum level of independent variables leading to the desirable response goals. Experimental data were compared with predicted values (method validation) in order to verify the adequacy of final reduced models. Close agreement and no significant difference must exist between the experimental and predicted values. The experimental design matrix, analysis of variance, regression coefficients, generation of three-dimensional graphs and optimization procedure were performed using the Minitab Release 14.0 statistical software (Minitab Inc. State College, PA, USA). Schematic diagram of experimental procedure is presented in Figure 3.1.

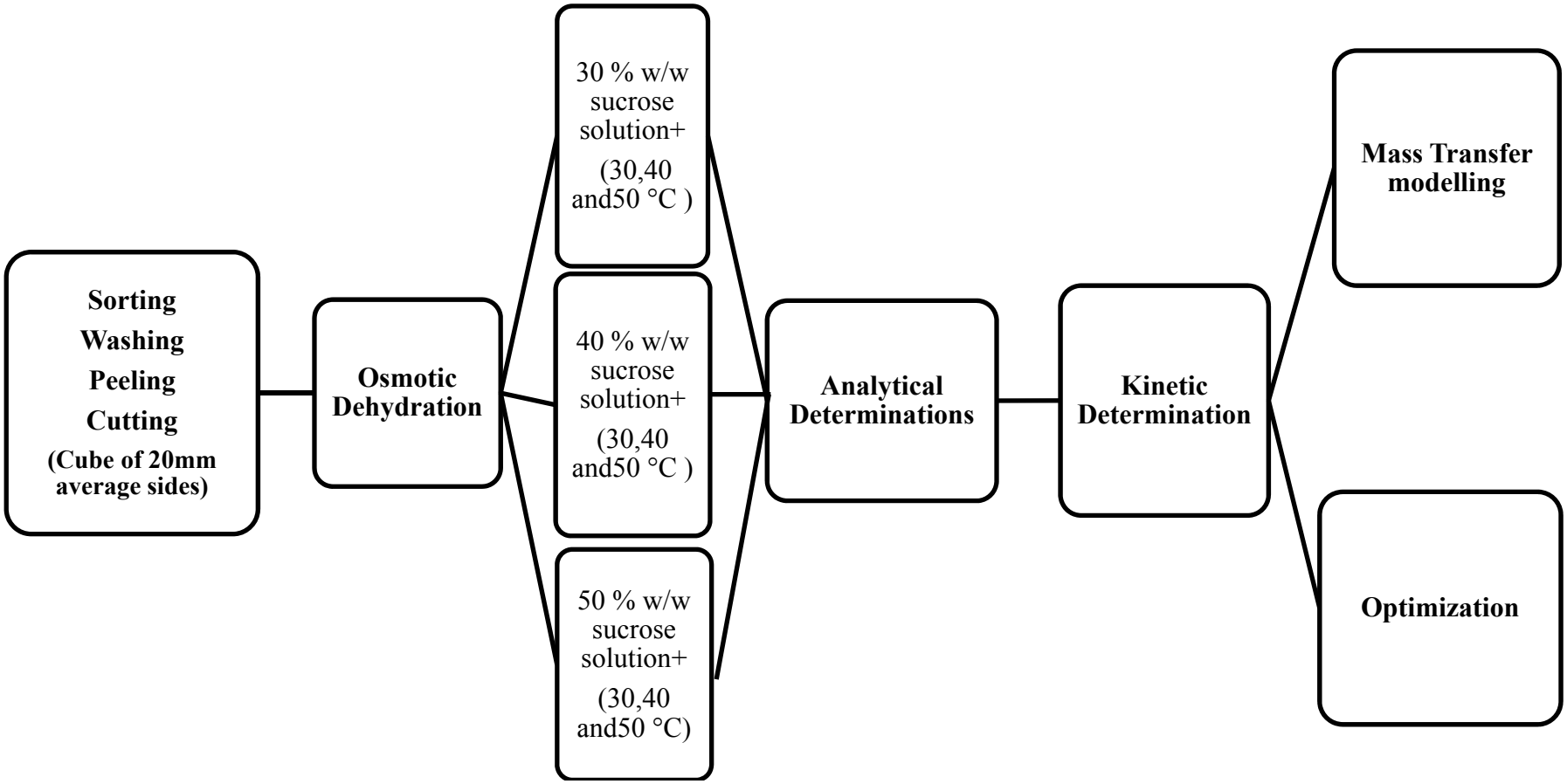


Figure 3.1. Schematic Diagram of Experimental Procedure

3.3 Results and Discussion

3.3.1 Effect of Process Parameters on Osmotic Dehydration of Seedless Guava

The effects of sucrose solution concentration, temperature and immersion time as process parameters was studied in terms of WR, SG, WL and NMC during osmotic dehydration and was statistically analyzed through ANOVA. By considering the probability results, the process variables and some of its interaction affected WR, SG, WL and NMC as dependent variables significantly ($p < 0.05$).

Temperature was the first variable analyzed. Results revealed that the temperature affected the kinetic terms of osmotic dehydration significantly ($p<0.05$). As our expectation, more intensive dehydration was achieved by increasing temperature from 30 to 50 ºC obtaining higher amounts of WR, SG, WL and lower values of NMC (see Figures 3.2-3.5). The higher intensity of temperature effect was observed in the most concentrated solution. This result accords with those obtained by previous researchers (Chenlo *et al*., 2006; Falade *et al*., 2007; Tortoe *et al*., 2007). Application of high temperature leads to increasing the kinetics of mass transfer due to diffusion rate enhancement in this condition. This could be related to high degree of freedom and mobility of water molecules which promote escape from the constituent resulting to increased WL (Azoubel and Murr, 2002; *et al*., 2006). Acceleration in WL and SG was obtained using higher temperatures through swelling and plasticizing of cell membranes as well as the better water transfer characteristics on the sample surface as a result of lower viscosity of the osmotic solution (Lenart and Flink, 1984; Le Maguer, 1988; Uddin *et al*., 2004; Singh *et al*.,

2007; Mercali *et al*., 2010). Similar results have been provided for different fruits and vegetables (Pokharkar, 2001; Corrêa *et al*., 2010). However, Pointing *et al*. (1966) claimed that biochemical reaction rate of enzymatic browning and flavor deterioration enhances with temperature about 45 ºC.

As second variable, the effect of solution concentration was analyzed. ANOVA results showed that the osmotic solution concentration affecting the kinetic terms of osmotic dehydration significantly ($p < 0.05$). As expected, using a higher osmotic solution concentration leads to higher extent of the dehydration process due to the larger osmotic driving force between the fresh fruit and the surrounding osmotic solution (Azoubel and Murr, 2004; Falade *et al*., 2007). This result corroborates with those obtained by several research groups for osmotic dehydration of cantaloupe, mango slices, apricot and guava halves (Fermin and Corzo, 2005; Mastrantonio *et al*., 2005; Ito *et al*., 2007; Ispir and Togrul, 2009; Corrêa *et al*., 2010). On the other hand, Lazarides *et al*. (1997) demonstrated that improvement of mass transfer in term of SG with increasing solution concentration due to membrane swelling effect can lead to increase permeability of the cell membrane. Thus, it is clear that enhancement of WL can be achieved using a higher solution concentration while a greater gain of solids was observed (Azoubel and Murr, 2004). The highly concentrated solutions (>50% w/w) may promote the formation of a sugar surface layer which acts as a barrier against penetration of the solutes into the food and the removal of water from fruits tissue. Khoyi and Hesari (2007) stated that viscosity of the sucrose solution limited the rate of mass transfer during dehydration process. They reported that the viscosity is high at 60-70% w/w resulting in the slowing down of the rate of WL.

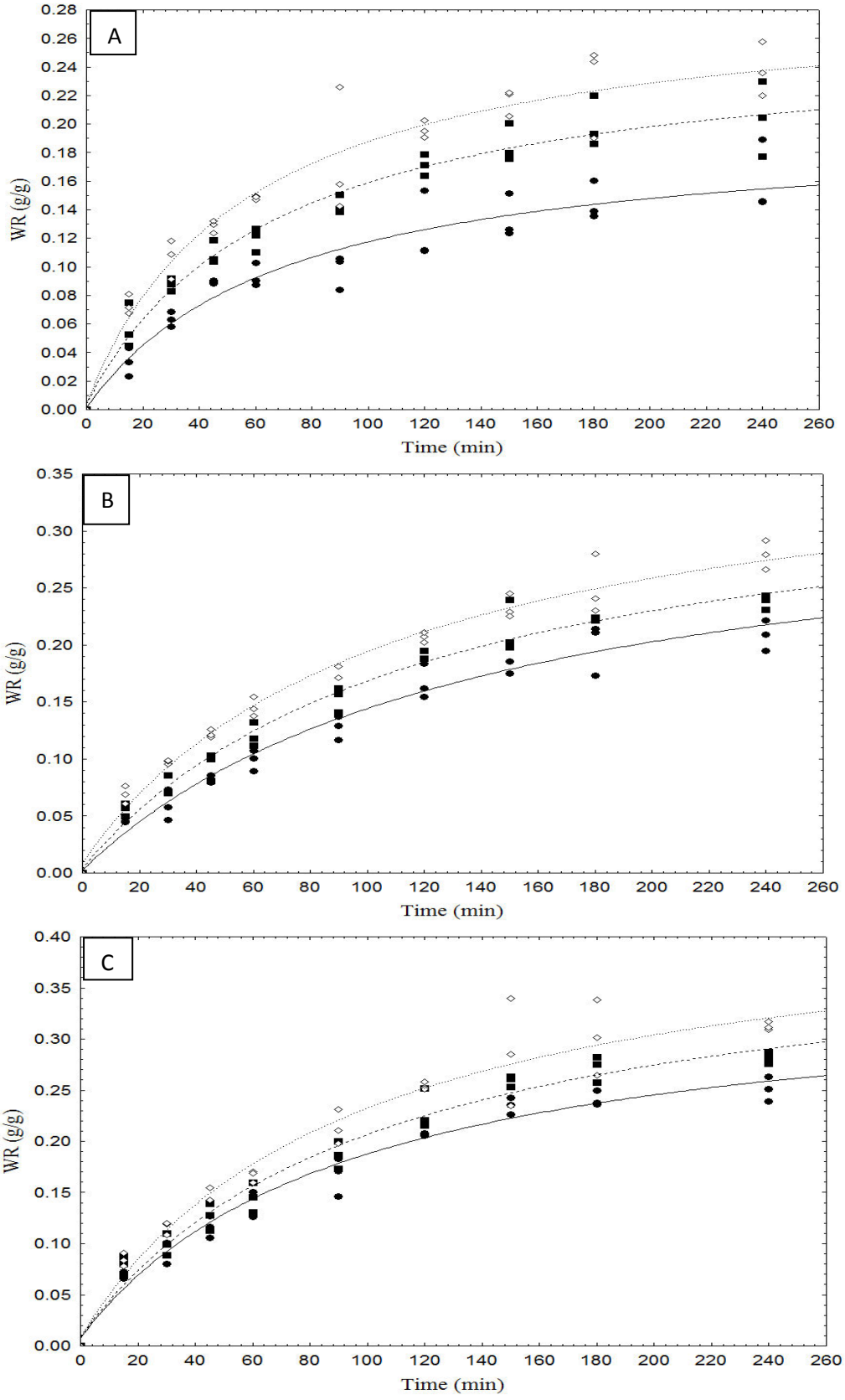


Figure 3.2. Weight Reduction (WR) of Seedless Guava during Osmotic Dehydration using Sucrose Solutions. (A) 30.0% (w/w); (B) 40.0% (w/w); (C) 50% (w/w) at Different Temperatures (●) 30 °C;(■) 40 °C;(◊) 50 °C (The lines represent Peleg model)

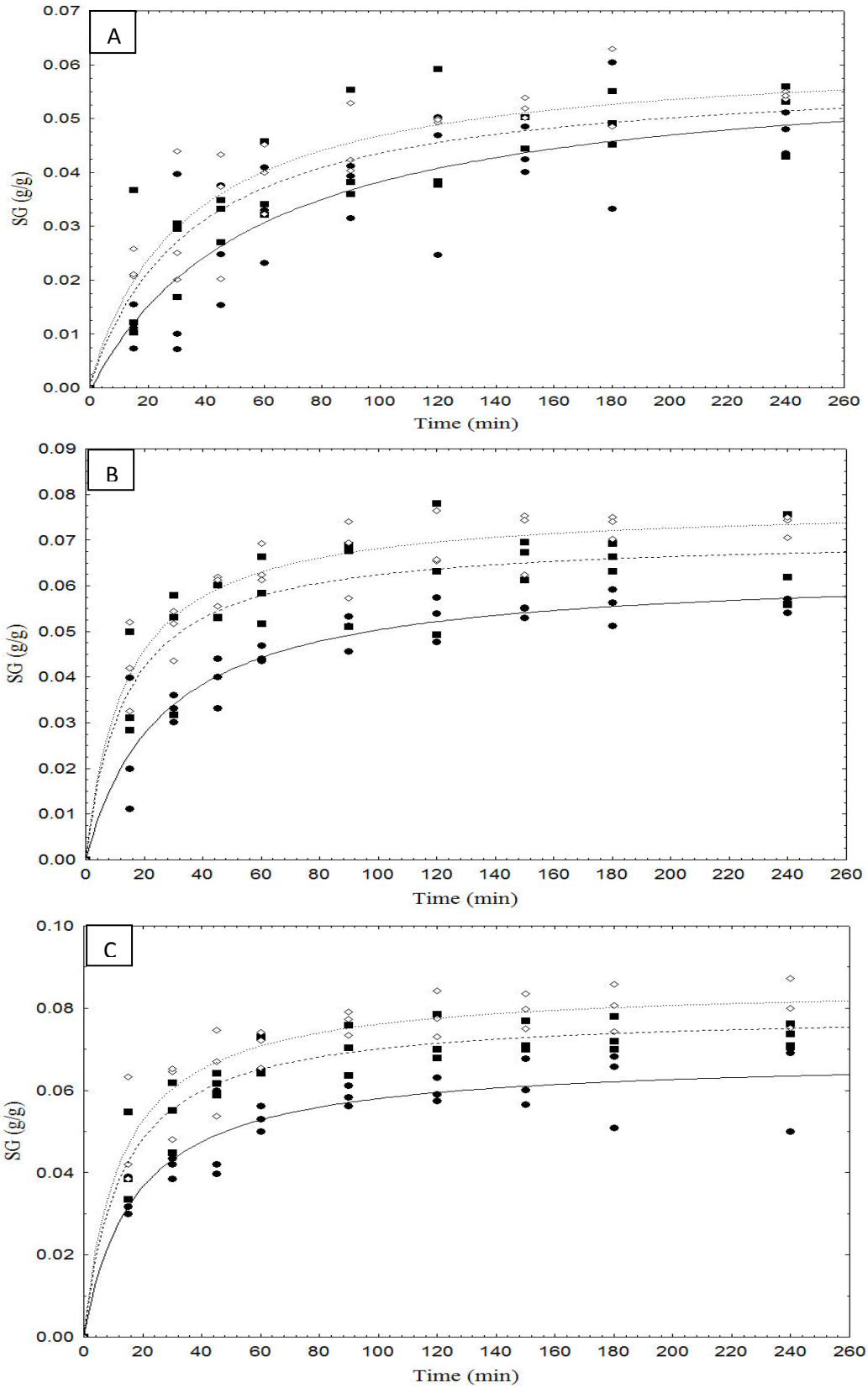


Figure 3.3. Solid Gain (SG) from the Osmotic Dehydration of Seedless Guava using Sucrose Solutions. (A) 30.0% (w/w); (B) 40.0% (w/w); (C) 50% (w/w) at Different Temperatures (●) 30 °C;(■) 40 °C;(◊) 50 °C (The lines represent Peleg model)

On the other hand, an initial rapid rate of mass transfer and then a reduction in the rate was observed and system getting closer to the end of the osmotic process (pseudoequilibrium). This behavior was similar to results reported on osmotic dehydration of different fresh fruits like papaya, kiwi, banana, apple and peach that reached equilibrium around 240 min (Palou *et al*., 1993; Askar *et al*., 1996; Panagiotou *et al*., 1999). Moreira *et al*. (2007) described that using low concentrated solutions lead to reach pseudoequilibrium condition faster due to the fact that the osmotic driving forces for moisture and solid transfer decreased with progression of time. In addition, the rapid WL and SG near the surface layers of tissue in the beginning of the process caused structural changes leading to increase resistance for water and solids transfer due to surface layers compactness (Tortoe *et al*., 2007). Andrade *et al*. (2003) pointed out that equilibrium stage is probably affected by the type of fruit´s membrane.

The ANOVA results for the effect of different time intervals (15–240 min) on WR, SG, WL and NMC showed significant difference ($p < 0.05$) at all combinations of concentration and temperature. As expected, the WR, SG, WL of seedless guava cubes increased and NMC decreased with an increase in immersion time. Depending on the sucrose concentration and the temperature range of the osmotic medium, the equilibrium conditions are reached in 240 min (Figures 3.1-3.5). Thus, the equilibrium values were predicted using Peleg's model (Eq 3.7).

The ratio of WL/SG applied to assess the efficiency of the osmotic treatment (Sereno *et al*., 2001; Matuska *et al*., 2006). The higher value is due to the very low value of SG which reflects the higher quality of the dehydration, whereas low values

correspond to good infusion of osmotic agents. The value of this ratio depends on physico-chemical characteristics of fresh fruit (initial moisture content, density, etc) and experimental conditions (type of osmotic agent, immersion time, solution concentration, temperature, etc.) (Sereno *et al*., 2001; Mayor *et al*., 2006). The final WL/SG values are presented in Table 3.2. The most efficient treatments were with 30% w/w sucrose solution at 40 and 50 ºC.

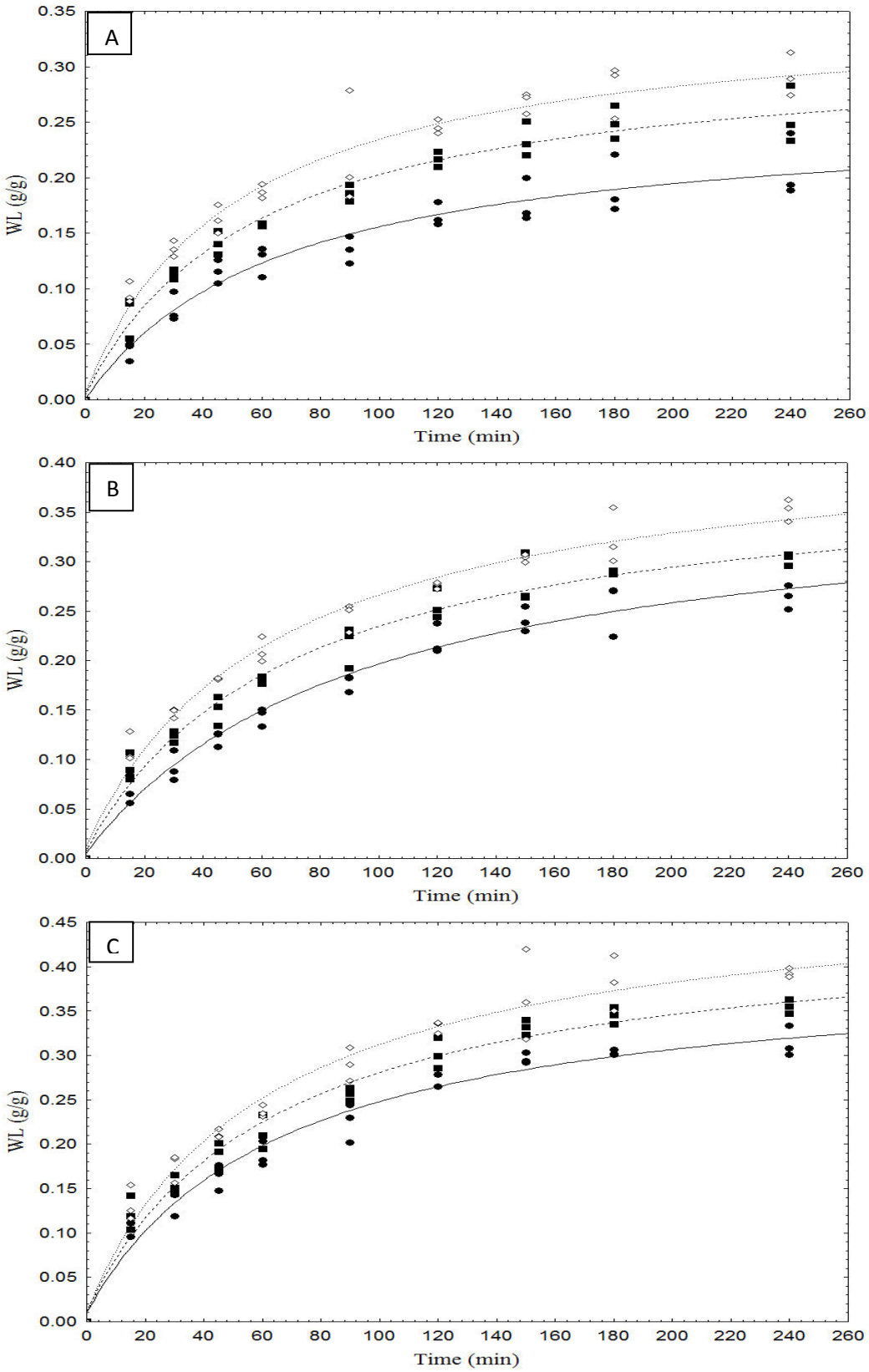


Figure 3.4. Water loss (WL) from the Osmotic Dehydration of Seedless Guava using Sucrose Solutions. (A) 30.0% (w/w); (B) 40.0% (w/w); (C) 50% (w/w) at Different Temperatures (●) 30 °C;(■) 40 °C;(◊) 50 °C (The lines represent Peleg model)

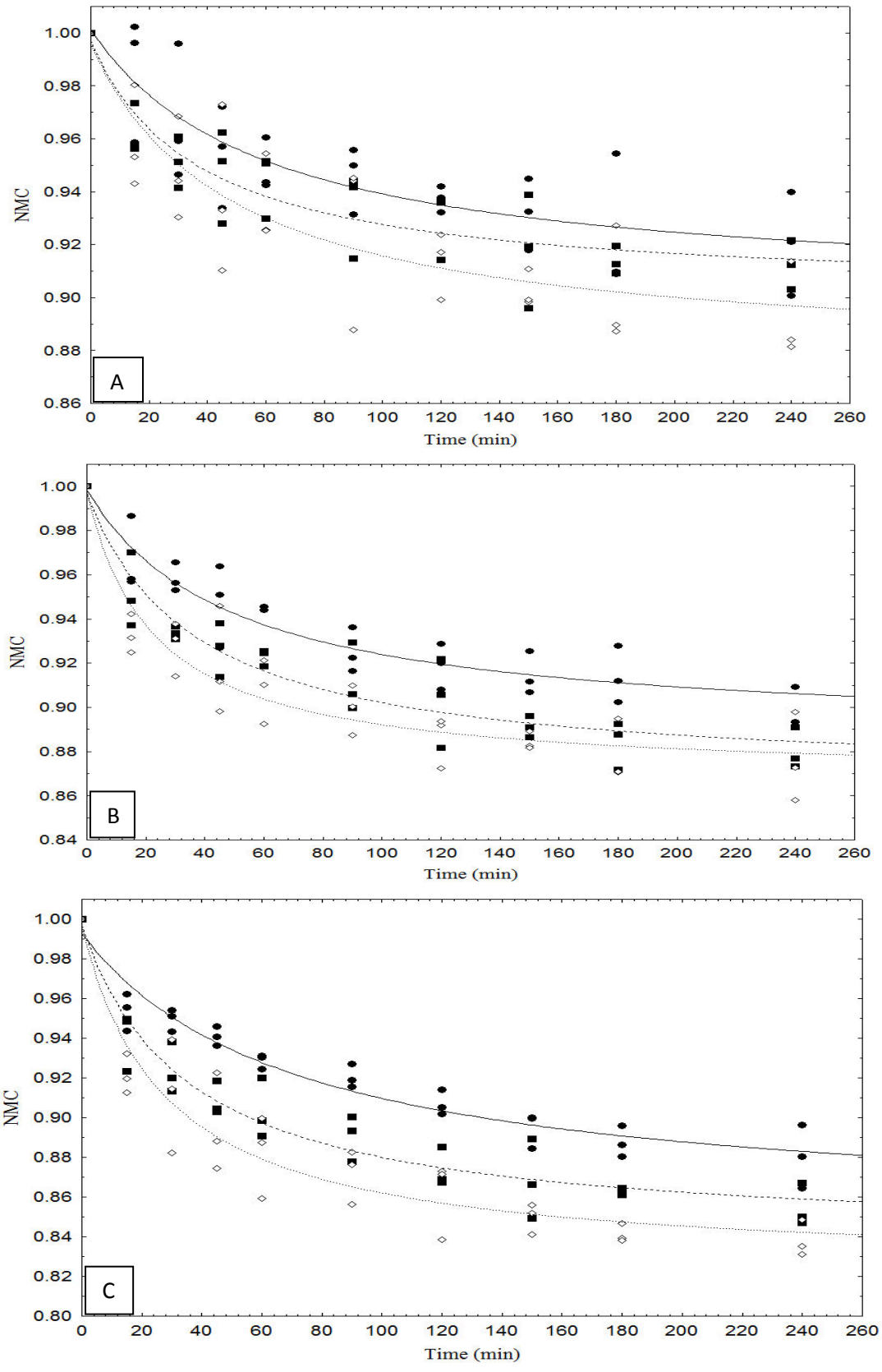


Figure 3.5. Normalized Moisture Content (NMC) from the Osmotic Dehydration of Seedless Guava using Sucrose Solutions. (A) 30.0% (w/w); (B) 40.0% (w/w); (C) 50% (w/w) at Different Temperatures (●) 30 °C;(■) 40 °C;(◊) 50 °C (The lines represent Peleg model)

Table 3.2. Values of WR, SG, WL and WL/SG after 240 min of Osmotic Dehydration of Seedless Guava at Different Employing Sucrose Concentration and Temperature

Concentration (% w/w)	Temperature (ºC)	WR (g/g)	SG (g/g)	WL (g/g)	NMC (%)	WL/SG
30	30	0.16±0.02 [a]	0.047±0.003 [a]	0.20±0.02 [a]	0.92±0.10 [a]	4.25
	40	0.20±0.20 [b]	0.050±0.006 [b]	0.25±0.02 [b]	0.91±0.00 [a]	5.00
	50	0.23±0.01 [c]	0.054±0.000 [c]	0.29±0.01 [c]	0.89[b]±0.01	5.37
40	30	0.20±0.01 [a]	0.055±0.001 [a]	0.26±0.01 [a]	0.90±0.00 [a]	4.72
	40	0.23±0.00 [b]	0.064±0.010 [b]	0.30±0.00 [b]	0.88±0.00 [b]	4.75
	50	0.27±0.01 [c]	0.073±0.000 [c]	0.35±0.01 [c]	0.87±0.02 [b]	4.79
50	30	0.25±0.01 [a]	0.063±0.010 [a]	0.31±0.01 [a]	0.88±0.01 [a]	4.92
	40	0.28±0.00 [b]	0.073±0.002 [b]	0.35±0.00 [b]	0.85±0.01 [b]	4.79
	50	0.31±0.00 [c]	0.080±0.006 [c]	0.39±0.00 [c]	0.83±0.00 [c]	4.85

Values of WR, SG, WL and NMC were obtained by triplicate and expressed as value±SD.
Different superscripts denote significant difference ($p<0.05$) at each temperature for a practical concentration.

3.3.2 Kinetics Study of Osmotic Dehydration

3.3.2.1 Performance of Peleg Model for Estimation of WR, SG, WL and NMC of Seedless Guava

In order to evaluate the goodness of fit of the Peleg model during osmotic dehydration experimental data within the dynamic segments of WR, SG, WL and NMC and away from equilibrium conditions was used. Estimated Peleg parameters are shown in Table 3.3 and Table 3.6. The values of R^2 varied from 0.85 to 0.99 for all the kinetics terms. Such R^2 indicate a good fit to the experimental data. The chi-square, RMSE and E values for each experimental condition investigated are also given in Table 3.3 and Table 3.6. The high values of R^2 and the small values of chi-square, RMSE and E suggest that Peleg equation is suitable for predicting the kinetic terms of mass transfer for seedless guava at given solution concentrations and temperatures over immersion time.

3.3.2.2 Initial Mass Transfer Rate or K_1

The K_1 values for different solution concentrations and temperatures are presented in Table 3.3. The reciprocal of K_1 describes the initial mass transfer rate, e.g., the lower the K_1, the higher mass transfer rate. The variations of K_1 constant were subjected to ANOVA which the obtained results showed that concentration and temperature significantly ($p<0.05$) affected K_1 parameter. From Table 3.3 it can be observed that K_1values decreased ($p > 0.05$) with increasing temperature of osmotic solution from 30 to 50 ºC at constant solution concentration suggesting a corresponding increase in the initial mass transfer terms rate. Similar finding have been pointed out by

Lazarides *et al*. (1995), Pokharkar (2001), Khoyi and Hesari (2007) and Sutar and Gupta (2007) with osmotic dehydration of green peas, apple slices, apricot and onion slices, respectively.

It was also observed from Table 3.3 that K_1 for all kinetic terms of mass transfer by seedless guava decreased with increasing osmotic solution concentration at constant temperature. This finding was expected due to the fact that the osmotic driving forces for WL and SG would increase with the increased sucrose concentration in osmotic solution. On the other hand, Sachetti *et al*. (2001) attributed the behaviour to response of cellular structure to the osmotic pressure increment. Similar finding have been pointed out for osmotic dehydration of onions and apricot by Sagar (2001), Sutar and Gupta (2007), Khoyi and Hesari (2007).

Table 3.3. Peleg Rate Constants and Goodness of Fit of Peleg Model for Mass Transfer during Osmotic Dehydration

Con. (%w/w)	Temp. (ºC)	WR					SG					WL					NMC				
		K_1	R^2	Chi-square	RMSE	E (%)	K_1	R^2	Chi-square	RMSE	E (%)	K_1	R^2	Chi-square	RMSE	E (%)	K_1	R^2	Chi-square	RMSE	E (%)
30	30	356.33± 70.34	0.96	0.003	0.002	5.24	939.09± 359.51	0.88	0.0003	0.0003	2.91	257.49± 43.08	0.97	0.002	0.002	3.96	608.37± 259.13	0.85	0.00007	0.0009	0.26
	40	260.76± 35.09	0.98	0.002	0.001	3.93	612.46± 191.59	0.91	0.0004	0.0003	3.05	187.09± 20.62	0.98	0.002	0.001	3.12	392.68± 124.82	0.91	0.0001	0.001	0.42
	50	199.28± 32.57	0.97	0.001	0.001	3.03	549.78± 136.16	0.94	0.0006	0.0004	3.21	148.96± 20.92	0.98	0.002	0.001	3.03	407.38± 164.60	0.86	0.0002	0.001	0.40
40	30	413.20± 52.84	0.98	0.004	0.002	6.40	412.23± 78.88	0.96	0.0001	0.0002	1.71	255.53± 29.61	0.98	0.004	0.002	4.87	441.90± 108.82	0.94	0.00005	0.0007	0.20
	40	331.34± 34.20	0.99	0.004	0.002	5.43	191.58± 53.63	0.93	0.0002	0.0003	1.76	183.64± 19.35	0.98	0.005	0.002	4.33	283.45± 63.00	0.95	0.0002	0.001	0.52
	50	278.24± 25.63	0.99	0.003	0.001	3.58	180.86± 29.94	0.97	0.0003	0.0004	1.81	154.24± 14.90	0.99	0.005	0.003	3.88	177.71± 44.87	0.93	0.0003	0.001	0.52
50	30	270.82± 31.78	0.98	0.007	0.003	6.15	258.09± 53.49	0.95	0.0003	0.0004	2.25	166.55± 18.42	0.98	0.008	0.003	5.20	500.06± 92.28	0.96	0.0003	0.001	0.45
	40	258.96± 29.23	0.98	0.008	0.003	5.88	162.56± 26.39	0.97	0.0001	0.0003	1.25	145.27± 15.22	0.98	0.009	0.003	4.84	223.84± 40.74	0.96	0.0002	0.001	0.37
	50	215.03± 31.19	0.98	0.005	0.002	4.80	148.43± 27.85	0.96	0.0001	0.0003	1.35	125.27± 15.88	0.98	0.007	0.003	4.42	163.98± 36.87	0.95	0.0004	0.002	0.62

Values of K_1 are means of three replicates.
K_1 Peleg rate constant (min g/g^{-1})

3.3.2.3 Modeling Effects of Temperature and Concentration on the Peleg Rate Constant

The negative slope of a straight line generated by plotting of the logarithm of the K_1 vs. 1/T equal E_a/R whereas intercept equal $\ln(k_0)$. The linearity of the data ($R^2 > 0.70$) indicates that the K_1 for all the kinetics terms followed an Arrhenius relationship as a function of temperature, regardless of concentration (Table 3.4). Table 3.4 presents the calculated values of activation energy (E_a) and natural logarithm of frequency factor ($\ln(k_0)$). Higher value of E_a revealed the greater temperature sensitivity of Peleg rate constant (K_1). It was found that K_1 of NMC is the most temperature sensitive term (E_a = 16.53–45.60 kJmol^{-1}) compared with other terms of mass transfer.

Table 3.4. Activation Energy and Frequency Factor Values for Peleg Rate Constant of WR, SG, WL and NMC during Osmotic Dehydration at Different Sucrose Concentrations

Parameter	Concentration (% w/w)		
	30	40	50
WR			
$\ln(k_0)$	-3.52±0.22	-0.37±0.28	1.92±0.13
E_a(KJ/mol)	23.67±0.58	16.11±0.75	9.32±3.48
R^2	0.99	0.99	0.87
SG			
$\ln(k_0)$	-1.90±0.273	-7.51±6.16	-3.50±0.319
E_a(KJ/mol)	21.93±7.10	33.84±16.03	22.68±8.12
R^2	0.90	0.81	0.88
WL			
$\ln(k_0)$	-3.31±0.66	-2.66±1.26	0.52±0.18
E_a(KJ/mol)	22.31±1.60	20.61±3.32	11.59±0.48
R^2	0.99	0.97	0.99
NMC			
$\ln(k_0)$	-0.22±0.04	-8.60±0.46	-11.95±4.13
E_a(KJ/mol)	16.53±10.16	37.07±1.19	45.60±10.74
R^2	0.69	0.99	0.94

Table 3.5 shows the multiple linear regression coefficients for K_1 of WR, SG, WL and NMC as a function of absolute temperature (1/T) and sucrose concentration (C). The models as fitted correspond to

$$\ln K_{WR} = -0.521 - 0.00342(C) + 1969.5(1/T) \quad (3.13)$$

$$\ln K_{SG} = -1.69 - 0.0655(C) + 3145.7(1/T) \quad (3.14)$$

$$\ln K_{WL} = -1.24 - 0.0144(C) + 2186.2(1/T) \quad (3.15)$$

$$\ln K_{NMC} = -5.82 - 0.0278(C) + 3977.4(1/T) \quad (3.16)$$

The obtained models explained 57.30%, 88.20%, 86.30%, and 83.00% of the variability in K_1 of WR, SG, WL and NMC at the 99% confidence level, respectively (Table 3.5). With the mentioned models, it is possible to predict the initial mass transfer rate when the seedless guava cubes are osmotically dehydrated in sucrose solution in the range of 30-50% w/w and temperatures in the range of 30-50 ºC. In Eqs. (3.13-3.16) the coefficients for sucrose concentration are negatives corresponding to decrease K_1 with increase in sucrose concentration. In Eqs. (3.13-3.16) the coefficients for temperature are positives indicating that the K_1 decrease with increase in temperature.

Table 3.5. Multiple Linear Regression for Peleg Rate Constant as a Function of Sucrose Concentration (C) and Temperature (1/T)

Sources of variation	WL		SG		WR		NMC	
	Estimate	S.E.	Estimate	S.E.	Estimate	S.E.	Estimate	S.E.
Constant	-1.24*	1.36	-1.69*	3.43	-0.52*	2.26	-5.81*	2.86
C	-0.014*	0.004	-0.065*	0.010	-0.0034*	0.007	-0.027*	0.009
1/T	2186.22*	423.26	3145.72*	1065.54	1969.53*	704.38	3977.44*	889.38
R^2	0.86		0.88		0.57		0.83	

ln k = A + B(C) + D(1/T).
* p-value < 0.05.

3.3.2.4 Equilibrium Mass Transfer Rate or K_2

Peleg capacity constants (K_2) at different sucrose concentrations and temperatures are shown in Table 3.6. The variations of K_2 constant were subjected to ANOVA which the obtained results revealed that K_2 values of all kinetic terms were affected by concentration ($p \square 0.05$). The K_2 is related to equilibrium mass transfer terms, e.g., the lower the K_2, the higher the equilibrium content. The equilibrium contents were calculated using Eq. (3.7) and the results are presented in Table 3.7. Rahman and Lamb (1990) and Parjoko *et al*. (1996) explained the relative transport of WL and SG to reach equilibrium. They identified that at higher temperatures the approach to osmotic equilibrium is achieved more by flow of water from cell compared to the solids transport due to the fact that water diffused easily compared to solutes through the cell membrane. Figure 3.6 (A-D) shows the comparison between the experimental and predicted values using Peleg equation for equilibrium WR, SG, WL and NMC. Considering good linear correlation between the experimental and predicted values (R^2 ranging from 0.78 to 0.94) it can be concluded that the experimental data could fit into the kinetic model very well.

Table 3.6. Peleg Capacity Constants and Goodness of Fit for Mass Transfer during Osmotic Dehydration

Con. (% w/w)	Temp. (ºC)	WR					SG					WL					NMC				
		K_2	R^2	Chi-square	RMSE	E (%)	K_2	R^2	Chi-square	RMSE	E (%)	K_2	R^2	Chi-square	RMSE	E (%)	K_2	R^2	Chi-square	RMSE	E (%)
30	30	5.03± 0.35	0.96	0.003	0.002	5.24	16.32± 2.00	0.88	0.0003	0.0003	2.91	3.85± 0.22	0.97	0.002	0.002	3.96	10.03± 1.40	0.85	0.0000	0.0009	0.26
	40	3.84± 0.17	0.98	0.002	0.001	3.93	17.06± 1.53	0.91	0.0004	0.0003	3.05	3.17± 0.11	0.98	0.002	0.001	3.12	10.49± 0.96	0.91	0.0001	0.0010	0.42
	50	3.46± 0.18	0.97	0.001	0.001	3.03	16.23± 1.15	0.94	0.0006	0.0004	3.21	2.87± 0.12	0.98	0.002	0.001	3.03	8.41± 1.03	0.86	0.0002	0.0010	0.40
40	30	2.95± 0.22	0.98	0.004	0.002	6.40	15.74± 0.84	0.96	0.0001	0.0002	1.71	2.66± 0.13	0.98	0.004	0.002	4.87	9.00± 0.67	0.94	0.0000	0.0007	0.20
	40	2.76± 0.14	0.99	0.004	0.002	5.43	14.06± 1.05	0.93	0.0002	0.0003	1.76	2.56± 0.09	0.98	0.005	0.002	4.33	7.74± 0.49	0.95	0.0002	0.0010	0.52
	50	2.60± 0.11	0.99	0.003	0.001	3.58	12.89± 0.57	0.97	0.0003	0.0004	1.81	2.37± 0.07	0.99	0.005	0.003	3.88	7.74± 0.54	0.93	0.0003	0.0010	0.52
50	30	2.85± 0.14	0.98	0.007	0.003	6.15	14.73± 0.84	0.95	0.0003	0.0004	2.25	2.53± 0.42	0.98	0.008	0.003	5.20	7.02± 0.46	0.96	0.0003	0.0010	0.45
	40	2.46± 0.12	0.98	0.008	0.003	5.88	12.61± 0.54	0.97	0.0001	0.0003	1.25	2.26± 0.07	0.98	0.009	0.003	4.84	6.32± 0.33	0.96	0.0002	0.0010	0.37
	50	2.30± 0.14	0.98	0.005	0.002	4.80	11.67± 0.58	0.96	0.0001	0.0003	1.35	2.06± 0.08	0.98	0.007	0.003	4.42	5.81± 0.37	0.95	0.0004	0.0020	0.62

Values of K_2 are means of three replicates.
K_2 Peleg capacity constant $((g/g)^{-1})$

Table 3.7. Estimated Equilibrium WR, SG, WL and NMC Content at Different Conditions of Sucrose Concentration and Temperature

Concentration (% w/w)	Temperature (ºC)	Equilibrium WR (g/g)	Equilibrium SG (g/g)	Equilibrium WL (g/g)	Equilibrium NMC (%)
30	30	0.19±0.01	0.06±0.003	0.26 ±0.02	9.01E-01 ±0.010
	40	0.26±0.02	0.05±0.004	0.31±0.02	9.01E-01±0.003
	50	0.29±0.01	0.06±0.005	0.35±0.01	8.76E-01±0.002
40	30	0.34±0.01	0.06±0.001	0.37 ±0.01	8.87E-01±0.020
	40	0.36±0.03	0.07±0.001	0.39±0.01	8.67E-01±0.020
	50	0.39±0.01	0.07±0.002	0.43±0.02	8.67E-01±0.004
50	30	0.35±0.00	0.06 ±0.001	0.40±0.02	8.50E-01±0.002
	40	0.41±0.02	0.07±0.004	0.45±0.01	8.38E-01±0.004
	50	0.44±0.00	0.08±0.006	0.49±0.00	8.23E-01±0.009

All data were obtained by triplicate and expressed as value±SD.

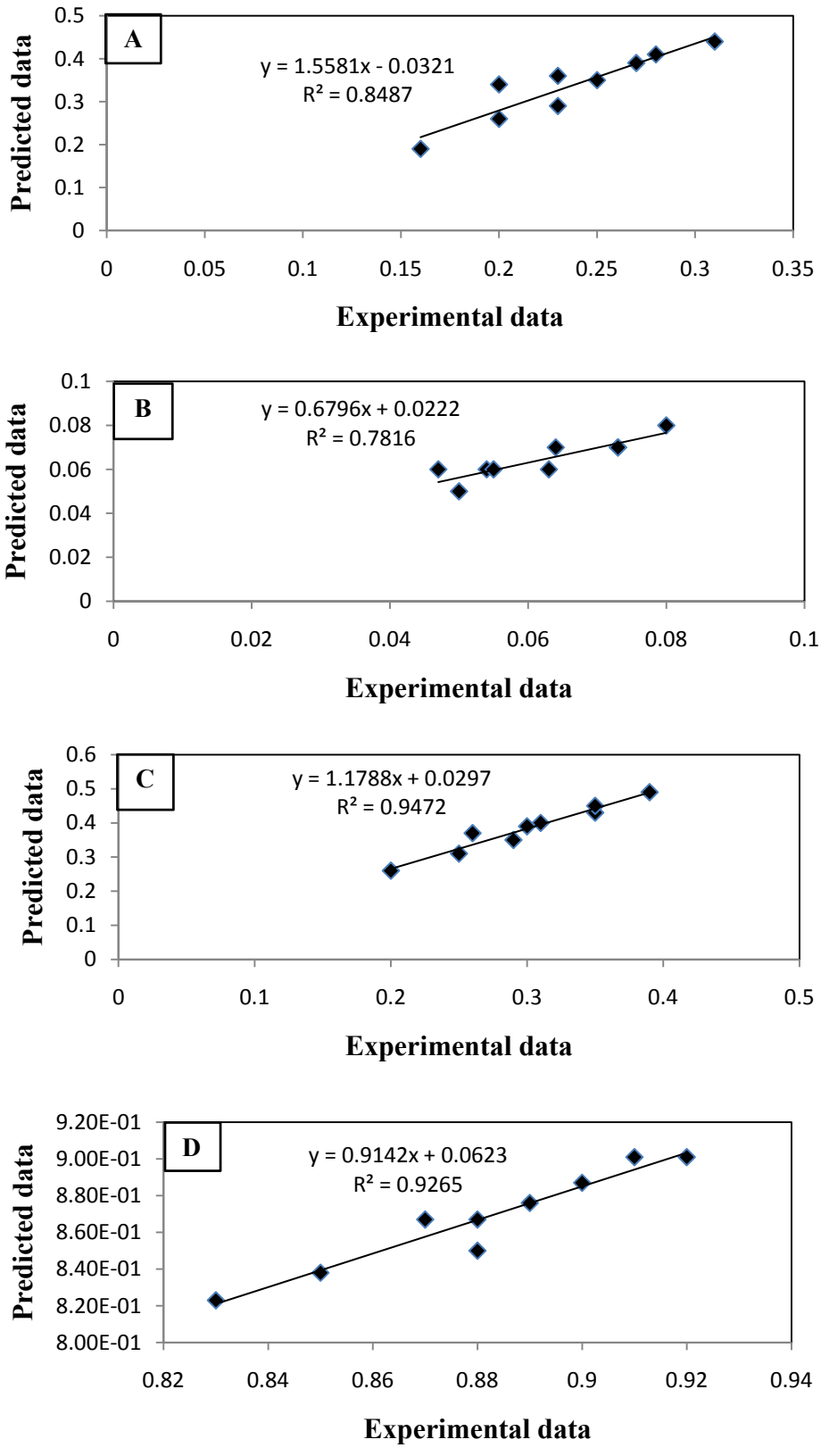


Figure 3.6. Comparison between the Experimental and Model Predicted Values of Equilibrium (A) WR (B) SG (C) WL and (D) NMC

3.3.3 Optimization of Osmotic Dehydration Process

3.3.3.1 Response Surface Analysis

Table 3.10 shows the experimental data for different runs of osmotic dehydration. ANOVA was carried out to determine the significant effects of process variables on WR, SG and WL. The estimated regression coefficients of the independent variables, along with the corresponding R^2, R-Sq (adj) and lack of fit test for the reduced response surface models are displayed in Table 3.8. The significant probability and the F-value of all main effects, linear, quadratic, and interaction of effects calculated for each response are also shown in Table 3.9; some non-significant terms ($p > 0.05$) were eliminated. The ANOVA results showed significant ($p < 0.05$) lack of fit ($p < 0.05$) for all responses (Table 3.8) which could be because of the wide ranges of the independent variables considered in current study (Capanzana and Buckle, 1997; Chua *et al*., 2009). Pua *et al*. (2007) claimed that when large amount of data were included in the analysis a model with a significant lack of fit could still be used. A good fit was obtained for WR ($R^2 = 0.98$), also the fit for SG ($R^2 = 0.97$) and WL ($R^2 = 0.98$) were satisfactory. Therefore, more than 97% of the response variation could be accurately explained as function of three osmotic dehydration process parameters. The response surface plots were generated as a function of two variables, while keeping other variable at the central value to visualize the combined effects of the two factors on the response (see Figures 3.7; 3.8; 3.9).

Table 3.8. Regression Coefficients and Analysis of Variance of the Reduced Regression Models for WR, SG and WL

Term	β		
	WR	SG	WL
Cons.	0.095788	-0.358834	-0.379189
Conc.	-0.001226	0.009273	-0.000337
Temp	-0.000289	0.007115	0.021295
Time	-0.000016	0.000791	0.001146
$(Conc.)^2$	-	-0.000099	-
$(Temp)^2$	-	-0.000068	-0.000248
$(Time)^2$	-0.000004	-0.000002	-0.000005
(Conc.*Temp)	-	-	-
(Conc.*Time)	0.000024	-	0.000025
(Temp*Time)	0.000017	-0.000007	-
Regression model (R^2)	0.98	0.97	0.98
Regression	0.000*	0.000*	0.000*
Lack-of-fit	0.000*	0.000*	0.000*
R-Sq (adj)(%)	96.9	95.6	97.0

β: regression coefficient.
* Significant at $p < 0.05$;

Table 3.9. Significant Probability (p-values and F- values) of the Independent Variable Effects in the Final Reduced Models

Variable		Main effects			Quadratic effects			Interaction effects		
		Conc.	Temp	Time	$(Conc.)^2$	$(Temp)^2$	$(Time)^2$	(Conc.*Temp)	(Conc.*Time)	(Temp*Time)
WR	p-value	0.187	0.747	0.967	-	-	0.000*	-	0.003*	0.017*
	F-value	1.93	0.10	0.001	-	-	88.736	-	14.28	7.56
SG	p-value	0.000*	0.001*	0.000*	0.000*	0.004*	0.000*	-	-	0.006*
	F-value	36.48	20.79	67.73	26.83	12.67	102.01	-	-	11.62
WL	p-value	0.753	0.002*	0.005*	-	0.003*	0.000*	-	0.007*	-
	F-value	0.10	16.16	11.83	-	14.13	108.99	-	10.69	-

* Significant at $p < 0.05$.

3.3.3.2 Analysis of Influence of Process Variables on Weight Reduction

The developed model (3.17) for WR, after neglecting nonsignificant ($p>0.05$) terms, was obtained as follow:

$$WR=0.095788-0.001226Conc-0.000289Temp-0.000016Time-0.000004Time^2 +0.0000240Conc*Time+0.0000170Temp*Time \quad (R^2=0.981) \qquad (3.17)$$

Table 3.9 indicates that linear terms of all the process variables have non-significant effect ($p>0.05$) on WR during osmotic dehydration. The quadratic effect of immersion time and interaction terms of 'concentration-immersion time' and 'temperature-immersion time' have also significant effects ($p<0.05$) on WR. The WR is decreased with increase of all process variables due to the negative signs of β-values of all linear terms. However, this decrease in WR was negligible with change in immersion time (β = 0.000016) and temperature (β =0.000289) as compared to sucrose concentration (β = 0.001226). The effect of sucrose concentration, temperature and immersion time on WR is given in Figure 3.7(A-B). The mass transfer rate was relatively high at the beginning of the process due to high osmotic driving potential between the hypertonic solution and the fresh fruit after that with progression of immersion time the rate gradually slowed down towards equilibrium condition (Eren and Kaymak-Ertekin, 2007).

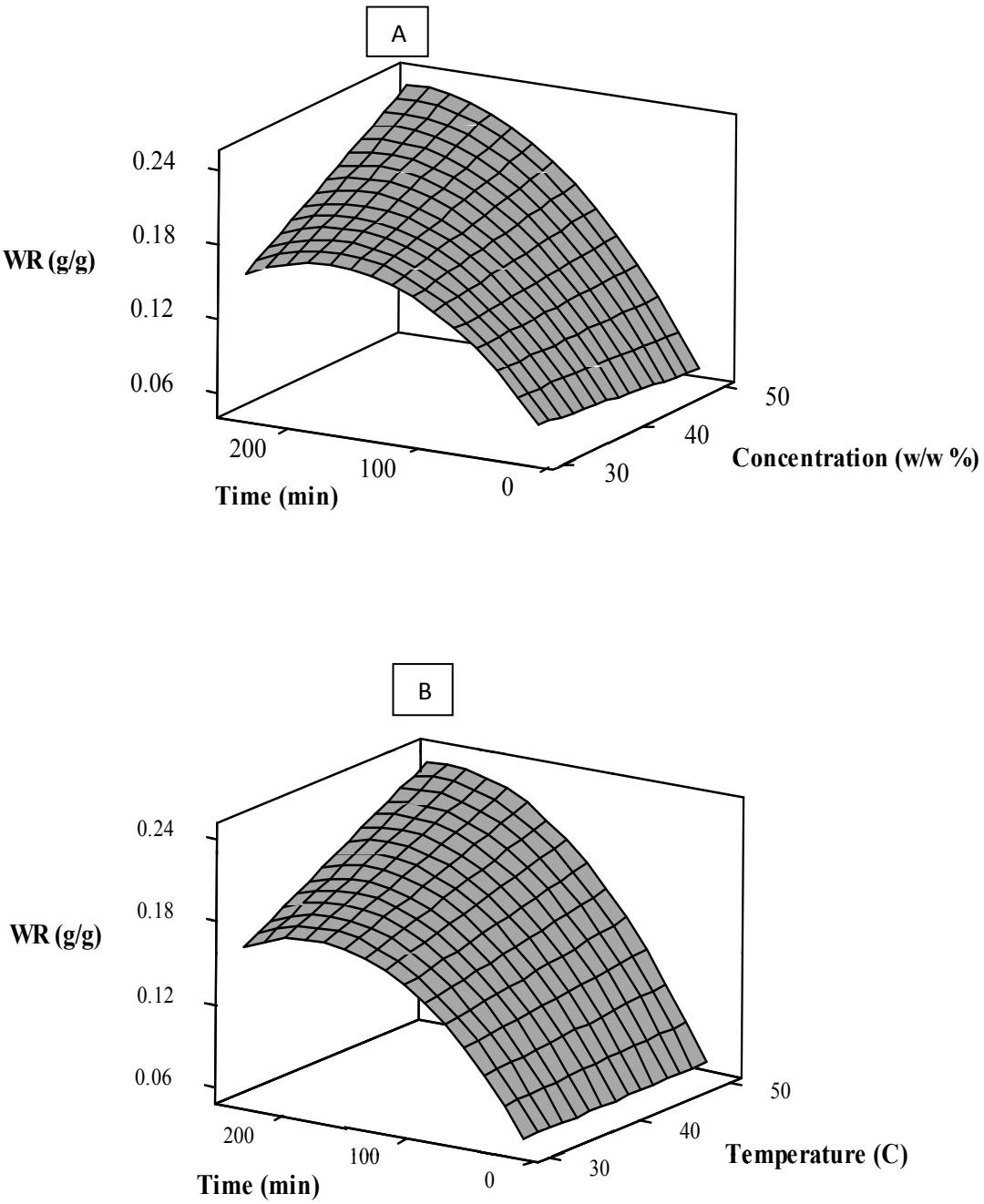


Figure 3.7. Response Surfaces for WR as a Function of: (A) Concentration and Immersion Time at 40 °C; (B) Temperature and Immersion Time at 40% w/w Sucrose Concentration

3.3.3.3 Analysis of Influence of Process Variables on Solid Gain

The following equation (3.18) for SG rendered concerning analysis of significant ($p<0.05$) coefficients of the uncoded second-order polynomial equation:

$$SG=-0.358834+0.009273Conc+0.007115Temp+0.000791Time-0.000099Conc^2-0.000068Tem^2-0.000002Time^2-0.000007Temp*Time \quad (R^2=0.974) \quad (3.18)$$

Table 3.9 shows the p-values which indicate that linear and quadratic terms of all process variables significantly affected ($p<0.05$) SG during osmotic dehydration. The interaction of 'temperature and immersion time' has a negligible negative significant effect ($p<0.05$) on SG. The magnitude of the β-values (Table 3.8) indicate the highest positive effect of sucrose concentration (β = 0.009273), followed by medium temperature (β=0.007115) and immersion time (β = 0.000791). These findings reveal an increased SG with increase of concentration and temperature of osmotic solution, and immersion time.

The effect of temperature on SG during osmotic dehydration can be seen from the Figure 3.8. High temperatures improve the cell membrane permeability to sugar molecules due to swelling and plasticizing of the membrane which leads to increases SG (Lazerides *et al*., 1997; Azoubel and Murr, 2003). The increase in SG blocks the surface layers of the tissue, and consequently lowering the rates of WR and WL at further processing times (Ertekin and Cakaloz, 1996; Shi and Le Maguer, 2002).

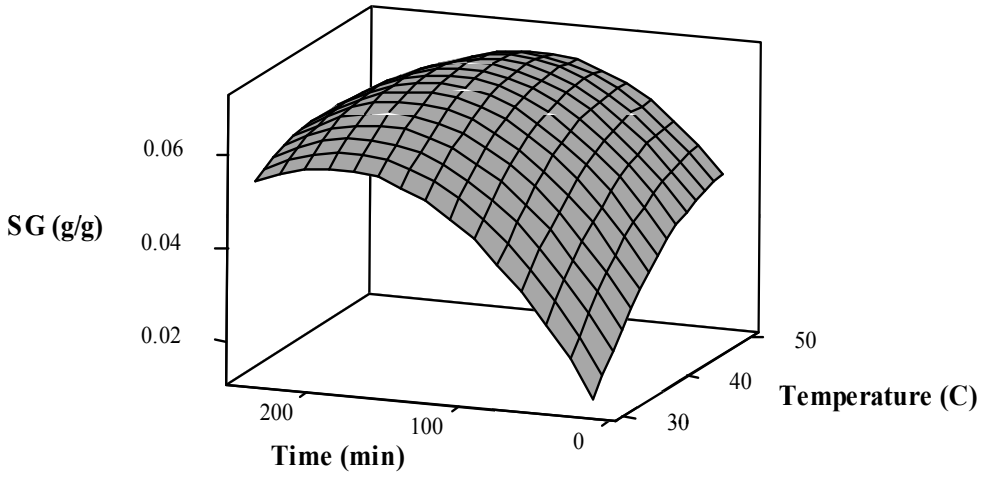


Figure 3.8. Response Surface for SG as a Function of Temperature and Immersion Time at 40% w/w Sucrose Concentration

3.3.3.4 Analysis of Influence of Process Variables on Water Loss

The following equation (3.19) for WL is obtained using all the significant ($p<0.05$) coefficients of the uncoded second-order polynomial equation:

$$WL=-0.37918-0.000337Conc+0.021295Temp+0.001146Time-0.000248Temp^2-0.000005time^2+0.000025Conc*Time \quad (R^2=0.981) \quad (3.19)$$

The p-values (Table 3.9) indicate that all linear terms of all process variables, the quadratic terms of temperature and time, and interaction of 'sucrose concentration and immersion time' affected WL significantly ($p<0.05$). The negative sign of concentration corresponds to decrease in the WL with increase of sucrose concentration. The magnitude of the β-values are presented in Table 3.8 which can conclude that solution temperature has the highest positive effect (β = 0.021295)

followed by immersion time (β = 0.001146). These findings revealed that increase of solution temperature and immersion time lead to an increase in WL values (Figure 3.9). Similarly, rapid WL at the beginning of osmotic dehydration of carrot and potato was reported (Uddin *et al*., 2004; Eren and Kaymak-Ertekin, 2007).

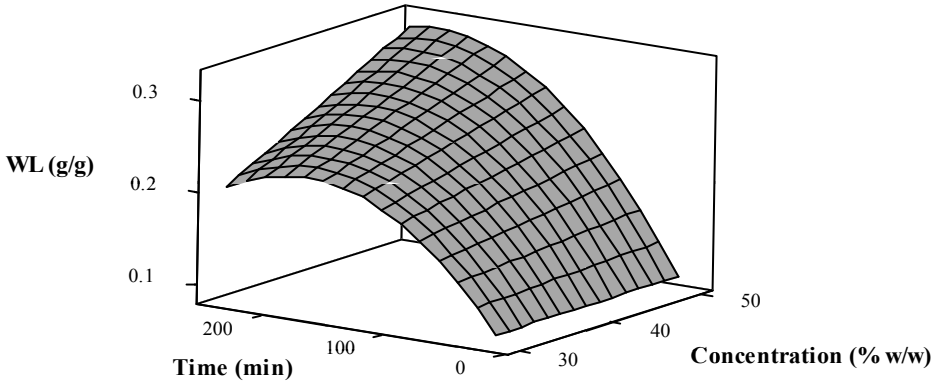


Figure 3.9. Response Surface for WL as a Function of Sucrose Concentration and Immersion Time at 40 °C

3.3.3.5 Numerical Optimization of Osmotic Dehydration Process

The obtained regression equations in current study are useful to determine desired optimum conditions which defined as the highest WR, WL and the lowest SG. According to Montgomery (2001), the reduced polynomial regression equations fitted to the experimental data was only a statistical empirical model in the selected range which may not be true out of the range of the process parameters. Thus, the model cannot be extrapolated out of these ranges. In order to find the optimum process conditions using numerical optimisation technique, equal importance of '1' was considered for all the responses. The optimal conditions for desired properties was estimated as 30% w/w concentration, 32.90 ºC temperature and 178.81 min

immersion time using response optimizer from the Minitab program. At the obtained optimum conditions, the predicted values for WR, WL and SG were 0.15 gg^{-1}, 0.20 gg^{-1} and 0.03 gg^{-1}, respectively.

3.3.3.6 Verification of the Final Reduced Models

Comparison between experimental and estimated values was carried out to validate the final reduced models (Figure 3.10). There was not any significant ($p > 0.05$) difference between the experimental and estimated values. Thus, the estimated values by the final reduced models were found to be in agreement with the experimental values. After osmotic dehydration under these optimal conditions, the WR, SG and WL were 0.14±0.01, 0.04±0.001, and 0.19±0.03, respectively. These values were not significantly different to the predicted values at 95% confidence interval.

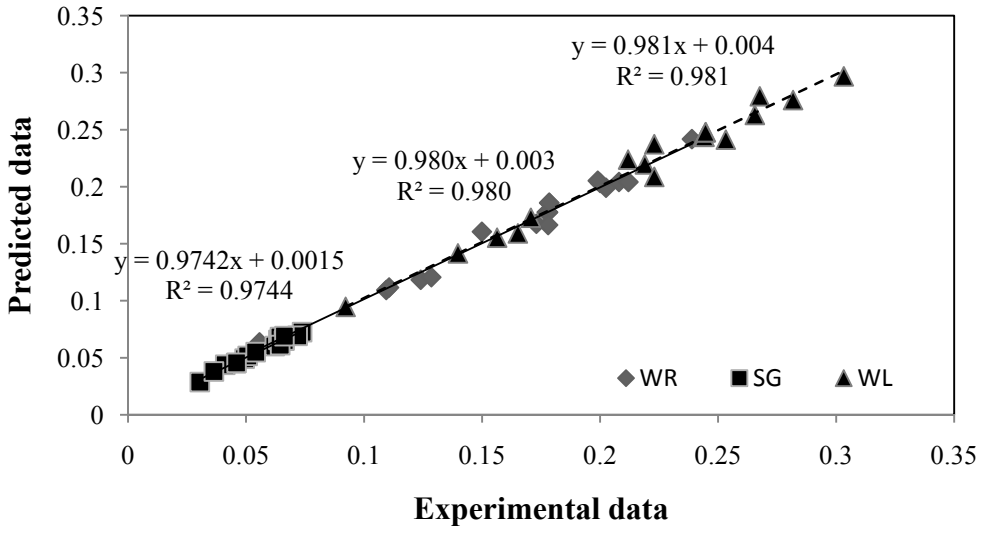


Figure 3.10. Comparison between Experimental and Predicted Values Based on the Final Reduced Models

3.4 Summary

The results of this study has shown that

1. Concentration and temperature of the osmotic solution, and immersion time has significant effect ($p < 0.05$) on the kinetics of WR, SG, WL and NMC.
2. The rate of osmotic dehydration increased as both sucrose concentration and temperature increased.
3. Peleg equation adequately described the kinetic terms of mass transfer, presenting high correlation coefficients ($R^2>0.85$) and low values of chi-square, RMSE and E (%).
4. Peleg parameters obtained from non-linear regression which K_1 describes the initial mass transfer rate whereas K_2 relates to equilibrium mass transfer terms.
5. The initial mass transfer rates increased significantly ($p<0.05$) with sucrose concentration and temperature whereas mass transfer rate at equilibrium stage affected significantly ($p<0.05$) by sucrose concentration.
6. The initial mass transfer rate constant for all kinetics terms all followed an Arrhenius relationship as a function of temperature ($R^2 > 0.69$).
7. The obtained models through multiple linear regression technique adequately explained the variability in the initial mass transfer rates.
8. The optimum condition for maximum WR and WL and minimum SG was found to be 30% w/w sucrose concentration, 33 °C solution temperature and 179 min immersion time.

CHAPTER IV

MODELLING THE KINETICS OF QUALITY ATTRIBUTES CHANGES DURING OSMOTIC DEHYDRATION OF SEEDLESS GUAVA (*Psidium Guaja*va L.)

4.1 Introduction

The low production and commercialization of tropical fruits products is due to the lack of technical processes for their preservation and transformation into good quality products (Pereira *et al*., 2006). Drying is probably the oldest method of food preservation, mainly because water removal and lowering of water activity lead to reduction in development of microbial contamination (Mandala *et al*., 2005). Osmotic dehydration presents clear advantages for producing good quality dried fruits due to its capability to minimize the heat damage on color and flavor (Krokida *et al*., 2001), prevents enzymatic browning and nutrient loss compared to traditional drying techniques (Torreggiani, 1993; Panagiotou *et al*., 1999; Sereno *et al*., 2001; Quiles *et al*., 2004). On the other hand, depending on characteristics of sample and conditions of osmotic process many changes in macro- and microscopic properties of sample including porosity /volume changes (Mayor and Sereno, 2004) and alteration in quality attributes (i.e. mechanical and optical properties) (Krokida *et al*., 2001; Telis *et al*., 2005) to varying degrees take place which associated mass and heat transfer during the process.

The knowledge on accurate kinetic parameters, rate constants and activation energies are important for prediction of quality changes (Steet and Tong, 1996). The kinetics of quality attributes changes of foods during processing and storage have been

addressed in several investigations (Avila and Silva, 1999; Ahmed *et al*., 2000; Ahmed *et al*., 2002; Elizalde *et al*., 2002; Zhu *et al*., 2004; Corzo *et al*., 2006; Garrote *et al*., 2008). Previous research findings have shown that degradation of food quality attributes during processing follows first-order kinetics model. So far, no previous study has investigated the kinetics of color and texture change of seedless guava during osmotic dehydration. Therefore, this study was dedicated to evaluate the influence of sucrose concentration and temperature on both optical (color) and mechanical (texture) properties during osmotic dehydration in which mathematical models were used to determine the kinetics of the selected quality changes.

4.2 Materials and Methods

4.2.1 Sample Preparation

Sample preparation procedure used in the present chapter is similar to that as described in Chapter 3 (i.e. 3.2.1).

4.2.2 Osmotic Dehydration Procedure

Osmotic dehydration procedure used in the present chapter is similar to that as described in Chapter 3 (i.e. 3.2.2).

4.2.3 Color Measurement

Color of fresh and osmotically dehydrated seedless guava were measured in Hunter scale using a portable tristimulus colorimeter (KonicaMinolta CR-300, USA) in terms of L-value (lightness), a-value (redness and greenness), and b-value (yellowness and blueness) as an average of three measurements at four different locations. Standardization of instrument was performed using white tile before measurements (L=97.67; a=0.08, and b=1.54). From these values, total color difference (ΔE) was calculated according to the following equations (Hunter, 1975):

$$\Delta E = \sqrt{(L-L_0)^2+(a-a_0)^2+(b-b_0)^2} \quad (4.1)$$

Where 0 subscript is the readings of ''L'', ''a'' and ''b'' at time zero.

4.2.4 Texture Analysis

Texture measurement was performed using a TA-XT2i texture analyser (TA/XT/PLUS Stable Micro Systems Ltd, Godalming, UK) using a 50 kg load cell and a 2-mm diameter cylindrical stainless steel probe (P/2). The program was set to measure force in compression mode, considering a 75% relative deformation. The test parameters were 2 mm/s of pre-speed and post-speed, 1 mm/s of test speed and 10 g of trigger. Six measurements were performed on each sample at different locations, two on the middle and four on the sides of the fruit in the corners of an imaginary square. Hardness (peak maximum force), area under the curve and slope of the initial section of the curve were used to characterize the textural properties of the samples. Data were analyzed using Windows based software, Texture Expert version 1.19 (Stable Micro Systems Ltd, Godalming, UK).

4.2.5 Vitamin C Determination

Ten grams of fruits were homogenized with 100 mL of 3% metaphosphoric acid and then filtered through Whatman (no.4) filter paper. An aliquot of 5 mL filtrate was titrated with 2, 6-dichlorophenol iodophenol (DCPIP) indicator to the end point according to according to the AOAC 967.21 (AOAC, 1995). Vitamin C content was reported as mg vitamin C per 100 g of sample. Determination of vitamin C contents was done in triplicate.

4.2.6 Calculation of Quality Changes Kinetics

The zero- (Eq. (4.2)) and first-order (Eq. (4.3)) equations have been proposed in the literature to describe the color and texture changes in fruits (Krokida *et al*., 2001):

$$C = C_0 \pm kt \qquad (4.2)$$

$$\ln \frac{C}{C_0} = -k \times t \qquad (4.3)$$

Where 0 is the initial value at time zero, k is the rate constant, and t is time.

Linearized Arrhenius Law describes the temperature dependence of the rate constant (Cruz *et al.,* 2008):

$$\ln(k) = \frac{E_a}{RT} + C \qquad (4.4)$$

4.2.7 Experimental Design and Statistical Analysis

The experimental design applied was a 3 × 3 × 9 factorial design in a frame of Complete Randomized Design (CRD), corresponding to the solution concentration

of sucrose (30, 40 and 50% w/w), temperature (30, 40 and 50 ºC) and immersion time (15, 30, 45, 60, 90, 120, 150, 180 and 240 min). The results were statistically analyzed as described in Chapter 3 (i.e. 3.2.6.1). Linear regression was used to fit data to Eq. (4.2) and (4.3) in order to estimate rate constant of quality attributes changes using STATISTICA 6.0 (Statsoft Inc.). The correlation coefficient (R^2) value was used to identify the goodness of fit between the experimental and predicted data.

4.3 Results and Discussion

4.3.1 Influence of Process Variables on Color Parameters of Seedless Guava

The selection of an adequate method of dehydration of a food material is based on the quality of the dried product. Color plays an important role in appearance, processing, and acceptability of food materials. Loss of color during osmotic dehydration process is one of the most significant changes. The factors responsible for the loss of color during osmotic process are physical and chemical including degradation, loss or concentration of fruit pigments and development of browning. Therefore, the color parameters of seedless guavas were measured before and after osmotic dehydration process.

The Lightness (L), redness (a) and yellowness (b) values of fresh seedless guava were 72.51±1.35,-3.18±0.8, and 28.14±1.33, respectively. Normalization of the individual quality parameters values was done by dividing the parameters by the corresponding initial values to minimize the biological variability between different

raw samples (Cruz *et al*., 2007). Therefore, all the figures start from 1. During osmotic dehydration of seedless guava ‘‘L’’, ‘‘a’’, and ‘‘b’’ values decreased with an increase in sucrose concentration, temperature and therefore ΔE increased in all cases suggesting that seedless guavas lose lightness, greenness and yellowness. Analysis of variance (ANOVA) of the experimental data showed significant effect of sucrose concentration, temperature and process duration ($p<0.05$) on the color parameter changes of seedless guava.

The variations of normalized ‘‘L’’, ‘‘a’’, ‘‘b’’, and total color difference (ΔE) with dehydration time as function of sucrose concentration and solution temperature were illustrated in Figures 4.1- 4.4. These results are in agreement with Forni *et al*. (1997), Waliszewsli *et al*. (1999), Valencia *et al*. (2003) and Moreira *et al*. (2005) who observed similar behavior in color changes of osmotically treated apricots, banana and chestnuts.

Generally, the color parameters ‘‘L’’ and ‘‘a’’ are well correlated to browning reaction (Mastrocola and Lerici, 1991; Krokida *et al*., 2001; Medina-Torresa *et al*., 2008). Enzymatic and non-enzymatic reactions are the most important reason of browning development in fruits and vegetables during dehydration. Polyphenoloxidase and peroxidase which is present in low acid fruits involved in enzymatic browning (Badui, 1993; Moreno-Castillo *et al*., 2005). High concentration of peroxidase in seedless guava confirms this assumption. As browning increases, ‘‘L’’ values decrease (Deng and Zhao, 2008). This reduction of lightness values (Figure 4.1) could also be attributed to the shrinkage of plant tissue which leads to increase in samples opacity (Contreras *et al*., 2008; Heredia *et al*., 2009). The

changes of redness and yellowness (Figures 4.2-4.3) are clear and seem to be relevant to alteration of fruit pigments and solids uptake (Forni *et al*., 1997; Rodrigues *et al*., 2003; Falade and Igbeka, 2007). The value of ΔE was reached to a maximum value at temperatures of 40 and 50° C which revealed that temperature is the strongest factor affecting color (Figure 4.4).

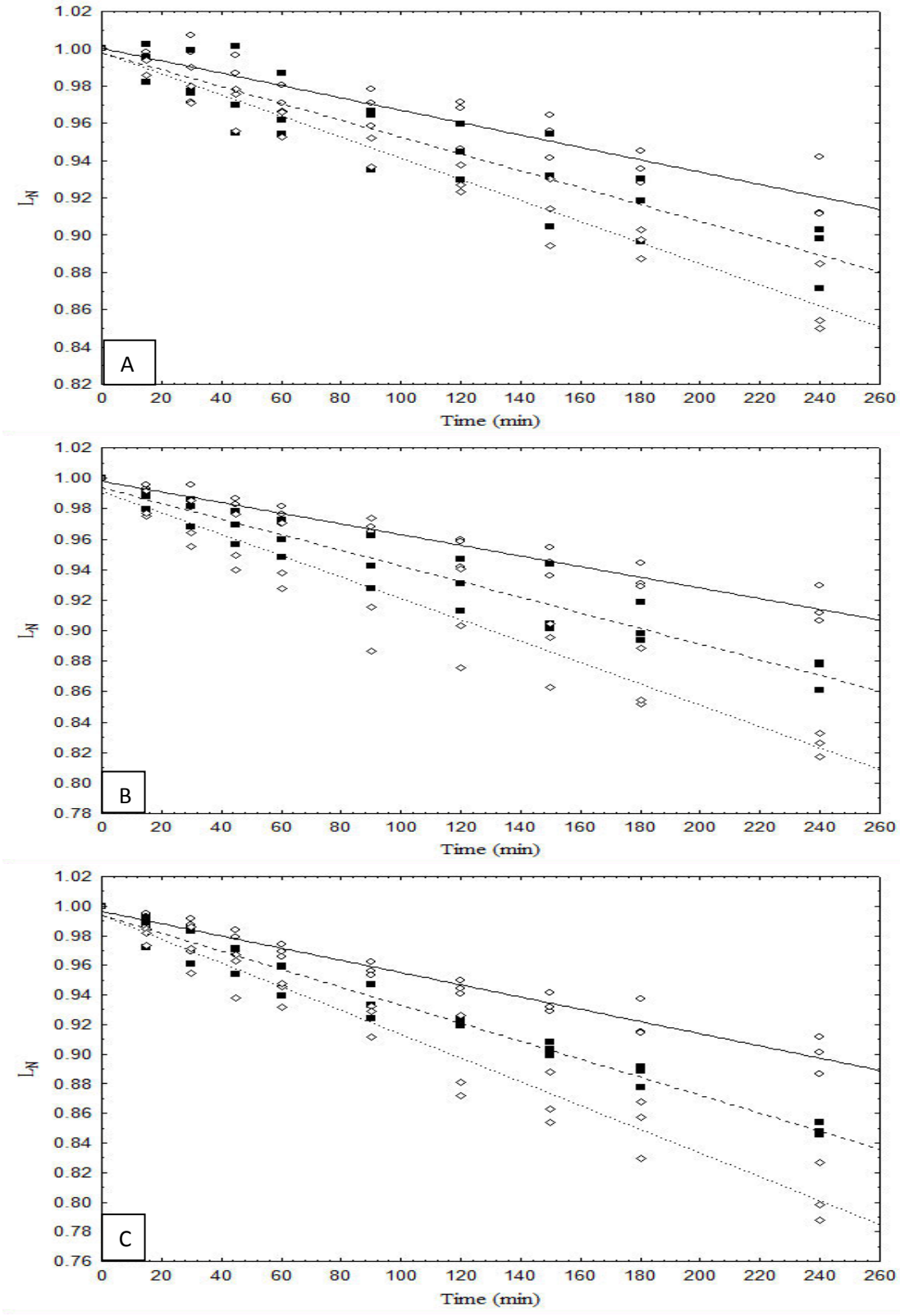


Figure 4.1. Plot of Normalized ‘‘L’’ Value Changes of Seedless Guava at (A) 30% (B) 40% (C) 50% Sucrose Solution, and Different Temperatures (○) 30 °C; (■) 40 °C; (◊) 50 °C (The lines represent Zero-order kinetic model)

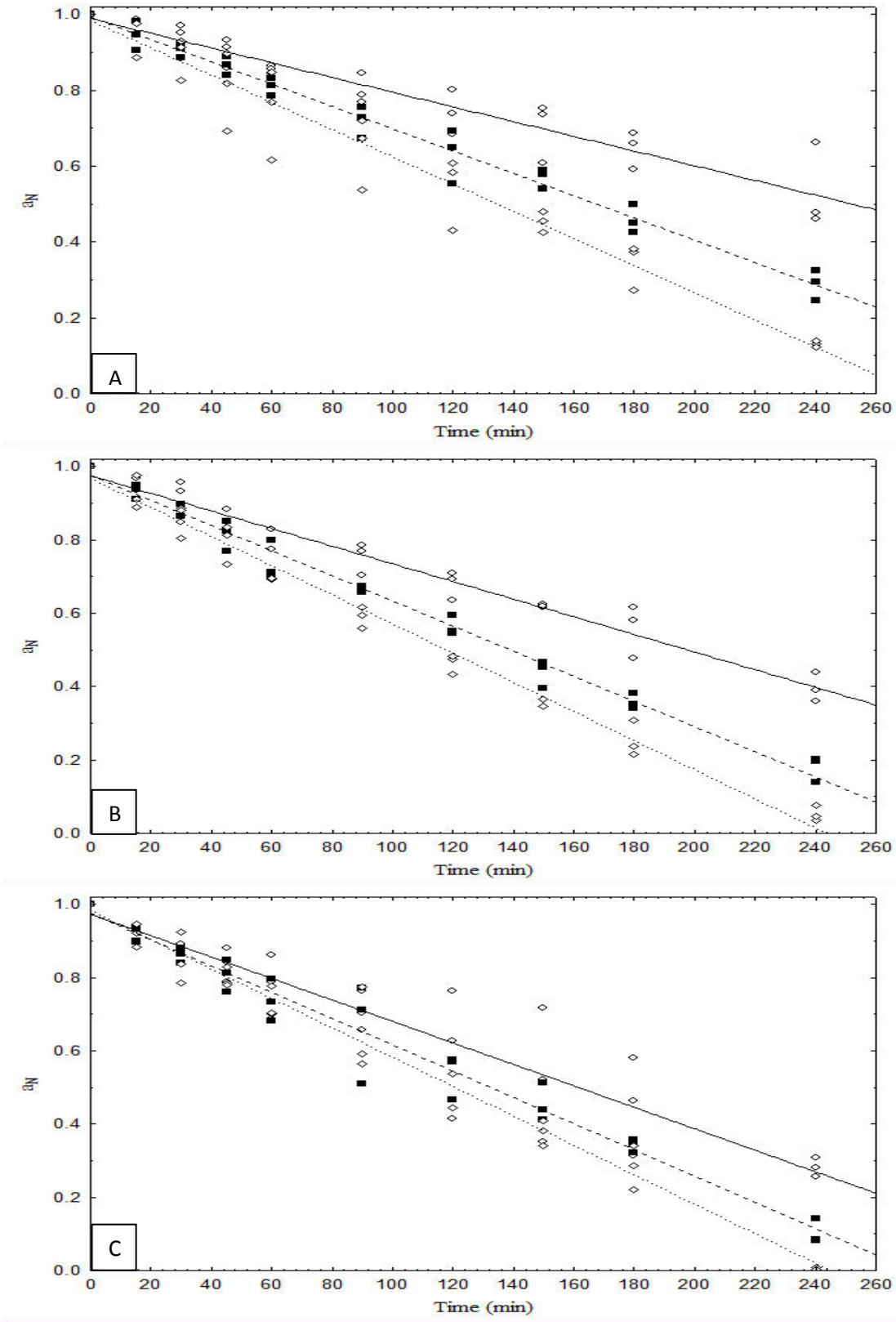


Figure 4.2. Plot of Normalized ''a'' Value Changes of Seedless Guava at (A) 30% (B) 40% (C) 50% Sucrose Solution, and Different Temperatures (○) 30 °C; (■) 40 °C; (◊) 50 °C (The lines represent Zero-order kinetic model)

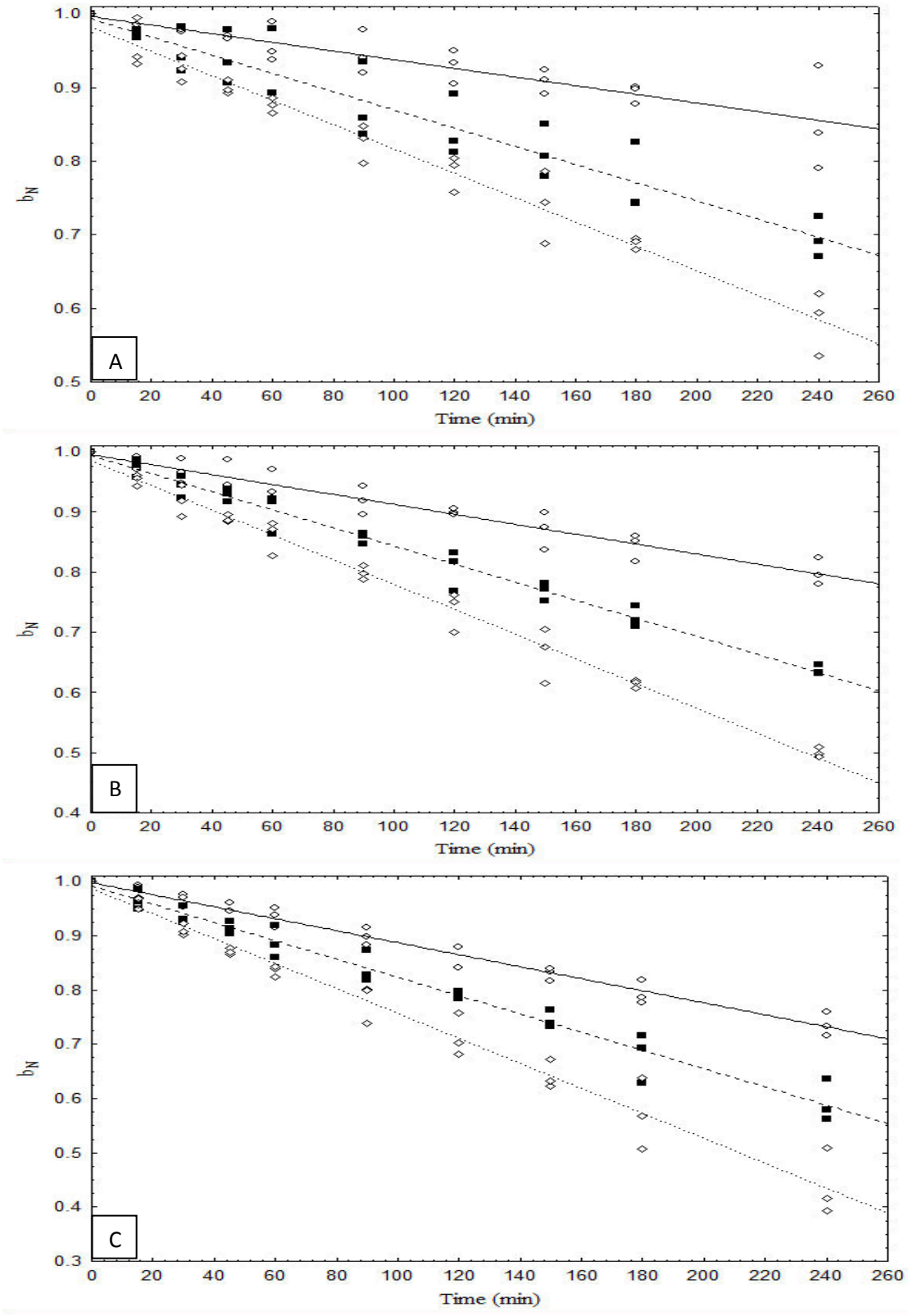


Figure 4.3. Plot of Normalized ‘‘b’’ Value Changes of Seedless Guava at (A) 30% (B) 40% (C) 50% Sucrose Solution, and Different Temperatures (●) 30 °C; (■) 40 °C; (◊) 50 °C (The lines represent Zero-order kinetic model)

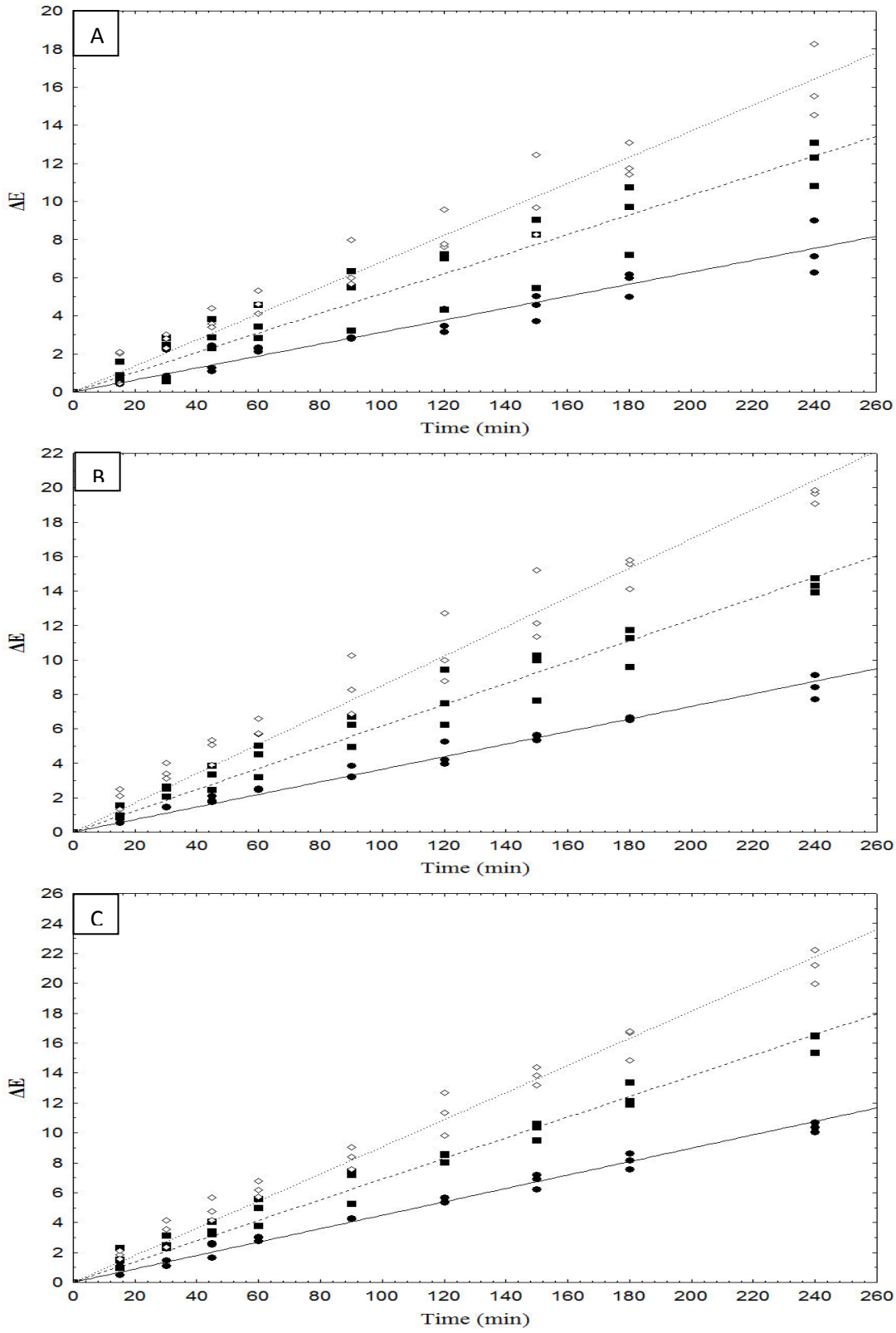


Figure 4.4. Plot of ΔE Value Changes of Seedless Guava at (A) 30% (B) 40% (C) 50% Sucrose Solution, and Different Temperatures (●) 30 °C; (■) 40 °C; (◊) 50 °C (The lines represent Zero-order kinetic model)

4.3.2 Kinetics of Color Parameters Changes during Osmotic Dehydration of Seedless Guava

To quantify the influence of the process variables on the color parameter changes during osmotic dehydration, it is useful to model the kinetics of color changes. There have been numerous studies carried out to apply kinetics models to the changes in quality of foods during processing and storage (Rocha *et al*., 1993; Avila and Silva, 1999; Elizalde *et al*., 2002). So far, no research has been found on the kinetics of color parameter changes during osmotic dehydration of seedless guava.

The experimental data of normalized ‘‘L’’, ‘‘a’’, and ‘‘b’’ well fitted to a zero order kinetics model. The R^2 values for each condition of osmotic dehydration were always greater than 0.88. Table 4.1 shows the rate constants (k) for changes in ‘‘L’’, ‘‘a’’, ‘‘b’’ and ΔE values. ANOVA results revealed the significant ($p \square 0.05$) influence of sucrose concentration and temperature on rate constants of color parameters changes. At constant sucrose concentration the rate constant for ‘‘L’’ value (k_L), ‘‘a’’ value (k_a), ‘‘b’’ value (k_b) and ΔE value ($k_{\Delta E}$) increased ($p < 0.05$) with increase in temperature. At constant temperature the k for ‘‘L’’ value, ‘‘a’’ value, ‘‘b’’ value and ΔE value increased ($p < 0.05$) with increase in sucrose concentration.

Table 4.1. Rate Constants for Kinetics of Color Changes of Seedless Guava during Osmotic Dehydration

Concentration (%w/w)	Temperature (ºC)	L/L_0		a/a_0		b/b_0		ΔE	
		k (min^{-1})	R^2	k (min^{-1})	R^2	k (min^{-1})	R^2	k (min^{-1})	R^2
30	30	0.00033±0.000025	0.92	0.0019±0.00011	0.95	0.00058±0.000057	0.88	0.031±0.00090	0.96
	40	0.00045±0.000036	0.92	0.0029±0.00007	0.99	0.0012±0.000077	0.94	0.051±0.00172	0.95
	50	0.00056±0.000023	0.97	0.0035±0.00015	0.97	0.0016±0.000059	0.98	0.068±0.00162	0.97
40	30	0.00035±0.000015	0.97	0.0024±0.00009	0.98	0.0008±0.000044	0.96	0.036±0.00055	0.99
	40	0.00051±0.000029	0.95	0.0034±0.00007	0.99	0.0015±0.000040	0.99	0.061±0.00133	0.98
	50	0.00070±0.000048	0.93	0.0039±0.00009	0.99	0.0020±0.000050	0.99	0.085±0.00182	0.98
50	30	0.00041±0.000017	0.97	0.0029±0.00020	0.93	0.0011±0.000037	0.98	0.044±0.00062	0.99
	40	0.00060±0.000019	0.98	0.0035±0.00012	0.98	0.0016±0.000054	0.98	0.069±0.00107	0.99
	50	0.00080±0.000035	0.97	0.0040±0.00011	0.98	0.0023±0.000075	0.98	0.090±0.00133	0.99

4.3.3 Modelling Temperature and Concentration Effects on the Rate Constant

The activation energy (E_a) obtained from the Arrhenius equation (Eq. 4.4) allowed quantifying the influence of temperature on rate constant values. The calculated values of E_a and natural logarithm of frequency factor ($\ln(k_0)$) are presented in Table 4.2. The greater temperature sensitivity of each rate constant was pointed out by the higher E_a value. It is apparent from Table 4.2 that the rate constant of ''b'' value was the greatest temperature sensitive term among other color parameters ($E_b > E_{\Delta E} > E_L > E_a$). Elevation of sucrose concentration led to increase in temperature sensitivity of ''L'' rate constant while the temperature sensitivity of rate constant for ''a'' and ''b'' values both decreased.

Table 4.2. Activation Energy and Frequency Factor for Color Change of Seedless Guava during Osmotic Dehydration at Different Sucrose Concentrations

Parameter	Concentration (% w/w)		
	30	40	50
L_N			
$\ln(k_0)$	0 55±0.06	3.25±0.34	3.02±0.64
E_a(KJ/mol)	21.56±0.02	28.2±0.01	27.25
R^2	0.993	0.999	0.996
a_N			
$\ln(k_0)$	3.67±1.94	1.87±0.17	0.63±0.39
E_a(KJ/mol)	24.97±0.04	19.85±0.40	13.11±0.01
R^2	0.960	0.949	0.993
b_N			
$\ln(k_0)$	9.07±3.67	7.77±2.81	5.10±0.10
E_a(KJ/mol)	41.49±0.88	37.44±0.80	30.03±0.22
R^2	0.949	0.963	0.999
ΔE			
$\ln(k_0)$	9.27±1.67	10.61±1.51	8.49±1.45
E_a(KJ/mol)	32.06±0.40	35.05±0.38	29.21±0.40
R^2	0.981	0.987	0.983

Subscript N presents Normalized value.

Multiple linear regression (Table 4.3) fitted data of rate constant for color parameter changes as a function of sucrose concentration (C) and absolute temperature (1/T). The models as fitted are:

$$\ln k_L = 1.70 + 0.0144\,(C) - 3089.8\,(1/T) \qquad (4.5)$$

$$\ln k_a = 1.14 + 0.0124\,(C) - 2323.1\,(1/T) \qquad (4.6)$$

$$\ln k_b = 6.46 + 0.0215\,(C) - 4369.4\,(1/T) \qquad (4.7)$$

$$\ln k_{\Delta E} = 8.84 + 0.0155\,(C) - 3862.6\,(1/T) \qquad (4.8)$$

Where k_L, k_a, k_b and $k_{\Delta E}$ are the rate constants for ‘‘L’’ ‘‘a’’, ‘‘b’’ and ΔE values, respectively. The models as fitted explained the 99.30%, 91.60%, 95.60% and 98.00% of the variability in k_L, k_a, k_b and $k_{\Delta E}$ at the 99% confidence level, respectively (Table 4.3). Rate constants for color parameters can be calculated using the above models for osmotically dehydrated seedless guava in sucrose solution in the range of 30-50% (w/w) and temperatures in the range of 30–50°C. In Eq. (4.5) - Eq.(4.8), the positive coefficients for sucrose concentration indicate that rate constants increase with increase in sucrose concentration. In Eq. (4.5) - Eq. (4.8) the negative coefficients for temperature indicate rate constants increase with increase in temperature.

Table 4.3. Multiple Linear Regression for k_L, k_a. k_b and $k_{\Delta E}$ as a Function of Sucrose Concentration (C) and Temperature (1/T)

Sources of variation	k_L		k_a		k_b		$k_{\Delta E}$	
	Estimate	Standard error	Estimate	Standard error	Estimate	Standard error	Estimate	Standard error
Constant	1.70	0.52	1.14	0. 40	6.45	1.36	8.84	0.77
C	0.014*	0.0016	0.012*	0.0033	0.021*	0.0043	0.015*	0.0024
1/T	-3089.75*	163.0018	-2323.09*	324.17	-4369.37*	424.24	-3862.59*	239.53
R^2	0.993		0.916		0.956		0.980	

ln k = A + B(C) + D(1/T)
*p -Value < 0.01.

According to Jackman and Stanley (1995), textural properties of fruits and vegetables are determined mainly by characteristics of the cell wall and middle lamella, and the turgor pressure. The typical force-time curve after compression of fresh seedless guava is shown in Appendix 1. From the curves, the maximum force, indicative of the hardness of the tissue; the initial slope of the curve, related to the rigidity or the elastic behavior of the tissue; and the area under the curve, related to the energy need for the penetration into the tissue were calculated.

Hardness, area under the curve and initial modulus average values of fresh seedless guava were 765.33±154.63 g, 13024.31±3939.87 g.sec, and 187.33±66.37 g/sec, respectively. The effects of process variables on hardness, initial slope and area under the curve of seedless guava during osmotic dehydration were investigated. An ANOVA reflects a significant ($p < 0.05$) decrease of the hardness, initial slope and area under the curve due to osmotic treatments. Table 4.4 shows the achieved mean values (±SD) of determined textural parameters in each treatment for osmotically dehydrated seedless guava. As expected, the hardness, initial slope and the area under the curve during osmotic treatments decreased with increase in concentration and temperature of osmotic solution. Those results are associated with the loss of turgor, the collapse of cell structure and the high level of alteration of the cell bonding zone which their levels relate to the intensity of dehydration process (Scanlon *et al*., 1996; Mauro *et al*., 2002; Chiralt and Talens, 2005). Similar finding were also reported by Chirlat and Talens (2005), katsiferis *et al*. (2008), Castelló *et*

pectin and separation of cells during process.

Since slope of the initial linear zone of the curves decreases after osmotic dehydration, consequently the apparent modulus of elasticity also decreases (Mayor *et al*., 2007). Previous studies conducted by Scanlon *et al*. (1996) and Chiralt *et al*. (2001) showed a decrease in the initial linear zone after osmotic dehydration of potato, mango, kiwifruit and strawberries.

Table 4.4. Normalized Values of Hardness, Area under the Curve and Initial Modulus after 240 min of osmotic Dehydration of Seedless Guava

Concentration (%w/w)	Temperature (°C)	Hardness (g)	Area under curve (g.sec)	Initial Modulus (g/sec)
30	30	0.87±0.01	0.90±0.02	0.81±0.03
	40	0.81±0.02	0.82±0.02	0.76±0.02
	50	0.74±0.01	0.77±0.02	0.70±0.02
40	30	0.81±0.02	0.84±0.03	0.63±0.02
	40	0.76±0.01	0.78±0.01	0.59±0.01
	50	0.72±0.02	0.72±0.02	0.52±0.01
50	30	0.78±0.03	0.77±0.01	0.60±0.02
	40	0.72±0.03	0.73±0.01	0.50±0.02
	50	0.68±0.02	0.70±0.02	0.45±0.04

All data were obtained by triplicate and expressed as value±SD.

4.3.5 Kinetics of Hardness Changes during Osmotic Dehydration of Seedless Guava

Hardness is the most imortant mechanical characteristic of dried fruits which influenced the acceptability of product by consumers (Konopacka and Plocharski,

vegetables followed a first-order kinetic model (Bourne, 1987; Rizvi and Tong, 1997). The variations of normalized hardness values with dehydration time as function of sucrose concentration and solution temperature are shown in Figure 4.5. The experimental data well fitted to a zero order kinetics model. The R^2 values from linear regression performed on each condition of osmotic dehydration were always greater than 0.93. Table 4.5 shows the rate constants (k) were determined from the slopes of normalized curves for changes of hardness values. Analysis of variance (ANOVA) of rate constants revealed the significant (p□0.05) influence of sucrose concentration and temperature on rate constants. An Arrhenius model was satisfactorily (R^2>0.96) fitted to experimental data for quantifying the influence of temperature on rate constant values of hardness degradation for the osmotically dehydrated seedless guava. Estimated activation energies for hardness were 31.25±2.13-17.41±3.18 $KJmol^{-1}$ at the studied range of sucrose concentration (Table 4.6).

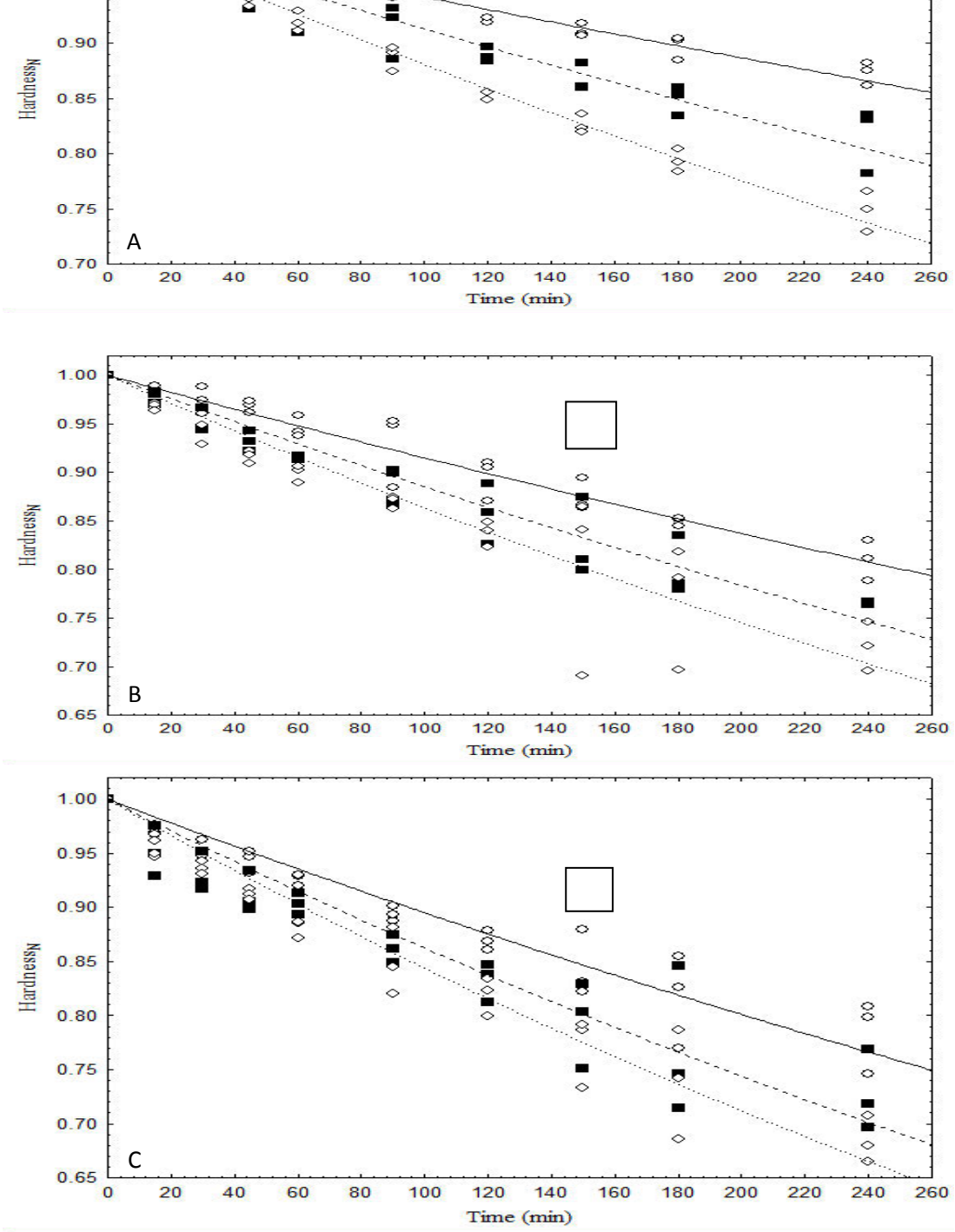


Figure 4.5. Plot of Hardness Value Changes of Seedless Guava at (A) 30% (B) 40% (C) 50% Sucrose Solution, and Different Temperatures(●) 30 °C; (■) 40 °C; (◊) 50 °C (The lines represents Zero-order kinetic model)

Concentration (%w/w)	Temperature (ºC)	Normalized Hardness	
		k (min^{-1})	R^2
30	30	0.00059±0.00001	0.97
	40	0.00091±0.00003	0.96
	50	0.00127±0.00001	0.99
40	30	0.00088±0.00002	0.96
	40	0.00122±0.00003	0.97
	50	0.00147±0.00006	0.93
50	30	0.00111±0.00003	0.95
	40	0.00148±0.00006	0.93
	50	0.00170±0.00004	0.97

Table 4.6. Activation Energy and Frequency Factor for Hardness Change of Seedless Guava during Osmotic Dehydration at Different Sucrose Concentrations

	Sucrose concentration (% w/w)		
Parameter	30	40	50
ln (k_0)	4.97±0.68	1.29±0.01	0.12±0.01
E_a(KJ/mol)	31.25±2.13	20.94±2.91	17.41±3.18
R^2	0.99	0.98	0.96

models are:

$$\ln k_{Hardness} = 1.19 + 0.0235\,(C) - 2791.25\,(1/T) \qquad (4.9)$$

The model as fitted explained 95.0% of the variability in rate of hardness degradation at the 99% confidence level (Table 4.7). With this model the rate constant of hardness degradation can be calculated when the seedless guava cubes are osmotic dehydrated in sucrose in the range of 30-50% (w/w) and temperatures in the range of 30–50 °C.

Table 4.7. Multiple Linear Regression for $k_{Hardness}$ as a Function of Sucrose Concentration (C) and Temperature (1/T)

	$k_{Hardness}$	
Sources of variation	Estimate	S.E.
Constant	1.19	0.07
C	0.0235*	0.0034
1/T	-2791.25*	333.5209
R^2	0.95	

$\ln k = A + B(C) + D(1/T)$
*p -Value < 0.001.

4.3.6 Influence of Process Variables on Vitamin C Retention of Seedless Guava

Vitamin C contents of seedless guava cubes were measured during osmotic dehydration at the studied range of sucrose concentration and temperature which

et al (2006) observed that 14% of vitamin C content of apricots was lost during osmotic dehydration. ANOVA results revealed insignificant ($p > 0.05$) effect of process variables including sucrose concentration, temperature and immersion time on vitamin C content of seedless guava. The kinetic of vitamin C retention was investigated to quantify the influence of the process variables on vitamin C content variation of seedless guava during osmotic dehydration. There have been numerous studies carried out to apply kinetics models to the changes of vitamin C during processing and storage (Blasco *et al*. 2004; Castro *et al*. 2004; Tiwari *et al*. 2009). So far, no research has been found on the kinetics of vitamin C degradation during osmotic dehydration of seedless guava. The experimental data of vitamin C content variation well fitted to a zero order kinetics model with R^2 values higher than 0.93. Table 4.9 shows the rate constants (k) for changes in vitamin C content values. ANOVA results revealed the insignificant ($p > 0.05$) influence of sucrose concentration and temperature on rate constants of vitamin C content changes.

Table 4.8. Vitamin C Retention after 240 min of Osmotic Dehydration of Seedless Guava

Concentration (%w/w)	Temperature (ºC)	Vitamin C retention (%)
30	30	97^{a}±0.002
	40	97^{a}±0.003
	50	96^{a}±0.005
40	30	97^{a}±0.002
	40	96^{a}±0.002
	50	96^{a}±0.001
50	30	97^{a}±0.001
	40	96^{a}±0.003
	50	95^{a}±0.004

All data were obtained at least by triplicate and expressed as value ± SD.

Concentration (%w/w)	Temperature (ºC)	k (min^{-1})	R^2
30	30	0.0001±0.000	0.96
	40	0.0001±0.000	0.93
	50	0.0002±0.000	0.94
40	30	0.0001±0.000	0.98
	40	0.0002±0.000	0.95
	50	0.0002±0.000	0.99
50	30	0.0001±0.000	0.94
	40	0.0002±0.000	0.99
	50	0.0002±0.000	0.98

4.4 Summary

The results of this study can be summarized as follows:

1. Sucrose concentration and temperature affect all the quality attributes evaluated for seedless guava cubes during osmotic dehydration significantly ($p < 0.05$).
2. In all cases, ‘‘L’’, ‘‘a’’, and ‘‘b’’ values decreased with an increase in sucrose concentration, temperature and therefore ΔE increased, suggesting that seedless guavas lose lightness, greenness and yellowness during osmotic dehydration.
3. The hardness, initial slope and the area under the curve decreased which resulted in softer and less elastic seedless guava after dehydration process.
4. The variation of color parameter changes as well as hardness of seedless guava followed zero order kinetic equation and an Arrhenius relationship for

6. Rate constant for ‘‘b’’ was the most temperature sensitive terms which followed by rate constant of ‘‘L’’ and ‘‘a’’terms.
7. Models were obtained for estimation of rate constants of quality parameter changes as a function of absolute temperature and sucrose concentration during osmotic dehydration by using the Multiple linear regression technique.

EFFECT OF THERMAL PRETREATMENTS ON OSMOTIC DEHYDRATION OF SEEDLESS GUAVA (*Psidium Guajava* L.)

5.1 Introduction

Fruits and vegetables can be dehydrated osmotically by immersion in a hypertonic solution which is characterized by removing large amounts of water through the cell membrane while uptaking solute is limited (Raoult-Wack, 1994). Bidwell (1974) attributed to the differential permeability of cell membranes. The cellular membrane exerts high resistance to transfer which lead to slow down the rate of mass transfer during osmotic dehydration (Erleo and Shubert, 2001). It has been demonstrated that the mass transfer largely controlled by the plasmalemma (Nobel, 1991); and cell membrane permeability strongly affects the dehydration rate (Zhiming and Le Maguer, 1997; Rastogi and Niranjan, 1998; Rastogi *et al*., 1999). Therefore, the damage of cell membranes can be advantageous for acceleration of mass transfer. Rastogi *et al*. (1999) pointed out that permeabilization of the cell membrane occurs as a result of pretreatments which may vary between partial to total permeabilization depending on the treatment. A number of pretreatment techniques such as blanching, partial vacuum (Corzo *et al*., 2007; Corrêa *et al*., 2010), high hydrostatic pressure (Rastogi and Niranjan, 1998; Rastogi *et al*., 2000) and pulsed electric fields (Ade-Omowaye *et al*., 2003; Amami *et al*., 2006) have shown capabilities to increase mass transfer rates during the osmotic process by affecting membrane integrity and functionality.

(Dell Valle *et al*., 1998; Moreno *et al*., 2000; Martinez-Monzo *et al*., 2001; Chafer *et al*., 2003). The hot water blanching ensures inactivation of enzymes involved in quality deterioration and facilitates mass transfer as a result of the changes promoted at structural level (Chafer *et al*., 2003; Gornicki and Kaleta, 2007; Doymaz, 2008).

Unfortunately, application of heat for food processing leads to quality losses due to the degradation of flavors, color, texture and loss of vitamins (Tijskens and Biekman, 2001; Song *et al*., 2003; Gonçalves *et al*., 2007; Liua and Scanlon, 2007; Koukounaras *et al*., 2008). This is the driving force for the growing interest in alternative methods able to reduce the intensity of the heat input of conventional technologies (Vercet *et al*., 2002). A large body of literature has focused on the development of power ultrasound uses in food industry (Piyasena *et al*., 2003; Zenker *et al*., 2003; Cheng *et al*., 2007). Cruz *et al*. (2008) suggested thermosonication as a good alternative to the heat treatment, since it is in favor of less severe heat blanching conditions. To date, no in-depth studies have been made on the effect of different blanching methods such as hot water and thermosonication on the kinetics of mass transfer during osmotic dehydration of seedless guava. The aim of this study was to investigate the effect of hot water blanching (traditional pretreatment) and thermosonicationon the kinetics of mass transfer and quality attributes (color and texture) during osmotic dehydration of seedless guava.

5.2.1 Sample Preparation

Sample preparation procedure used in the present chapter is similar to that as described in Chapter 3 (i.e. 3.2.1).

5.2.2 Pretreatments

The fresh cubes (2 cm^3) subjected to heat treatment in a circulating water bath (Memmert, WNE14. Memmert GmbH Co. KG, Germany) maintained at desired temperatures (±0.5°C). The studied temperatures were 80, 85, 90 and 95°C, with different times of exposure between 90 to 240 s. After preset times, the samples were removed from the water bath and placed immediately in cooled water (2–5°C) for 5 min in order to stop thermal inactivation instantaneously. The temperature of the water bath and cooled water was verified with a digital thermometer (Ellab CTD-85, Ellab, Denmark) and a thermocouple (1.2 mm needle diameter constantan type T). Each experiment was run in triplicate. An untreated sample was taken as control.

In second approach, the samples were processed with an ultrasonic processor (Sonics & Materials Inc., Model VC505, Danbury, CT, USA), set at 500W, 20 kHz and fitted with a 13 mm diameter titanium probe. Thermosonication was carried out at 25, 50 and 75 % (31, 62 and 93 µm, respectively) amplitude of ultrasonic wave. To avoid overheating during thermosonication, the temperature of water bath reduced to

Seedless guava cubes immersed in sucrose solution (fruit/syrup ratio1:2 w/w) with 8 ºBrix in order to limit the mass transfer during pretreatments. Non-blanched cubes of seedless guava with the same dimensions were used as control. Each experiment was run in triplicate.

5.2.2.1 Extraction of Crude Peroxidase

Treated samples were mixed with cold phosphate buffer in the proportion of 3:25 w/v based on preliminary experiments. Each sample was homogenized in an Ultra-Turrax (T25 Janke & Kunkel) for 1 min at 13,500 rpm under chilled condition (Cruz *et al*., 2006). The homogenate was filtered through filter paper (Whatman No.1) and then centrifuged at 6,000×g and 4 °C for 20 min with polypropylene tubes. The supernatants were kept on ice until the analysis (<5 min).

5.2.2.2 Determination of Crude Peroxidase Activity

Peroxidase activity was determined following the method of Morales-Blancas *et al*. (2002). For this purpose, aliquot of 0.12 mL of enzyme extract was added to 3.48 mL of substrate solution in a 10 mm path-length quartz cuvette. A 0.1 mL guaiacol (99.5%, BDH Chemicals Ltd.), 0.1 mL hydrogen peroxide (30%, Panreac), and 99.8 mL phosphate buffer (0.1 mol/L, pH6.5, Merck) was mixed daily to prepare

min to measure peroxidase activitiy. Enzyme activity was calculated from the slope of the initial linear portion of a plot of absorbance versus time. All the experiments were performed in triplicate. Residual enzyme activity is expressed as a fraction of initial activity (C_0):

$$\text{Residual enzyme activity} = C/C_0 \times 100 \quad (5.1)$$

Where C is enzyme activity after heating for time t.

5.2.2.3 Vitamin C Determination

Amount of vitamin C was measured in heated and thermosonicated samples achived t0o a 90% reduction in peroxidase activity. Vitamin C determination procedure used in this chapter is similar to that as described in Chapter 4 (i.e. 4.2.5).

5.2.3 Osmotic Dehydration Procedure

In order to study the effect of pretreatments on kinetics of mass transfer in seedless guava cubes, other process parameters such as sucrose concentration, temperature and immersion time were held constant at optimum conditions as determined in chapter 3 (30%w/w sucrose concentration, 33 °C solution temperature and immersion time of 180 min).

sample to syrup) and then placed in a circulating water bath (Memmert, WNE14. Memmert GmbH Co. KG, Germany) maintained at 33 ºC. When the immersion time was reached (15, 30, 45, 60, 120 and 180 min, respectively), samples were taken out from the beaker, gently rinsed under tap water to eliminate excess syrup from fruit surface, and slightly blotted with absorbent paper and analyzed. The osmotic dehydration procedure is similar for untreated samples (control). Each experiment was replicated thrice.

5.2.4 Analytical Determinations

Analytical determinations procedure used in this chapter is similar to that as described in Chapter 3 (i.e. 3.2.3).

5.2.5 Determination of Mass Transfer Kinetic Parameters

Kinetic determination procedure used in this chapter is similar to that as described in Chapter 3 (i.e. 3.2.4).

5.2.6 Mass Transfer Model

Mass transfer model used in this chapter is similar to that as described in Chapter 3 (i.e. 3.2.5).

Color measurement used in the present study is similar to that as described in Chapter 4 (i.e. 4.2.3).

5.2.8 Texture Analysis

Mechanical measurement used in the present study is similar to that as described in Chapter 4 (i.e. 4.2.4).

5.2.9 Determination of Membrane Damage or Conductivity

Conductivity of the solution after the desired osmotic dehydration interval was measured using a conductometer (model 30/10FT, Yellow Spring Instrument Co., Inc., USA) according to dell Valle *et al*. (1998). Two seedless guava cubes were placed in 100 ml distilled water at room temperature for 4 h, prior to measurement of the conductivity.

5.2.10 Experimental Design and Statistical Analysis

The experiments were arranged in compeletly randomized design (CRD) with three replications. The procedure of statistical analysis at $p < 0.05$ level, non linear regression for fitting database to Peleg model and determination the goodness of fit is similar to that as described in Chapter 3 (i.e. 3.2.6.1)

5.3.1 Inactivation of Peroxidase by Hot Water Blanching Treatment

Figure 5.1 shows the thermal inactivation curves of peroxidase at different temperatures ranging from 80 to 95 ºC for hot water blanching treatments. Inactivation of peroxidase in seedless guava significantly ($P<0.05$) affected by temperature and inactivation time. It is apparent that increase in temperature and length of treatment resulted in increase in amount of peroxidase inactivation. Linear patern ($R^2>0.99$) was obtained for all temperatures by plotting the residual peroxidase activity on a logarithmic scale versus heating time which proved that the heat-labile fraction of the enzyme inactivated rapidly during the first seconds of treatment (See Figure 5.1), so the observed kinetic would correspond to the inactivation of the heat-resistant fraction of peroxidase. This finding can be explained adequetly with the monophasic first order kinetic reaction (Aguero *et al*., 2008) and silimilar to those reported for peroxidase in potato or carrot (Anthon and Barrett, 2002), tomato juice and pumpkin (Anthon *et al*., 2002; Soysal and Soylemez, 2005; Gonçalves, *et al*., 2007).

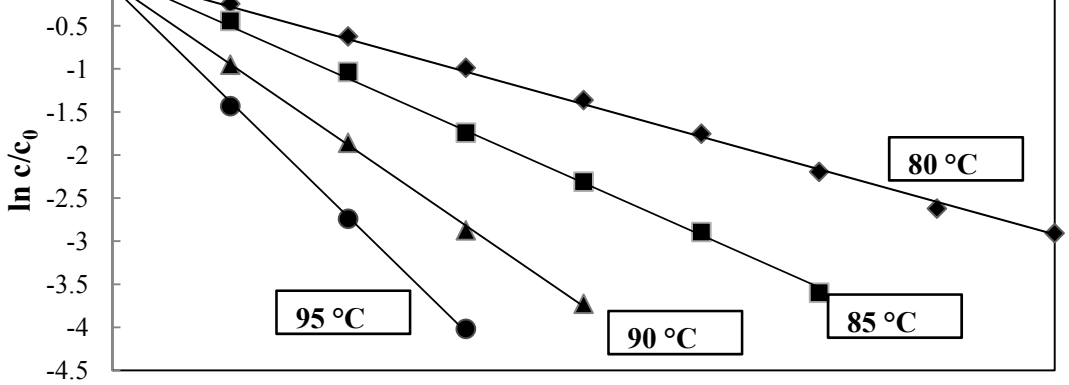


Figure 5.1. Thermal Inactivation of Seedless Guava Peroxidase. Remaining Peroxidase Activity versus Heating Time (Standard Deviation Bars are Smaller than the Symbol Size)

The inactivation rate constants (k) constants, estimated by linear regression analysis, varied from $1.1\times10^{-2}\pm0.0001$ s^{-1} at 80 ºC to $4.6\times10^{-2}\pm0.0004$ s^{-1} at 95 ºC, with the corresponding D values of 191.91 to 51.00 s, respectively (See Table 5.1). The activation energy for the thermal inactivation of the heat resistant fraction of seedless guava peroxidase was determined to be 96.39±4 $kJmol^{-1}$ (R^2 = 0.99).

Table 5.1. Reaction Rate Constants and Decimal Reduction Times of Peroxidase Inactivation in Seedless Guava by Thermal Inactivation

Temperature (°C)	k (s^{-1})[a]	R^2	D(Sec)	E_a($kJmol^{-1}$)[b]
80	$1.2\times10^{-2}\pm0.0001$	0.99	191.91	
85	$1.9\times10^{-2}\pm0.0003$	0.99	121.21	
90	$3.1\times10^{-2}\pm0.0002$	0.99	74.29	
95	$4.5\times10^{-2}\pm0.0004$	0.99	51.00	
				96.39±4

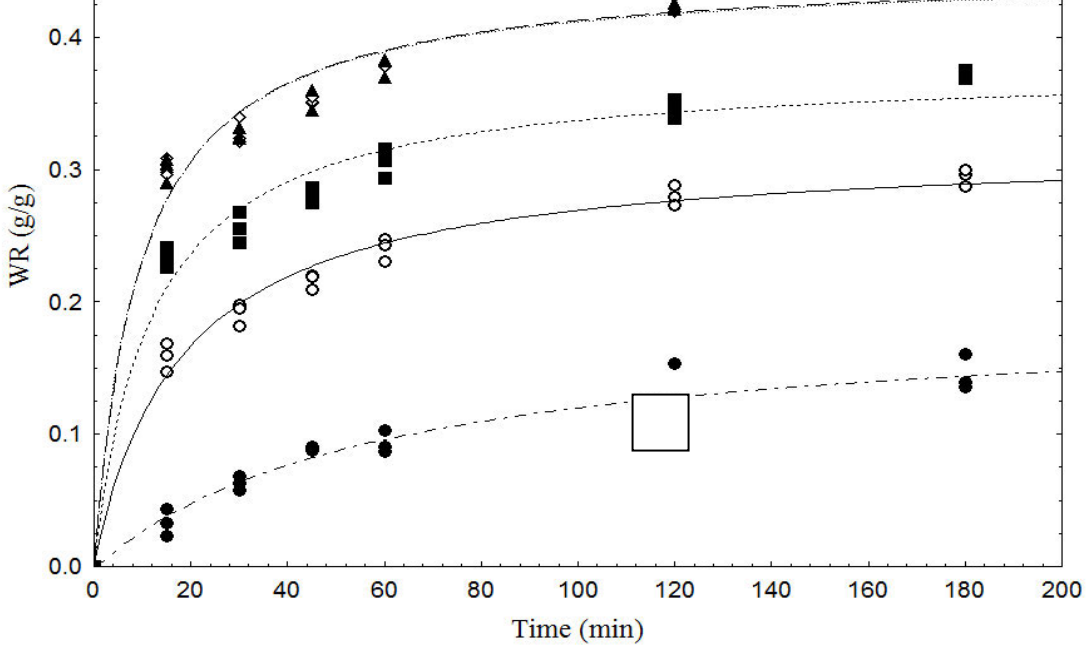
All measurements were replicated at least three times.
[a] k : Rate constants for thermal inactivation of seedless guava peroxidase.
[b] E_a: Activation energy of seedless guava peroxidase inactivation.

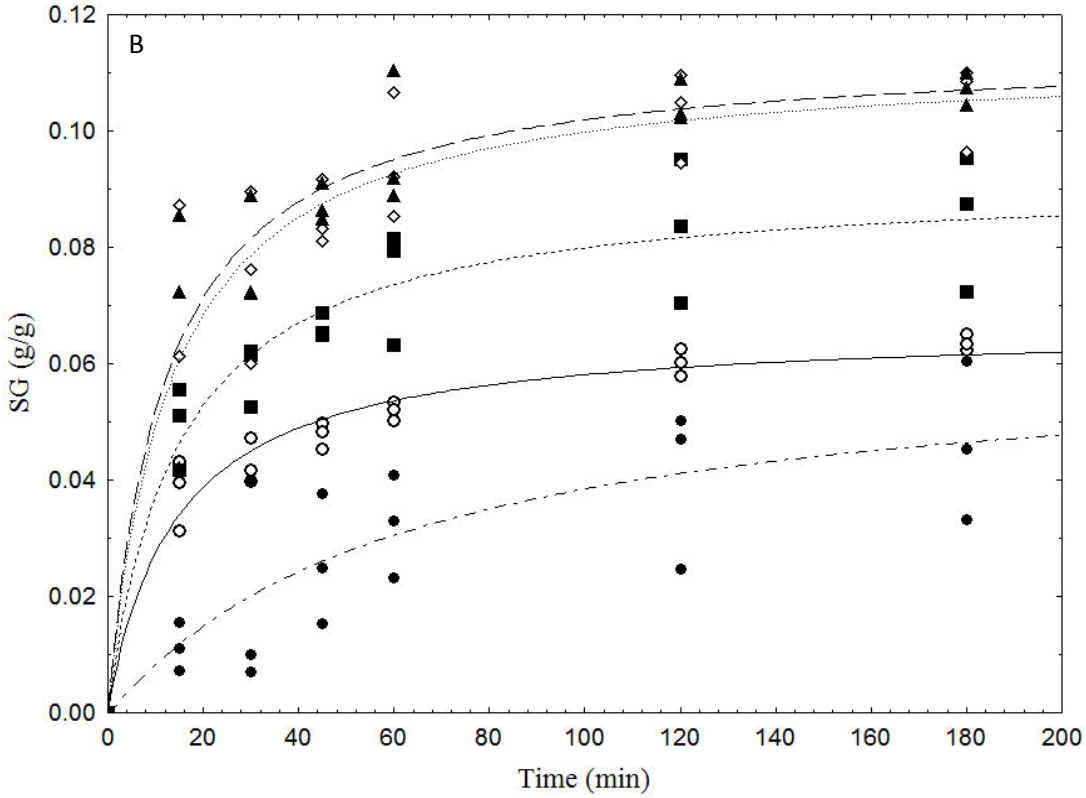
Effects of hot water blanching pretreatments at the temperature range of 80-95 °C on the kinetics of mass transfer during osmotic dehydration were investigated. The impacts of pretreatments on mass transfer are important especially in the beginning of osmotic dehydration (during the first hour). Osmotic dehydration coupled with hot water blanching at the temperature range of 80-90 °C lead to significant ($p < 0.05$) rapid enhancement of mass transfer. Results were compared with those obtained for osmotic dehydration of untreated seedless guavas (30%w/w sucrose concentration, 33 °C and 180 min of process duration) as a reference.

The solid gained by seedless guava during osmotic treatment is shown in Figure 5.2 (B). The pretreated seedless guava had higher SG than untreated samples. After 60 min of dehydration, SG of the untreated seedless guava was 0.04±0.03 g/g, whereas it reached 0.09±0.001g/g when osmotic dehydration combined with hot water blanching at 90 °C. Figure 5.2 (C) shows the amount of WL from seedless guava during osmotic treatment. For example, the WL reaches 0.48±0.008g/g after 60 min of osmotic dehydration for seedless guava previously subjected to hot water blanching (90 °C), while it is just 0.12±0.01g/g after the same duration of osmotic dehydration for the untreated seedless guava. As observed, the WR of pretreated seedless guava follows the same trend as SG and WL (See Figure 5.2 A). Furthermore, the amount of WL and SG increased during osmotic dehydration (Figure 5.2). These results can be explained by the capacity of heating to damage efficiently cellular membranes (cell decompartmentation), which affect the cell

WL and SG reduced with time of process indicating that the system getting closer to pseudoequilibrium condition due to the fact the osmotic driving force for mass transfer decreased with progression of time (Figure. 5.3).

The NMC of hot water pretreated as well as control seedless guava was experimentally determined during osmotic dehydration. Figure 5.2(D) shows typical plot of variations of NMC for seedless guava during the process. It is obvious that the moisture diffusion increased due to hot water pretreatment and also its temperature. This is attributed to the increase in cell permeability due to hot water blanching pretreatment. The present findings seem to be consistent with other researches which found increased in mass transfer rates during osmotic dehydration of thermally pretreated fruits and vegetables (Moreno *et al*., 2000; Rastogi *et al*., 2006).


WR (g/g)
0.0
0.1
0.2
0.3
0.4
0
20
40
60
80
100
120
140
160
180
200
Time (min)


B
SG (g/g)
0.00
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40
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140
160
180
200
Time (min)

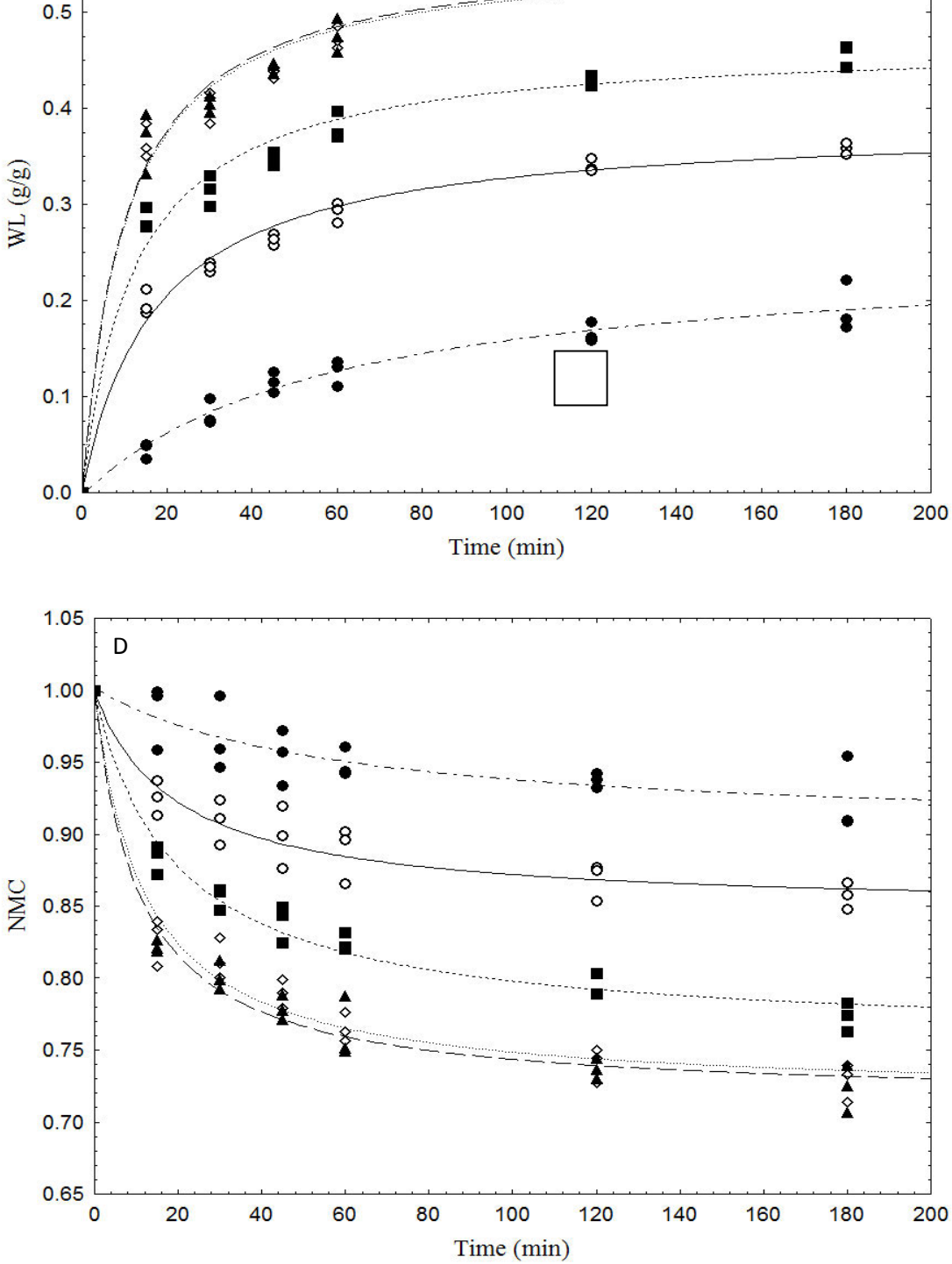
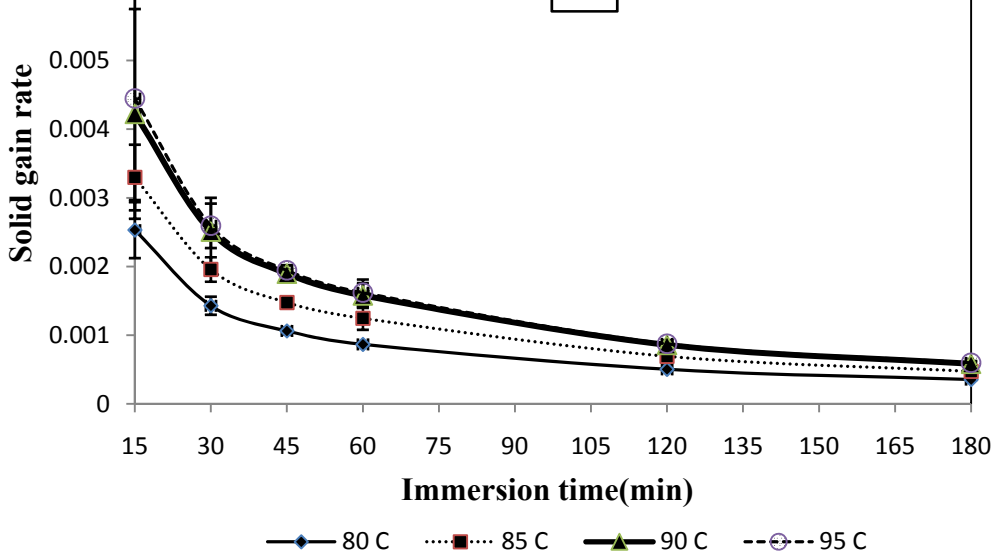


Figure 5.2. Effect of Hot Water Pretreatment at (●) Control at optimized condition , (○) 80 ºC, (■) 85 ºC, (◊) 90 ºC and (▲) 95 ºC on (A) WR; (B) SG; (C) WL and (D) NMC; (The lines represent the Peleg model)



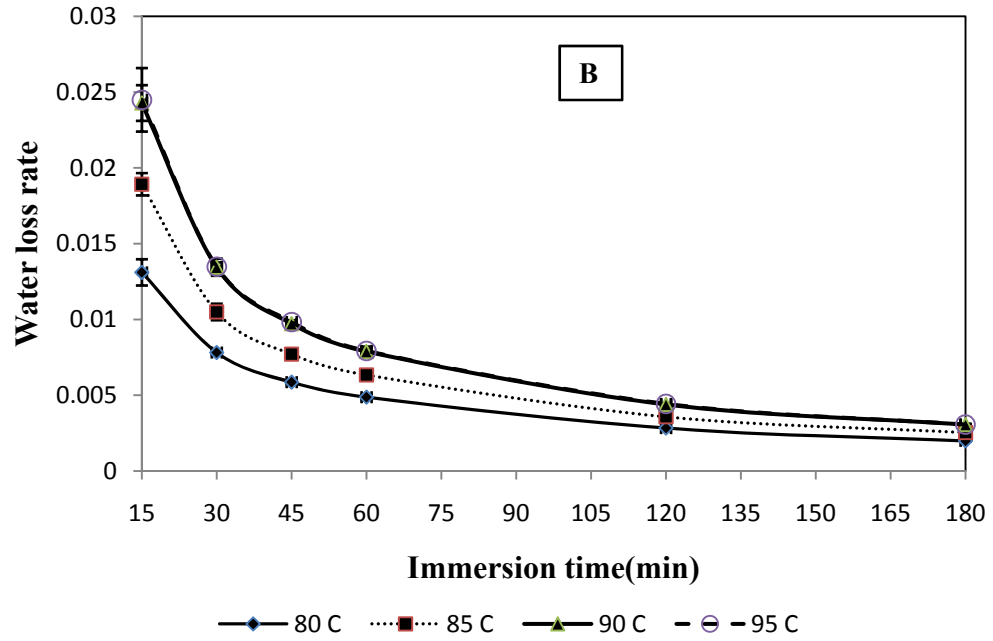


Figure 5.3. Effect of Pretreatment on Rate of Change of (A) SG and (B) WL (ds/dt) with Osmotic Dehydration Time

The obtained experimental values of WR, SG, WL and NMC after 180 min of osmotic dehydration are given in Table 5.2 for pretreated and untreated samples. It is

noticeable influence on WL/SG ratio. Thermal treatments cause deep changes in the fruit structure which is recognized by loss of cell membrane selectivity (Lebovka *et al*., 2002; Lebovka *et al*., 2004; Allali *et al*., 2009). For the untreated seedless guava, the WL/SG ratio amounted to 4.75 after 180 min of osmotic dehydration. After the hot water blanching at the temperature range of 80-90 °C, the ratio WL/SG slightly decreased from 5.83 to about 5.50, all the cells were probably destroyed. This behavior for pretreated samples is due to the fact that increase in amount of SG was more pronounced than WL. Thus, it could be concluded that increased WL had a lower impact on the WL/SG ratio than increased SG. This result is in agreement with those reported by (Kowalska *et al*., 2008; Allali *et al*., 2009). Several studies have revealed that physico-chemical properties of raw material, process variables and applied pretreatment before osmotic dehydration can influence the mass transfer selectivity value (Piotrowski *et al*., 2004; Arevalo-Pinedo and Murr, 2007; Escobar *et al*., 2007).

Peleg's model parameters (K_1 and K_2) for WR, SG, WL and NMC were obtained using non-linear estimation for different pretreatments and shown in Table 5.3. The curve fitting criteria and estimated parameters for Peleg's model for osmotic dehydration of seedless guava cubes pretreated with hot water blanching at temperatures of 80, 85, 90 and 95 °C are also summarized in Table 5.3. In all cases, the high values of R^2 (> 0.96) and the lowest values of chi-square (0.0001-0.0058), RMSE (0.0005-0.0050) and E (0.28-3.34%) display good correlations between the

temperature range of 80-95 °C. Lines representing the simulation obtained by the application of Peleg model to the experimental data were also plotted on Figure 5.2. It is apparent from this table that Peleg's model parameters (K_1 and K_2) for all the kinetic terms of mass transfer changed depending on temperature of hot water pretreatment before osmotic dehydration of seedless guava. Previous studies have reported the good performance of Peleg's kinetic model for estimating the rate of mass transfer and equilibrium mass content of potatoes, pumpkin and banana (Khin *et al*., 2006; Kowalska *et al*., 2008; Mercali *et al*., 2010). The equilibrium contents were estimated and the results are presented in Table 5.4. Figure 5.4 compares experimental and equilibrium predicted mass transfer terms values by the Peleg models for different treatments. The good linear correlation was found between the experimental and predicted values with determination coefficient (R^2) ranging from 0.98 to 0.99.

Table 5.2. Values of WR, SG, WL and WL/SG after 180 min of Osmotic Dehydration of Pretreated (Hot Water Blanching) and Untreated Seedless Guava

Temperature of Pretreatment(ºC)	WR (g/g)	SG (g/g)	WL (g/g)	NMC (%)	WL/SG
Untreated	0.14±0.010[a]	0.04±0.010[a]	0.19±0.020[a]	0.92±0.020[a]	4.75
80	0.29±0.006[b]	0.06±0.001[b]	0.35±0.005[b]	0.85±0.008[b]	5.83
85	0.37±0.003[c]	0.08±0.011[c]	0.45±0.012[c]	0.77±0.010[c]	5.62
90	0.44±0.005[d]	0.10±0.007[d]	0.55±0.005[d]	0.72±0.013[d]	5.50
95	0.44±0.007[d]	0.10±0.002[d]	0.55±0.009[d]	0.72±0.016[d]	5.50

Values of WR, SG, WL and NMC were obtained by triplicate and expressed as value±SD.
Different superscripts at each column denote significant difference ($p<0.05$).

Table 5.3. Effect of Hot Water Pretreatment on Peleg's Parameters Constants and Goodness of Fit

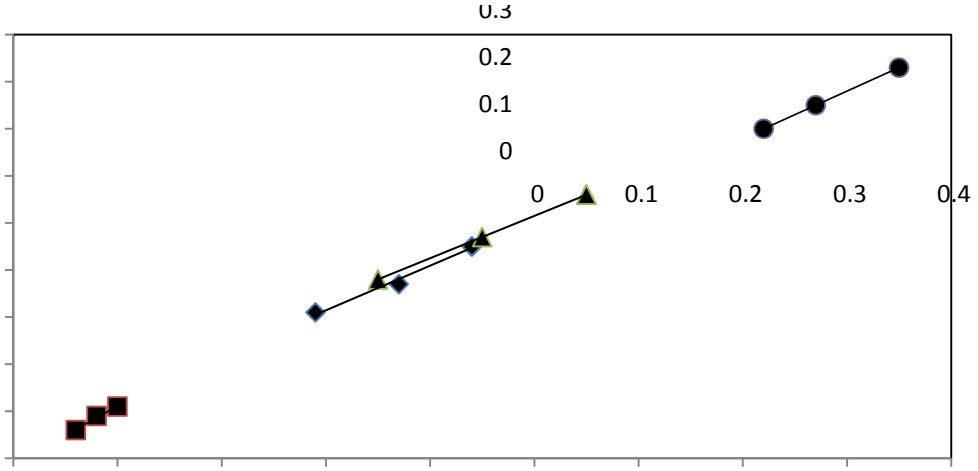
		Treatment				
		OD	80 °C+OD	85 °C+OD	90 °C+OD	95 °C+OD
WR	K_1	305.60±52.85	58.15±5.13	32.18±4.00	21.11±2.39	21.26± 2.39
	K_2	5.18±0.37	3.16±0.08	2.66±0.09	2.22±0.06	2.22±0.06
	R^2	0.97	0.99	0.99	0.99	0.99
	Chi-square	0.0012	0.0023	0.0055	0.0050	0.0048
	RMSE	0.0010	0.0022	0.0042	0.0046	0.0044
	E (%)	3.03	2.44	3.34	2.98	2.96
SG	K_1	992.61±194.18	219.78±32.49	162.72±35.78	116.02±30.08	105.90±24.05
	K_2	15.80±3.35	15.17±0.65	10.94±0.70	8.88±0.65	8.78±0.55
	R^2	0.84	0.98	0.96	0.95	0.96
	Chi-square	0.0002	0.0008	0.0004	0.0004	0.0004
	RMSE	0.0002	0.0006	0.0005	0.0006	0.0006
	E (%)	1.96	3.20	2.06	1.69	1.78
WL	K_1	233.76±35.61	46.24±4.05	27.04±3.14	18.10±1.90	17.84±2.02
	K_2	3.90±0.24	2.61±0.06	2.14±0.07	1.78±0.04	1.77±0.05
	R^2	0.98	0.99	0.99	0.99	0.99
	Chi-square	0.001	0.0031	0.0058	0.0050	0.0049
	RMSE	0.001	0.0029	0.0047	0.0050	0.0050
	E (%)	2.41	2.59	3.08	2.65	2.65
NMC	K_1	578.04±185.73	129.21±30.32	82.80±9.67	43.32±6.27	38.59±5.04
	K_2	9.99±2.01	6.60±0.48	4.17±0.15	3.56±0.14	3.53±0.12
	R^2	0.84	0.96	0.98	0.98	0.98
	Chi-square	4.2E-05	0.0001	0.0003	0.0008	0.0005
	RMSE	0.0007	0.0012	0.0019	0.0028	0.0022
	E (%)	0.15	0.28	0.51	0.73	0.63

OD osmotic dehydration process, K_1 Peleg rate constant (min g/g^{-1}), K_2 Peleg capacity constant $((g/g)^{-1})$

Table 5.4. Equilibrium WR, SG, WL and NMC Predicted using Peleg Model for Hot Water Pretreated Osmo-dehydrated Seedless Guava

Temperature of Pretreatment(ºC)	Equilibrium WR (g/g)	Equilibrium SG (g/g)	Equilibrium WL (g/g)	Equilibrium NMC (%)
Untreated	0.19±0.005	0.06±0.005	0.25±0.006	089±0.008
80	0.31±0.006	0.06±0.002	0.38±0.007	0.83±0.007
85	0.37±0.009	0.09±0.004	0.47±0.011	0.75±0.006
90	0.45±0.007	0.11±0.006	0.56±0.005	0.70±0.010
95	0.45±0.010	0.11±0.005	0.56±0.012	0.70±0.007

All data were obtained by triplicate and expressed as value±SD.



the Experimental and Model
, SG, WL and NMC of Hot water
Guava

osonication Blanching

The curves for inactivation of seedless guava peroxidase using thermosonication are presented in Figure 5.5 (A-D). The impact of the ultrasonic wave's intensity on the residual activity was significant ($P < 0.05$) for 50 and 75% level of ultrasonic wave's amplitude at the studied range of the temperature (80-95 °C). In all the experiments the peroxidase inactivation due to thermosonication treatment follows first order kinetics according to data reported in the literature (Lopez and Burgos, 1995; Raviyan *et al*., 2005). The estimated first order kinetic parameter, as well as decimal reduction times at different levels of ultrasonic wave's amplitude in the studied range of temperature is presented in Table 5.5. If the kinetic parameters obtained for

ultrasonic wave's amplitude at 90 ºC) compared to the thermal inactivation rate at the same temperature. De Gennaro *et al*. (1999) demonstrated that the inactivating capability of thermosonication in addition to ultrasound wave power depends on ultrasound intensity.

Damaging the integrity of the enzyme protein structure resulting in reduced enzyme activity which could be described by several mechanical and sonochemical processes, alone or in combination, such as purely thermal, due to the enormous temperatures achieved during cavitation; generation of free radicals by water sonolysis, and mechanical forces (shear forces) created by micro-streaming and shock waves (Price, 1992; Suslick, 1988).

Vercet *et al*. (1998) demonstrated that at low temperatures which the ultrasonic energy input is higher the production of hydroxyl radicals by ultrasound is favored. According to Figure 5.5 A, it was observed that there was not a faster inactivation of peroxidase at lower treatment temperature which could be concluded that the inactivation of peroxidase is probably not mediated by free radicals, but by the mechanical effects. Therefore, the thermosonication seems to be a good alternative to the traditional treatment in order to inactivate peroxidase enzyme at the studied range of temperature. Attending to an adequate peroxidase inactivation (90%), immersion time in hot water and thermosonication treatments were selected according to D-values at each studied temperature and used as a reference for hot

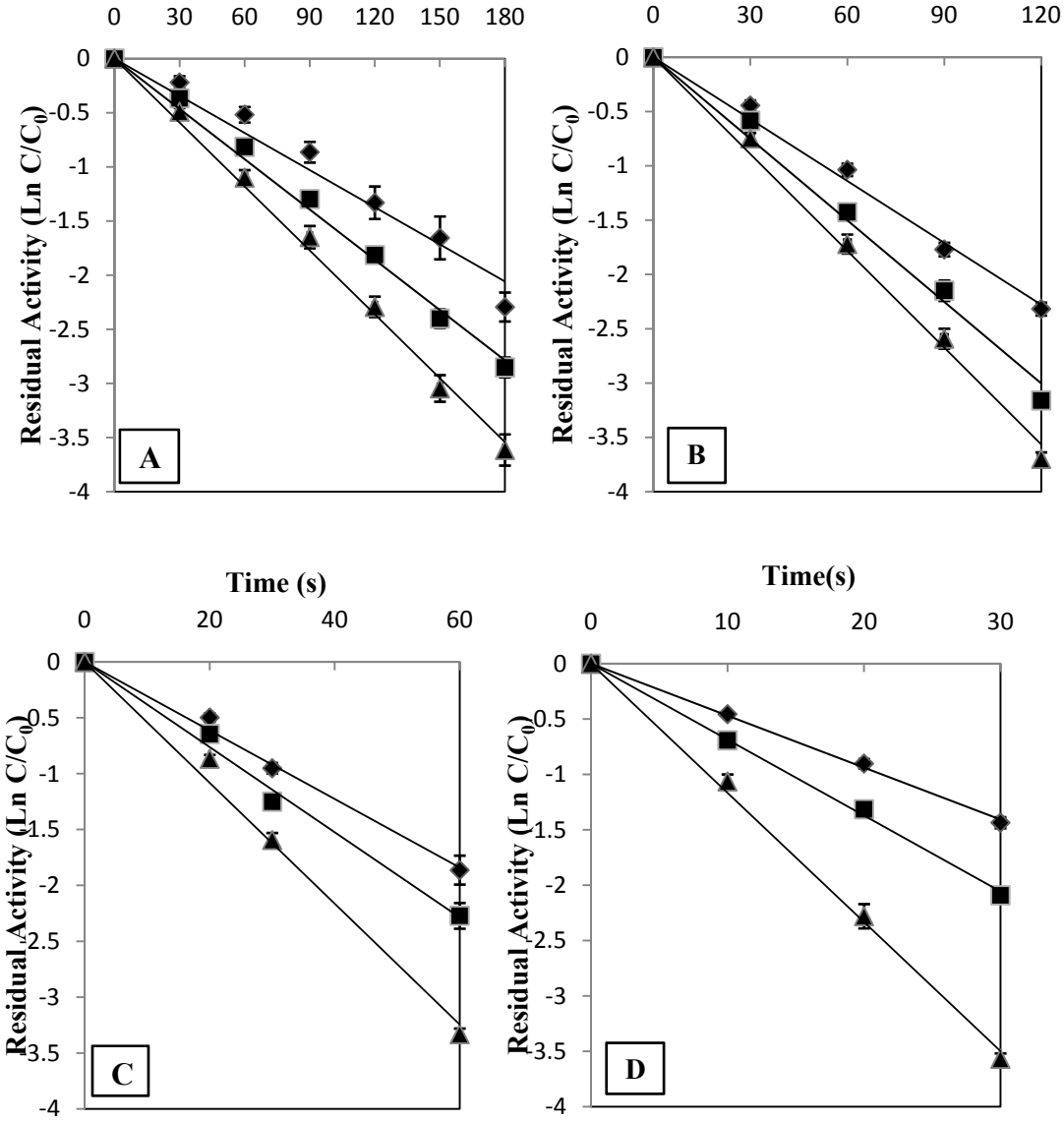


Figure 5.5. Thermal and Thermosonication inactivation of peroxidase in seedless guava. Remaining Peroxidase Activity versus Heating Time.(A) 80 °C; (B) 85 °C; (C) 90 °C and (D) 95 °C, Thermal Treatment(♦); Thermosonication at 50% (■) and 75% Ultrasonic Wave Amplitude (▲)

Temperature (°C)	Radiation (%)	k (s^{-1})[a]	R^2	D-value(Sec)
80	0	$1.2\times10^{-2}\pm0.0001$	0.98	191.91
	25	$1.1\times10^{-2}\pm0.0003$	0.98	209.36
	50	$1.6\times10^{-2}\pm0.0001$	0.99	143.93
	75	$2.0\times10^{-2}\pm0.0002$	0.99	115.14
85	0	$1.9\times10^{-2}\pm0.0003$	0.99	121.21
	25	$2.0\times10^{-2}\pm0.0001$	0.97	115.15
	50	$2.5\times10^{-2}\pm0.0005$	0.99	92.12
	75	$3.0\times10^{-2}\pm0.0004$	0.99	76.76
90	0	$3.1\times10^{-2}\pm0.0002$	0.99	74.29
	25	$3.1\times10^{-2}\pm0.0005$	0.99	73.81
	50	$3.8\times10^{-2}\pm0.0008$	0.99	60.60
	75	$5.5\times10^{-2}\pm0.0010$	0.99	41.87

All measurements were replicated at least three times.
[a] k: rate constants for inactivation of seedless guava peroxidase.

5.3.4 Vitamin C Retention

Although guava is a rich source of vitamin C, its level is subject to wide variations because of geographical location, horticultural practices, season and cultivar (Wilson 1980). The vitamin C content in fresh seedless guava was 122.31±3.60 mg/100 g of edible flesh. Vitamin C retention in seedless guava cubes after blanching treatments using time-temperature combinations equivalent to 90% of peroxidase inactivation is presented in Table 5.6. ANOVA results showed that differences in vitamin C retention among all treated samples were always significant ($P < 0.05$). In addition,

temperature (Lee and Kader, 2000; Oboh, 2005). As a result, the temperature of blanching would have inactivated the vitamin C in the seedless guava, while the water would have wash away the vitamin C during the course of blanching. It is apparent from Table 5.6 that samples treated with thermosonication showed the highest vitamin C contents due to the elimination of dissolved oxygen during cavitation that is essential for vitamin C degradation (Walkling-Ribeiro *et al*., 2007; Mason, 1991).

Table 5.6. Effect of Heat and Thermosonication Treatments on Retention of Vitamin C in Seedless Guava

Temperature (ºC)	Vitamin C retention (%)		
	Heat	TS 50%	TS 75%
80	49.46±2.70[a]	62.93±1.30[a]	71.60±2.01[a]
85	59.05±1.40[b]	70.32±2.30[b]	75.72±1.90[b]
90	61.17±2.80[c]	71.60±2.30[b]	80.36±1.50[c]

All data were obtained by triplicate and expressed as value±SD.
TS, thermosonication process.
Different superscripts at each column denote significant difference ($p<0.05$).

5.3.5 Effect of Thermosonication Pretreatment on Kinetics of Mass Transfer during Osmotic Dehydration

Attending to high retention of vitamin C at 90 °C, this temperature was used in combination with ultrasonic waves at the amplitude range of 25-75% to study the

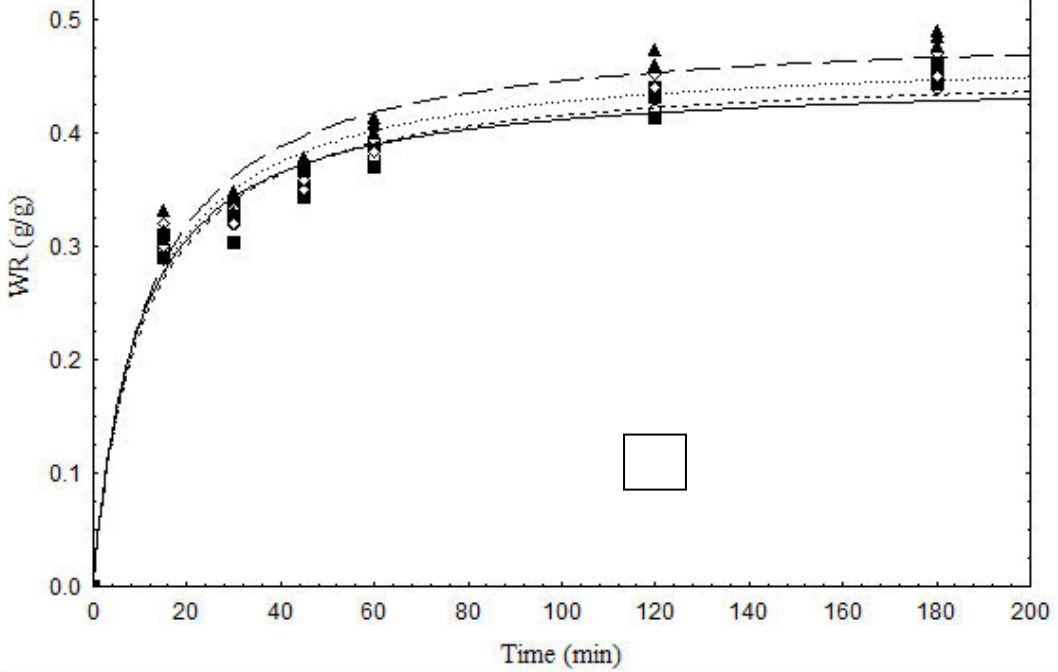
multiple comparisons test at 95% confidence level to distinguish the significant levels among the studied range of ultrasonic wave's amplitude. The ANOVA result indicated that 50 and 75% ultrasonic wave's amplitude had significant effect on WR and WL. The studied range of ultrasonic wave's amplitude didn't have significant effect on SG, whereas this range of ultrasonic wave's amplitude had significant effect on NMC during osmotic dehydration.

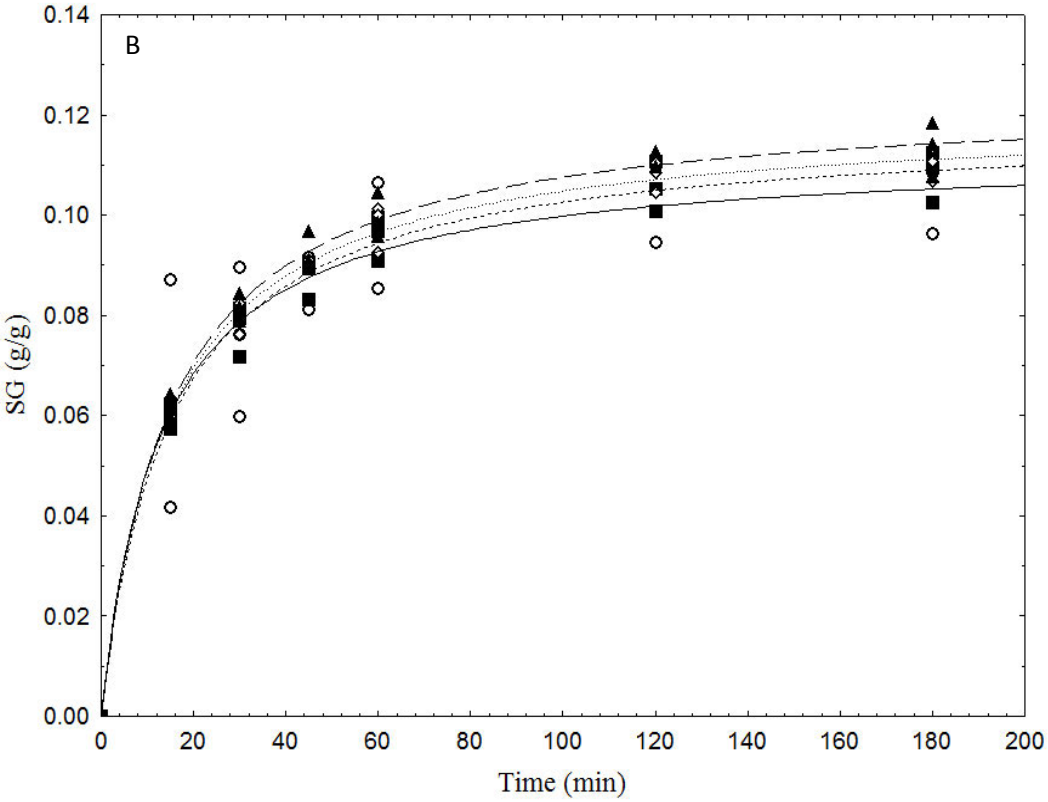
Figure 5.6 (A) shows the experimental data of WR obtained for pretreated seedless guava cubes using thermosonication at different level of amplitude (25-75 %) at 90 °C temperature. As it was expected the WR increased with time of osmotic process mainly to 60 min dependently on the kind pretreatment applied. Figure 5.6 (B) shows the influence of thermosonication pretreatment on SG of seedless guava during osmotic dehydration. SG by seedless guava during osmotic dehydration did not depend on the kind of pretreatment. After the first 60 min dehydration in sucrose solution, the penetration of osmoactive substance was obtained at about 0.094 g/g and 0.099 g/g for hot water and thermosonically pretreated seedless guava, respectively. After 180 min of the process, these values became 0.10 g/g in hot water blanching and 0.11 g/g for thermosonication at 75% of ultrasonic wave's amplitude (Table 5.7).

The WL parameter increased with operation time dependently on the kind of pretreatment. The most significant changes of WL were observed in the case of

sucrose solution WL from seedless guava amounted to 0.47 g/g for hot water (90 °C) and 0.50 g/g for thermosonically blanched samples at 75% of ultrasonic wave's amplitude.

In Figure 5.6 (D) there is a clear trend of decreasing in NMC of seedless guava during osmotic dehydration which depended on the kind of pretreatment applied. Reduction of moisture content during 60 min of osmotic dehydration was 3 and 8% higher in thermosonically treated seedless guava at the amplitude range of 25 and 75%, respectively. The smallest moisture content after osmotic dehydration in sucrose solution (Table 5.7) was observed in thermosonically pretreated seedless guava. The effectiveness of the osmotic dehydration process WL/SG ratio was calculated and reflected in Table 5.7. Applying thermosonication pretreatment before osmotic dehydration of seedless guava leads to limit WL/SG ratio. The thermosonicated sample showed the highest WR, WL and the lowest NMC. These results can be associated with extensive damage of cellular tissue with substantial decrease of the resistance to mass transfer caused by thermosonication blanching. The lower resistance to mass transfer is reflected by the faster decrease of moisture content and increase in solid content of the pretreated samples (Rastogi *et al*., 1999; Mavroudis, 2003). Therefore, slightly higher efficiency of thermosonication seems to be observed when applied before the osmotic dehydration process.


0.5
0.4
0.3
0.2
0.1
0.0
WR (g/g)
0
20
40
60
80
100
120
140
160
180
200
Time (min)


B
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
SG (g/g)
0
20
40
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80
100
120
140
160
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200
Time (min)

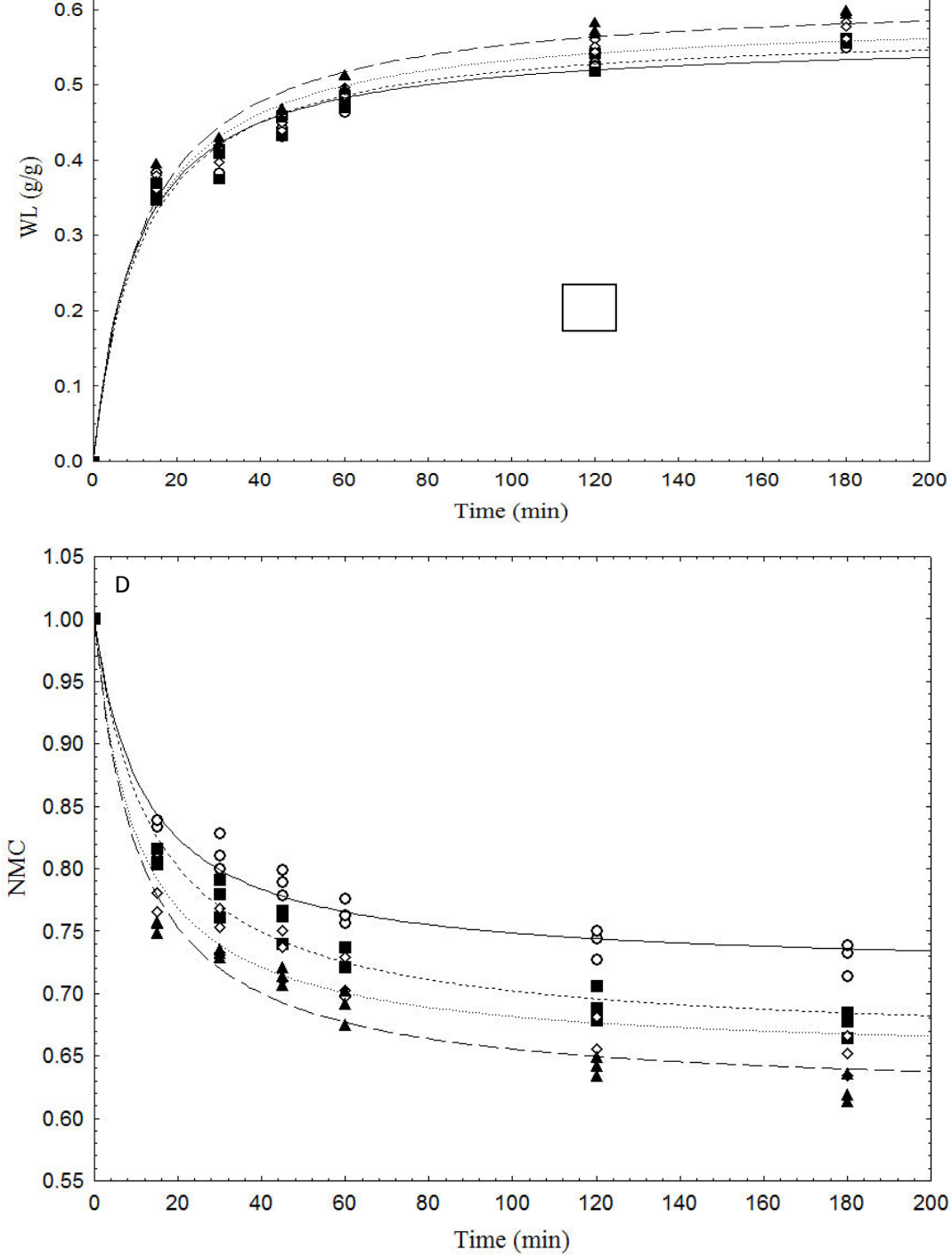


Figure 5.6. Effect of Thermosonication at (○) 0, (■) 25, (◊) 50 and (▲) 75% of Amplitude on (A) WR, (B) SG, (C) WL and (D) NMC (Lines represent Peleg model)

Table 5.7. Values of WR, SG, WL and WL/SG after 180 min of Osmotic Dehydration of Thermosonically Pretreated Seedless Guava

Type of Pretreatment	WR (g/g)	SG (g/g)	WL (g/g)	NMC (%)	WL/SG
Hot water (90 °C)	0.44±0.005[a]	0.10±0.007[a]	0.55±0.005[a]	0.72±0.013[a]	5.50
TS1	0.45±0.008[a]	0.10±0.005[a]	0.55±0.003[a]	0.67±0.010[b]	5.50
TS2	0.46±0.011[b]	0.11±0.003[a]	0.57±0.011[b]	0.65±0.016[c]	5.18
TS3	0.48±0.007[c]	0.11±0.005[a]	0.59±0.002[c]	0.62±0.011[d]	5.36

Values of WR, SG, WL and NMC were obtained by triplicate and expressed as value±SD.
TS1 Thermosonication at 25 % of amplitude, TS2 Thermosonication at 50 % of amplitude and TS2 Thermosonication at 75% of amplitude.
The column superscripts with same letter did not show significant difference ($p > 0.05$).

Mass transfer kinetic was calculated by employing the Peleg's model to WR, SG, WL and NMC for thermosonication pretreatment. A satisfactory correlation of Peleg model was found, and the corresponding values of K_1 and K_2 coefficients are presented in Tables 5.8. Lines representing the simulation obtained by the application of the Peleg model to the experimental data were also plotted in Figure 5.6. It is apparent from this table that Peleg's model parameters (K_1 and K_2) for all the kinetic terms of mass transfer changed independently on the kind of applied pretreatment before osmotic dehydration. The equilibrium contents were estimated and the results are presented in Table 5.9. Figure 5.7 compares experimental and Equilibrium predicted mass transfer terms values by the Peleg models for different treatments. The good linear correlation between the experimental and predicted values with the determination coefficient (R^2) ranging from 0.95 to 0.99 indicates the efficiency of Peleg equation to predict the kinetics of mass transfer during osmotic dehydration of thermally pretreated seedless guava.

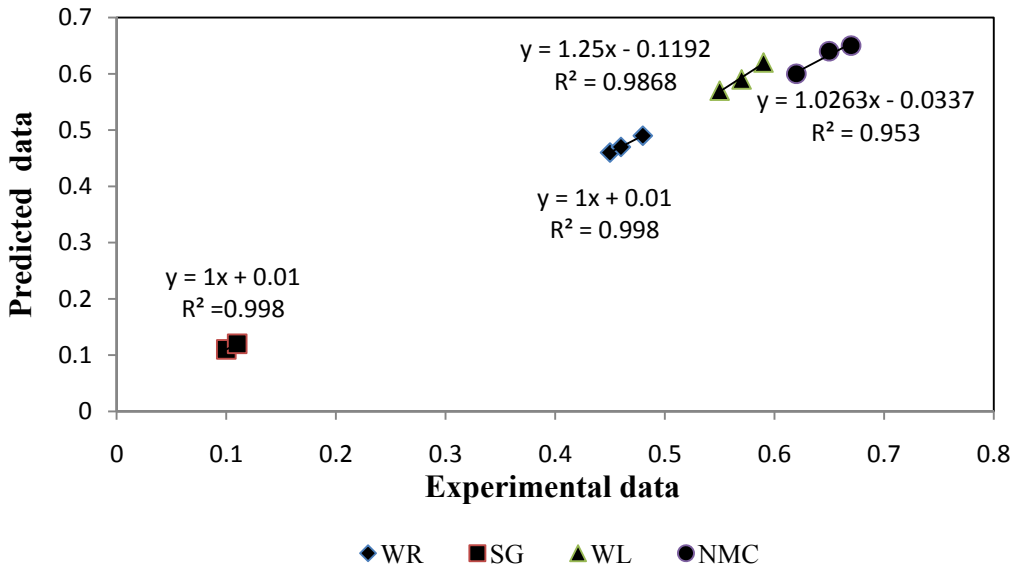


Figure 5.7. Comparisons between the Experimental and Model Predicted Values of Equilibrium WR, SG, WL and NMC of Thermosonically Pretreated Osmodehydrated Seedless Guava

Table 5.8. Effect of Thermosonication Pretreatment on Peleg's Parameters Constants and Goodness of Fit

		Amplitude Level (%)			
		Control	25	50	75
WR	K_1	21.11±2.39	23.01±2.80	22.52±2.81	22.77±2.85
	K_2	2.22±0.06	2.18±0.07	2.12±0.07	2.03±0.06
	R^2	0.99	0.99	0.99	0.99
	Chi-square	0.0050	0.000	0.000	0.000
	RMSE	0.0046	0.001	0.001	0.001
	E (%)	2.98	0.72	1.01	1.10
SG	K_1	116.02±30.08	127.24±10.62	122.30±7.39	121.99±7.85
	K_2	8.88±0.65	8.47±0.20	8.32±0.14	8.07±0.15
	R^2	0.95	0.99	0.99	0.98
	Chi-square	0.0004	5.2E-05	7.0E-05	2.0E-05
	RMSE	0.0006	0.000	0.000	0.000
	E (%)	1.69	0.60	0.64	0.37
WL	K_1	18.10±1.90	19.84±1.95	19.33±1.91	19.47±1.94
	K_2	1.78±0.04	1.74±0.04	1.69±0.04	1.62±0.04
	R^2	0.99	0.99	0.99	0.99
	Chi-square	0.0050	0.000	0.000	0.000
	RMSE	0.0050	0.001	0.001	0.001
	E (%)	2.65	0.63	0.85	0.86
NMC	K_1	43.32±6.27	42.68±4.90	29.84±4.79	29.12±3.91
	K_2	3.56±0.14	2.95±0.09	2.86±0.12	2.63±0.09
	R^2	0.98	0.99	0.98	0.98
	Chi-square	0.0008	4.8E-0.5	6.2E-05	9.8E-05
	RMSE	0.0028	0.000	0.000	0.000
	E (%)	0.73	0.20	0.21	0.31

K_1 Peleg rate constant (min g/g^{-1}), K_2 Peleg capacity constant $((g/g)^{-1})$

Table 5.9. Equilibrium WR, SG, WL and NMC Predicted using Peleg Model for Thermosonically Pretreated Osmodehydrated Seedless Guava

Type of Pretreatment	Equilibrium WR (g/g)	Equilibrium SG (g/g)	Equilibrium WL (g/g)	Equilibrium NMC (%)
Hot water (90 °C)	0.45±0.007	0.11±0.006	0.56±0.005	0.70 ±0.010
TS1	0.46±0.01	0.11±0.002	0.57±0.01	0.65±0.008
TS2	0.47±0.02	0.12±0.010	0.59±0.01	0.64±0.003
TS3	0.49±0.01	0.12±0.008	0.62±0.01	0.60±0.005

All data were obtained by triplicate and expressed as value±SD.
TS1, Thermosonication at 25 % of amplitude; TS2, Thermosonication at 50 % of amplitude and TS2, Thermosonication at 75% of amplitude.

5.3.6 Influence of Thermal Pretreatments on Color Parameter Changes

Color is a property appreciated by the consumer and also an important quality index for the food industry, and specifically for the fruit processing industry. The use of tristimulus color measurement provided a means of quantifying color changes in osmosed and pretreated-osmo dehydrated seedless guava cubes.

The Lightness (L), redness (a) and yellowness (b) values of fresh seedless guava were 72.51±1.35,-3.18±0.8, and 28.14±1.33, respectively. During hot water pretreatment at the temperature range of 80-95°C, the color parameters of fresh seedless guava decreased an average of 15-24%, 38-47% and 31-39% for L, a and b values, respectively. Table 5.10 shows the average and standard deviation of normalized lightness (L), redness (a), yellowness (b) and total color difference (ΔE) values obtained for pretreated osmotically dehydrated seedless guava using hot water blanching at the temperature range of 80-95 °C. The ANOVA for the color parameters (L, a, b and ΔE) indicated significant different ($P < 0.05$) between fresh and thermally treated seedless guava. Further statistical analysis also showed significant difference ($P < 0.05$) between osmo-dehydrated and pretreated (hot water blanching) osmo-dehydrated seedless guava. This result in accordance with that obtained by Chafer *et al*. (2003) who studied the effect of blanching on osmotic dehydration of pear.

With regard to color analysis, high temperatures yielded negative effects over the appearance of the samples (Chenlo *et al*., 2006). According to Moreira *et al.* (2010) browning of thermally pretreated seedless guava is related to non-enzymatic

browning reactions promoted by heat treatments. Stojanovic and Silva (2007) pointed out that the heat degradation of color compounds during dehydration causes reduction in 'a' value. Chlorophyll degradation and pheophytin formation as stated by Robertson (1985) and Llano *et al*. (2003) in kiwifruit slices heated at 100 °C for 5 and 8 min, are also responsible for color changes during dehydration process.

Table 5.10. Effect of Osmotic Dehydration Combined with Hot Water Blanching Pretreatment on Color Parameters of Seedless Guava

Treatments		Color parameters			
		L/L_0	a/a_0	b/b_0	ΔE
OD		0.93 ± 0.008^{a}	0.64 ± 0.04^{a}	0.89 ± 0.01^{a}	5.72 ± 0.63^{a}
HW+OD	80 °C	0.83 ± 0.008^{b}	0.52 ± 0.02^{b}	0.64 ± 0.02^{b}	14.50 ± 0.89^{b}
	85 °C	0.80 ± 0.012^{c}	0.49 ± 0.00^{b}	0.60 ± 0.01^{b}	18.22 ± 0.42^{c}
	90 °C	0.77 ± 0.008^{d}	0.44 ± 0.02^{c}	0.57 ± 0.00^{bc}	20.60 ± 0.32^{d}
	95 °C	0.74 ± 0.006^{e}	0.41 ± 0.02^{c}	0.55 ± 0.01^{c}	22.40 ± 0.76^{e}

All data were obtained at least by triplicate and expressed as value±SD.
Homogeneous values of each color co-ordinate, according with a Tukey test, were reflected by the same letter superscript.
OD, Osmotic dehydration process; HW, Hot water blanching treatment.

Table 5.11 compares the average and standard deviation of normalized lightness (L), redness (a), yellowness (b) and total color difference (ΔE) values of pretreated osmotically dehydrated by using hot water blanching at 90 °C with pretreated osmotically dehydrated seedless guava using thermosonication blanching at the amplitude range of 25-75% combined with 90 °C. Thermosonically osmosed samples show the same trends ('L', 'a' and 'b' decreased and ΔE increased) with that of hot water blanched seedless guava. The application of both kind of thermal

pretreatment provoked browning in seedless guava cubes before osmotic dehydration but no significant differences ($P > 0.05$) were found between pretreatments. It is apparet from Table 5.11 that the greatest total color differences (ΔE) occurred during osmotic dehydration of thermosonicaly pretreated samples. This suggests that a combination of high temperature, high ultrasonic wave's amplitude, and oxygen availability had the largest influence on color change of the seedless guava cubes.

Table 5.11. Effect of Osmotic Dehydration Combined with Thermosonication Blanching Pretreatment on Color Parameters of Seedless Guava

Treatments		Color parameters			
		L/L_0	a/a_0	b/b_0	ΔE
HW(90 °C)+OD		0.77±0.002[a]	0.44±0.01[a]	0.57±0.080[a]	20.60±0.23[a]
TS+OD	25%	0.76±0.004[a]	0.42±0.02[a]	0.56±0.007[a]	21.10±0.48[a]
	50%	0.75±0.009[a]	0.40±0.02[a]	0.55±0.013[a]	21.86±0.92[a]
	75%	0.75±0.008[a]	0.39±0.01[a]	0.54±0.018[a]	22.18±1.12[a]

All data were obtained at least by triplicate and expressed as value±SD.
Homogeneous values of each color co-ordinate, according to a Tukey test, were reflected by the same letter superscript.
OD, Osmotic dehydration process; TS, Thermosonication treatment.

5.3.7 Influence of Thermal Pretreatments on Textural Parameter Changes

Textural values (hardness, area under the curve and initial modulus) of seedless guava cubes at the end of osmotic dehydration with/without hot water pretreatment are shown in Table 5.12. Textural attributes of samples thermally treated at the temperature range of 80-95 °C were much lower than those of fresh samples, suggesting a more pronounced effect of heat on cellular structure. The hardness of

seedless guavas decreased to average values of 27%, 33%, 44% and 54% due to hot water treatment at temperatures of 80, 85, 90 and 95 °C, respectively. The area under the curve also decreased with hot water blanching pretreatment of seedless guava, by values of 30% or higher. Initial modulus showed an average decrease with the hot water blanching step of 49%, relatively to fresh seedless guava. These results indicate that the thermal treatment lead to significant ($P < 0.05$) loss of hardness and elasticity. Membrane disruption and pectic polymers degradation in fruits and vegetables which are associated with cell to cell adhesion and increase of the intercellular spaces could be led to loss of hardness during heat treatment (Waldron *et al*., 1997). Similar behavior reported by Moreno *et al*. (2000), Pereira *et al*. (2006) and Nunes *et al*. (2008), for thermally treated strawberries, guava, papaya and palms. Further statistical analysis reflects a significant ($p < 0.05$) decrease of the hardness, area under the curve and initial modulus in hot water pretreated osmo-dehydrated seedless guava in comparison with non-treated osmo-dehydrated sample.

Table 5.12. Effect of Hot Water Pretreatment and Osmotic Dehydration Combined with Pretreatment on Texture Parameters of Seedless Guava

Treatments		Texture parameters		
		Hardness (g)	Area under curve (g.sec)	Initial slope (g/sec)
OD		0.89±0.01[a]	0.91±0.05[a]	0.85±0.04[a]
HW+OD	80	0.68±0.01[b]	0.64±0.02[b]	0.62±0.03[b]
	85	0.61±0.02[c]	0.59±0.03[c]	0.55±0.01[c]
	90	0.52±0.01[d]	0.45±0.01[d]	0.51±0.00[d]
	95	0.51±0.01[d]	0.45 ±0.00[d]	0.47±0.01[e]

All data were obtained at least by triplicate and expressed as value±SD.
Homogeneous values of each texture parameter, according to a Tukey test, were reflected by the same letter superscript.
OD, Osmotic dehydration process; HW, Hot water blanching treatment.

Thermosonication pretreatments provoked a decrease in values of the hardness, area under the curve and initial slope comparing with hot water pretreatment (Table 5.13). When the effect of thermosonication pretreatment conditions were analyzed by analysis of variance (ANOVA) for the mentioned textural parameters, it was observed that there were no significant differences ($p>0.05$) between the thermal and thermosonication treatments studied. Thermosonication enhanced these changes probably due to the mechanical damage as a consequence of the ultrasonic waves travelling through a medium (Mulet *et al*., 2003). It should be noted that there was a significant differences ($p<0.05$) in the final textural properties of the thermosonically pretreated osmodehydrated seedless guava when compared with non-treated osmo-dehydrated samples. On the other hand, no significant differences were found between osmo-dehydrated seedless guava pretreated with hot water and thermosonication. These results showed that the dominant influence is the pretreatment, not the osmotic dehydration process.

Table 5.13. Effect of Thermosonication Pretreatment and Osmotic Dehydration Combined with Thermal Pretreatments on Texture Parameters of Seedless Guava

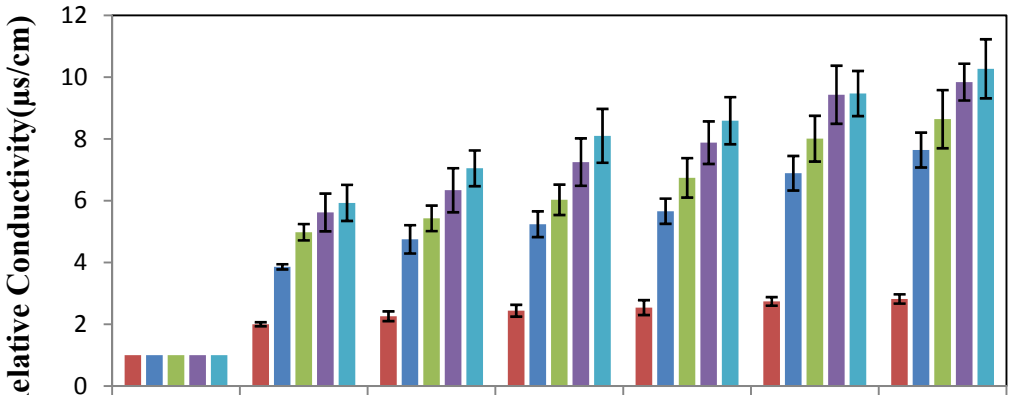
Treatments		Texture parameters		
		Hardness (g)	Area under curve (g.sec)	Initial slope (g/sec)
HW(90 °C)+OD		0.52±0.01[a]	0.45±0.01[a]	0.51±0.00[a]
TS+OD	25%	0.52±0.00[a]	0.44±0.01[a]	0.50±0.02[a]
	50%	0.52±0.01[a]	0.43±0.02[a]	0.48±0.02[a]
	75%	0.51±0.03[a]	0.43±0.00[a]	0.47±0.01[a]

All data were obtained at least by triplicate and expressed as value±SD.
Homogeneous values of each texture parameter, according to a Tukey test, were reflected by the same letter superscript.
OD, Osmotic dehydration process; TS, Thermosonication treatment.

5.3.8 Influence of the Hot water and Thermosonication Blanching on the Leaching of Cell Constituents into the Osmotic Medium

Leaching of cell constituents into the osmotic solution was determined by measuring conductivity of the medium which will give more information on the cellular integrity. The damage to cell membranes was indicated by high amount of conductivity value (Tregunno and Goff, 1996; Knorr and Angersbach, 1998; Lebovka *et al*., 2002). Conductivity of osmotic medium increased with osmotic dehydration time. A similar trend in variation of conductivity of the solutions to WL is expected, since the cell constituents are released during WL. This is confirmed by results shown in Figure 5.2 and 5.6. Based on obtained results it was clear that osmotic dehydration itself caused damage to tissues. The conductivity values of control medium (30%w/w sucrose solution, 33 °C temperature and 180 min immersion time) reported here are lower than those reported by Taiwo *et al*. (2001) and Tedjo *et al*. (2002). The obtained conductivity at the beginning of osmotic dehydration process was 12.38±1.19 µs/cm. According to Schneider (1968) this difference may be due to the different sample to solution weight ratios, since the conductivity value changes being as a function of viscosity, composition and concentration of ions in solution.

Relative conductivity values of the osmotic solution contained thermally pretreated samples were higher than conductivity values of the solution containing untreated samples (Figure 5.8).



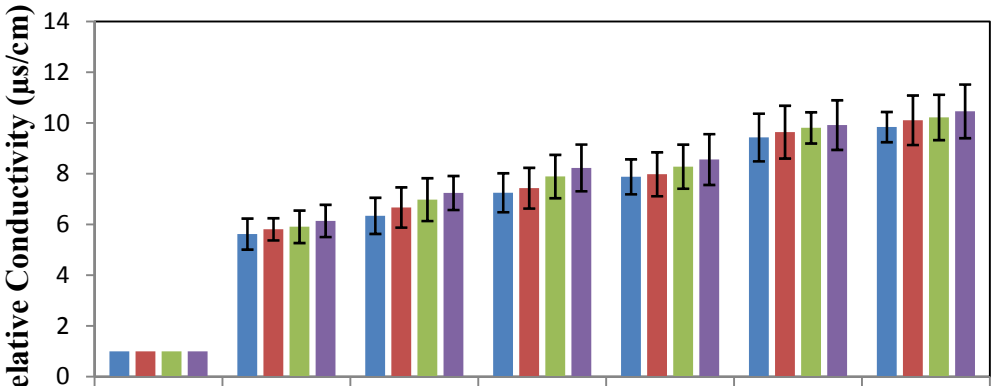
	0	15	30	45	60	120	180
OD	1	2	2.26	2.44	2.54	2.74	2.82
80°C	1	3.86	4.75	5.24	5.66	6.89	7.64
85°C	1	4.98	5.43	6.03	6.74	8.01	8.64
90°C	1	5.62	6.34	7.25	7.88	9.43	9.84
95°C	1	5.93	7.05	8.1	8.59	9.47	10.27

Figure 5.8. Effect of Hot Water Treatment on Relative Conductivity during Osmotic Dehydration

Relative conductivity values were higher at the high temperatures (95 °C) which the highest recorded increase was 364% as compared to the control. These results indicate that significant damage was inflicted upon cell membranes of blanched seedless guava.

Eshtiaghi and Knorr (1993) revealed that with increasing thermal treatment time the potassium leaching from potato pieces was increased. A similar finding was reported by Jackman and Stanley (1995) who suggested thermal treatment in order to increase the solid diffusivity in vegetable tissue as a result of cellular modification. Although the effect of the thermosonication pretreatment on WL was not significant ($P > 0.05$) at 25% of ultrasonic wave's amplitude, it is different ($P < 0.05$) to the cell constituents leaching where thermosonically treated samples had higher conductivity than either untreated and treated samples with hot water (Table 5.9). Therefore,

thermal and thermosonication pretreatments clearly damaged (weakened) the cell wall network and tissue structure due to the heat and heat plus mechanical effects.



	0	15	30	45	60	120	180
90°c	1	5.62	6.34	7.25	7.88	9.43	9.84
25%	1	5.81	6.67	7.43	7.98	9.64	10.11
50%	1	5.91	6.98	7.89	8.28	9.81	10.22
75%	1	6.14	7.24	8.23	8.56	9.92	10.46

Figure 5.9. Effect of Thermosonication Treatment on Relative Conductivity during Osmotic Dehydration

5.4 Summary

The following summary can be made based on the study:

1. Application of hot water blanching pretreatment at the temperature range of 80-90 °C lead to significant ($p < 0.05$) rapid enhancement of mass transfer in terms of WR, SG, WL and NMC.
2. Hot water treatment before osmotic dehydration of seedless guava had a noticeable influence on WL/SG ratio. The loss of selectivity indicates the deep changes in the fruit structure induced by thermal effects during hot water blanching.

3. The ANOVA result indicated that 50 and 75% of ultrasonic wave's amplitude had significant effect on WR, WL and NMC. The studied range of ultrasonic wave's amplitude didn't have significant effect on SG.
4. In all cases, the high values of R^2 and the lowest values of chi-square, RMSE and E (%) display good correlations between the experiments and the model described by Peleg model. This suggest that Peleg equation is suitable for describing the kinetics terms of mass transfer during osmotic dehydration of pretreated seedless guava by both hot water and thermosonication pretreatment.
5. High temperatures of traditional pretreatment applied yielded significant ($p < 0.05$) negative effects over the appearance and texture of the samples whereas no significant differences were found between traditional and thermosonication pretreatments.
6. Measuring the conductivity of the medium showed significant ($p < 0.05$) higher values for both method of pretreatment than untreated samples which indicate that significant damage was inflicted upon cell membranes.

CHAPTER VI

EFFECT OF NONTHERMAL TREATMENTS ON OSMOTIC DEHYDRATION OF SEEDLESS GUAVA (*Psidium Guajava* L.)

6.1 Introduction

Drying is the most popular and well-known postharvest technology to increase shelf-life of tropical fruits which is perishable and deteriorates rapidly after harvesting (Nguyen and Price, 2007). Hot air drying method has several drawbacks such as quality deterioration of the final product, energy intensive and consequently cost intensive because it is a simultaneous heat and mass transfer process accompanied by phase change (Barbanti *et al*., 1994; Mujumdar and Menon, 1995). Therefore, scientists and innovative food centres try to develop new drying technologies with the aim to introduce new, safer, fresher and better quality foods with longer life for local and export markets.

Among the drying technologies, osmotic dehydration is very promising due to the low temperature (minimal heat damage) and energy requirements (Panagiotou *et al*., 1999). Generally, osmotic dehydration is a low efficiency and slow process due to the fact that osmotic pressure is a sole driving force for mass transfer during the dehydration process (Toupin and Le Maguer, 1989; Deng and Zhao, 2008). Mavroudis *et al*. (1998) identified two types of resistances, internal and external, which control mass transfer during osmotic dehydration process. Several studies have shown that the structure/composition of the material (Mavroudis *et al*., 1998;

Mauro and Menegalli, 2003), the nature of the solutes (Raoult-Wack *et al*., 1991; Saurel *et al*., 1994) and the pretreatments such as freezing/thawing (Gonzalez-Mendez *et al*., 1985) are main factors which could affect internal resistance. Simal *et al*. (2001) and Bird *et al*. (2002) reported external resistance is affected by factors related to the turbulence of the solution. Van Nieuwenhuijezen *et al*. (2001) pointed out that both internal and external resistance could be affected by temperature. A strong relationship between type of pretreatment and improvement of the rate of mass transfer during osmotic dehydration has been reported in the literature (Tedjo *et al*., 2002).

Due to undesired nutritional, chemical, and physical changes during traditional pretreatments such as blanching, non-thermal processes combined with osmotic dehydration have been applied in order to improve processing efficiency and final product quality. These include the application of pulsed vacuum (Corrêa *et al*., 2010), high hydrostatic pressure (Rastogi *et al*., 2000), high intensity electric field pulses (Amami *et al*., 2007) and supercritical carbon dioxide (Tedjo *et al*., 2002). Wan *et al*. (1992) and Manson *et al*. (1996) claimed that the high intensity of the waves can generate the growth and collapse of bubbles (cavitation) inside liquids and microjets in the direction of the surface which can affect mass transfer was created due to the asymmetric implosions of the cavitation bubbles close to a solid surface. In a solid medium, the sound waves cause other series of effects include heating of the medium (Mason and Lorimer, 2002), structural effects such as the ‘‘sponge effect’’ (Tarleton, 1992; Stojanovic and Silva, 2007), acoustic streaming or microstreaming (Mason *et al*., 1996) or the generation of micro-channels (Muralidhara *et al*., 1985; Cárcel *et al*., 2007) that can affect mass transfer. Due to

the very different results obtained for different fruits (Simal *et al*., 1998; Cárcel *et al*., 2007; Deng and Zhao, 2008), more studies on the effect of power ultrasound on dehydration is still needed to evaluate for which type of fruits and vegetables this technology is viable. It has conclusively been shown that process variables and pretreatments accelerate the rate of mass transfer during osmotic dehydration by increasing the rate of WL and SG, simultaneously (Rastogi *et al*., 2002).

For many years, it has been the objective of many researchers to enhance the WL while retarding the SG due to this fact that high amount of solute, especially sugar, represent a potential human health risk. Unfortunately, the study of Azuara *et al*. (1996) and Amami *et al*. (2007) are the sole studies in the field of centrifugal osmotic dehydration. Therefore, further work is needed to investigate the effect of centrifugation on osmotic dehydration, both mass transfer and quality aspects, of different type of fruits and vegetables. The aim of this study was to investigate the influence of ultrasound and centrifugation on the kinetics of mass transfer and quality parameters (texture and color) during osmotic dehydration of seedless guava.

6.2 Materials and Methods

6.2.1. Sample Preparation

Sample preparation procedure used in the present chapter is similar to that as described in Chapter 3 (i.e. 3.2.1).

6.2.2 Osmotic Dehydration Procedure

6.2.2.1 Ultrasonic-assisted Osmotic Dehydration

The seedless guava cubes were treated in a beaker filled with the sucrose solution (30% w/w) by ultrasonic equipment (Sonics & Materials Inc., Model VC505, Danbury, CT, USA), set at maximum power of 500 W, frequency of 20 kHz, provided with a titanium ultrasonic probe (13 mm diameter) which fixed vertically at 1-2 cm from the samples. There was no direct contact between the fruit and the sonotrode. In order to change ultrasonic intensities the power input was controlled by the variation of the amplitude of the piezo crystals. Applied amplitudes were 25% (31 μm), 50% (62 μm) and 75% (93 μm). The immersed samples in the osmotic solution were subjected to ultrasonic waves during a period of 10 to 40 min. The temperature of the osmotic solution was maintained at 33 ºC throughout all treatments, and no significant increase in temperature (less than 2 ºC) was observed during treatment which was controlled by circulation of water in water bath (Memmert, WNE14. Memmert GmbH Co. KG, Germany). The ratio of fruit to osmotic solution was set at 1:10 (w/w). After the treatments, the samples were removed from the syrup, rinsed in distilled water (below 30 s) eliminate the excess solution adhered to the surface, and carefully blotted with tissue paper to remove the excess surface water. Sampling was performed in time intervals of 15, 30, 45, 60, 120 and 180 min of osmotic dehydration. The experimental set-up was done according to Cárcel *et al*. (2007) as shown in Figure 6.1. All experiments were performed in triplicate.

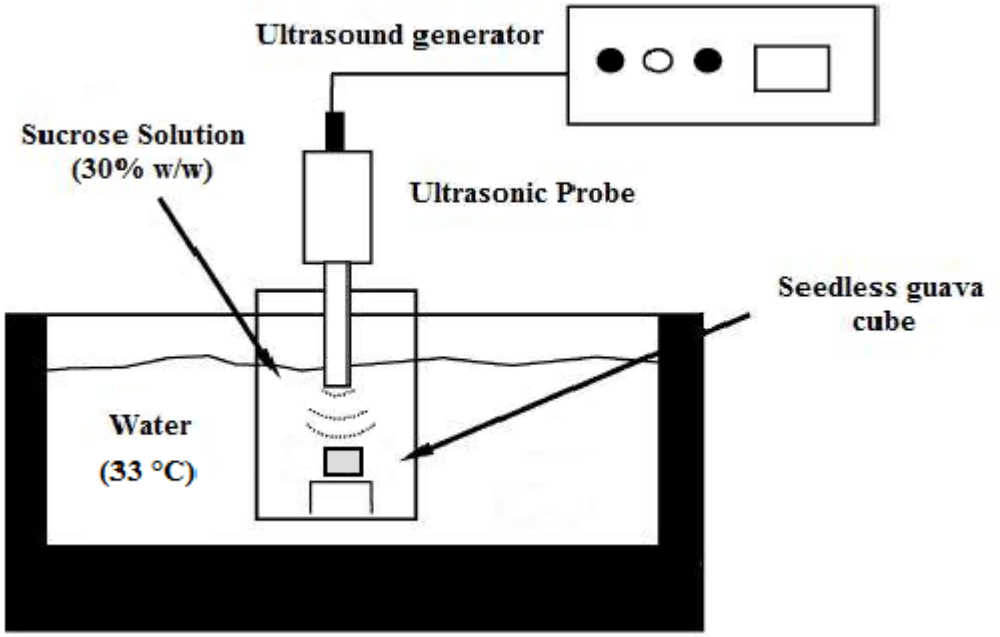


Figure 6.1. Experimental Set-up for Ultrasonic Treatments (Cárcel *et al.*, 2007)

6.2.2.2 Centrifugal Force–assisted Osmotic Dehydration

In the second part of experiment, osmotic dehydration was performed under centrifugation at different centrifugal speeds from 500 to 4000 RPM, using a general purpose centrifuge (Avanti J-25, Beckman Coulter Ireland, Inc., Galway, Ireland). The experiments were done with sucrose solution of 30 % w/w, 1:10 ratio of fruit to solution and temperature of 33 ºC. The centrifugal force-assisted osmotic dehydration experiments at different conditions were done in triplicate. A schematic diagram is presented in Figure 6.2.

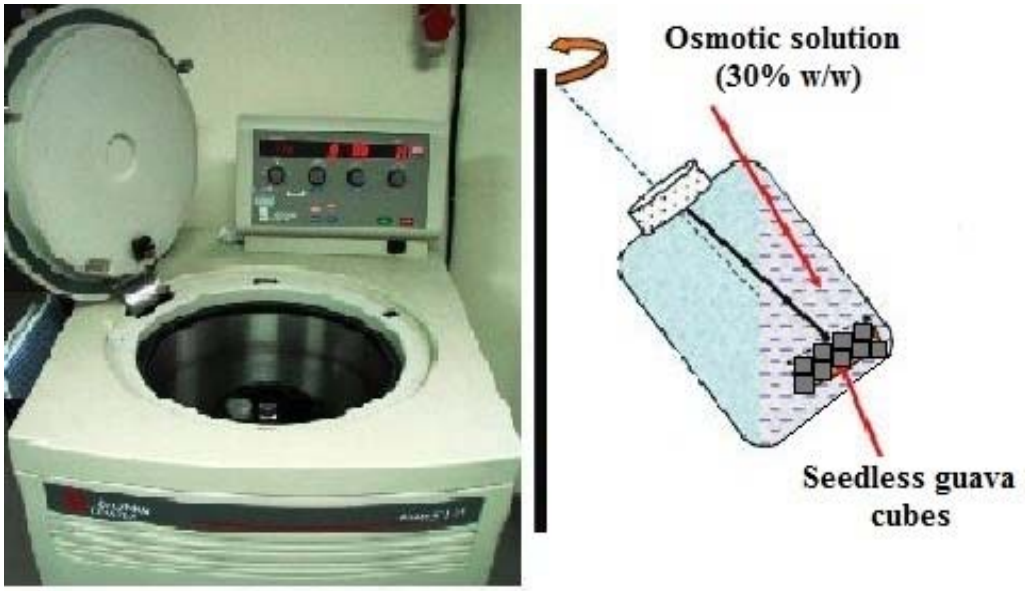


Figure 6.2. Experimental Set-up for Centrifuge Treatments

6.2.3 Analytical Determinations

Analytical determinations procedure used in the present chapter is similar to that as described in Chapter 3 (i.e. 3.2.3).

6.2.4 Determination of Mass Transfer Kinetic Parameters

Kinetic determination procedure used in the present chapter is similar to that as described in Chapter 3 (i.e. 3.2.4).

6.2.5 Mass Transfer Model

Mass transfer model used in the present chapter is similar to that as described in Chapter 3 (i.e. 3.2.5).

6.2.6 Color Measurement

Color measurement used is similar to that as described in Chapter 4 (i.e. 4.2.3).

6.2.7 Texture Analysis

Texture analysis used is similar to that as described in Chapter 4 (i.e. 4.2.4).

6.2.8 Vitamin C Determination

Vitamin C procedure used is similar to that as described in Chapter 4 (i.e. 4.2.5).

6.2.9 Calculation of Quality Changes Kinetics

Kinetics calculation used is similar to that as described in Chapter 4 (i.e. 4.2.5).

6.2.10 Determination Membrane Damage or Electrical Conductivity

Conductivivty measurement used as described in Chapter 5 (i.e. 5.2.9).

6.2.11 Experimental Design and Statistical Analysis

The experiments were arranged in a completely randomized design (CRD) with three replications. The effects of ultrasound and centrifugal force treatments on the changes in kinetics of mass transfer and quality attributes as well as non-Linear

regression and evaluation the goodness of fit were analyzed similar to that as described in Chapter 3 (i.e. 3.2.6.1). Linear regression was used to fit data to Eq. (4.2) and (4.3) in order to estimate rate constant of quality attributes changes using STATISTICA 6.0 (Statsoft Inc.).

6.3 Results and Discussion

6.3.1. Effect of Ultrasonic Treatment on Mass Transfer during Osmotic Dehydration

The experimental results obtained for WR, SG, WL and NMC after osmotic dehydration with and without ultrasound treatment are shown in Figure 6.3-6.6, respectively. As expected, the treated samples showed higher WR, SG, WL and lower NMC than non-treated fruit. To evaluate the significance of the differences, an ANOVA was performed which showed the existence of significant differences between treatments ($p < 0.05$). Tukey test showed that there is no significant difference between 30 and 40 min of ultrasonic treatment duration ($p > 0.05$) for all terms of mass transfer. Thus, ultrasound was only applied for 10-30 min, because the effect of ultrasound showed to be insignificant at lower and higher times. This result is in accordance with those obtained by Fernandes and Rodrigues (2007) and Azoubel *et al*. (2010).

Figure 6.3 shows the evolution of the WR during the osmotic dehydration process by ultrasound and without ultrasound treatment. The enhancement of WR was significantly ($p < 0.05$) dependent on ultrasound amplitude and duration of ultrasonic treatment (10-30 min). Highest WR was achieved by applying the highest amplitude

for 30 min of treatment duration. The curves obtained up to a maximum amplitude and duration of treatment applied, reveal an increase by 21% of WR in comparison with the untreated osmotically dehydrated seedless guava.

Figures 6.4 plotted SG as a function of ultrasonic wave's amplitudes and duration of ultrasonic treatment during the osmotic dehydration. In the graph, ultrasonic wave's amplitude and duration of ultrasonic treatment increased, SG by seedless guava samples also increased. An increase by 3.3% of solid content in seedless guava was observed for ultrasound-assisted osmotic dehydration. This probably occurred due to the cellular tissue modification (intracellular adhesion) as a result of ultrasonic treatment which leads to cellular collapse and fluxes of the solute to the fruit tissue (Heredia-Leon *et al.,* 2004; Quintero-Ramos *et al.,* 2003; Gabaldon-Leyva *et al.,* 2007). These findings were in agreement with the recent results of Siró *et al*. (2009), who have found that ultrasound treatment increased brine diffusion significantly for pork loins. In another study, Rastogi *et al*. (1999) used PEF during osmotic dehydration of carrot. They reported that with increasing electrical field strength the solid diffusion increased due to tissue softening resulting from cell damage caused by the treatment.

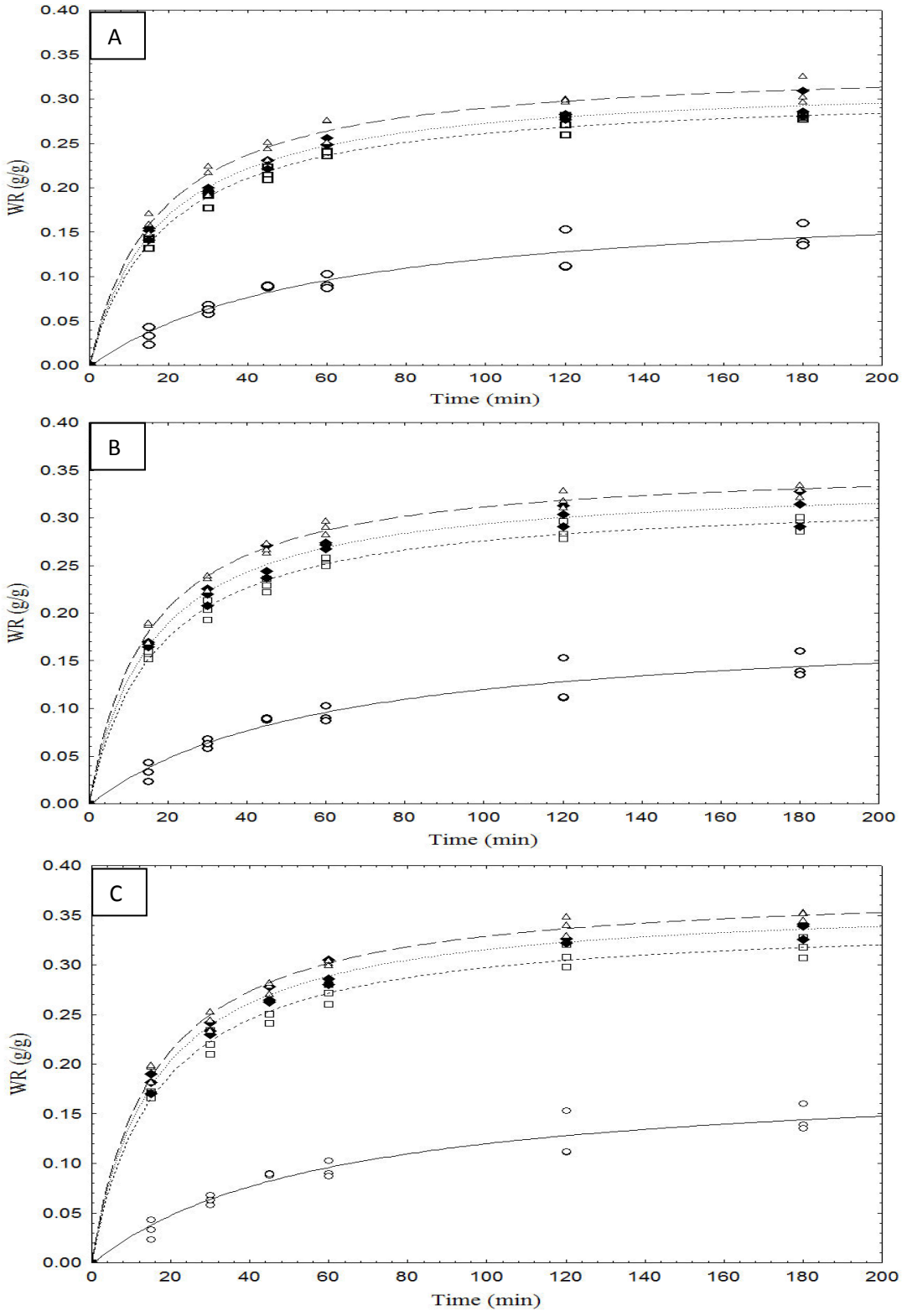


Figure 6.3. Effect of Ultrasound Intensity on WR (A) 25 (B) 50 (C) 75% at Different Time of Exposure (□) 10 (♦) 20 (Δ) 30 min; (○) Control (Lines represent Peleg model)

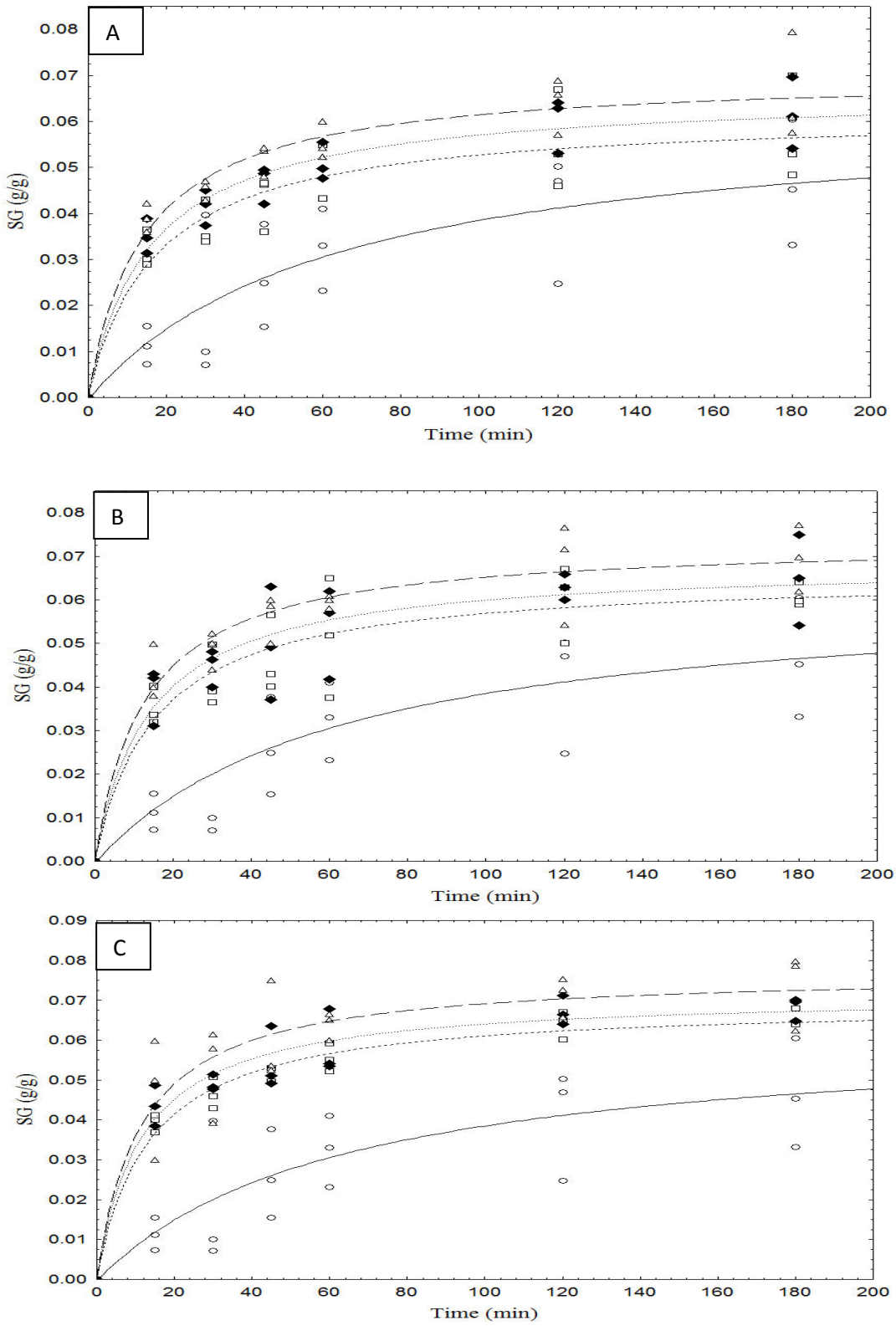


Figure 6.4. Effect of Ultrasound Intensity on SG (A) 25 (B) 50 (C) 75% at Different Time of Exposure (□) 10 (♦) 20 (Δ) 30 min; (○) Control (Lines represent Peleg model)

Figure 6.5 clearly shows the WL during the osmotic dehydration process with and without ultrasound at different ultrasonic wave's amplitudes and duration of ultrasonic treatment. The variation of WL in ultrasonic experiments significantly ($p<0.05$) depended on the amplitude of ultrasound waves and duration of ultrasonic treatment applied, in a similar way to the changes in SG. The result of osmotic dehydration of untreated seedless guava showed that seedless guava lost 19% water (30 %w/w sucrose solution at 33 °C). The water losses of ultrasonically treated seedless guava were 18% and 23% higher than those of untreated samples (25 and 75% amplitude for 30 min), respectively. Thus, the highest applied intensity produced an intensive dehydration due to deep changes in the structure of seedless guavas tissue including loses cell wall rigidity and intracellular adhesion, resulting in more water movement from the tissue to the sucrose solution (Lazarides and Mavroudis, 1995; Heredia-Leon *et al*., 2004). Due to fixed processing temperature, this ultrasonic system generated mechanical effects rather than temperature effects. Faribanks (1974) reported that the rate of dehydration enhanced because of decreased pressure above the sample which encourage WL during ultrasound treatment. Similar findings were reported by Simal *et al*. (1998), who showed that ultrasonic treatment in sliced apples immersed in osmotic solution produced significant WL.

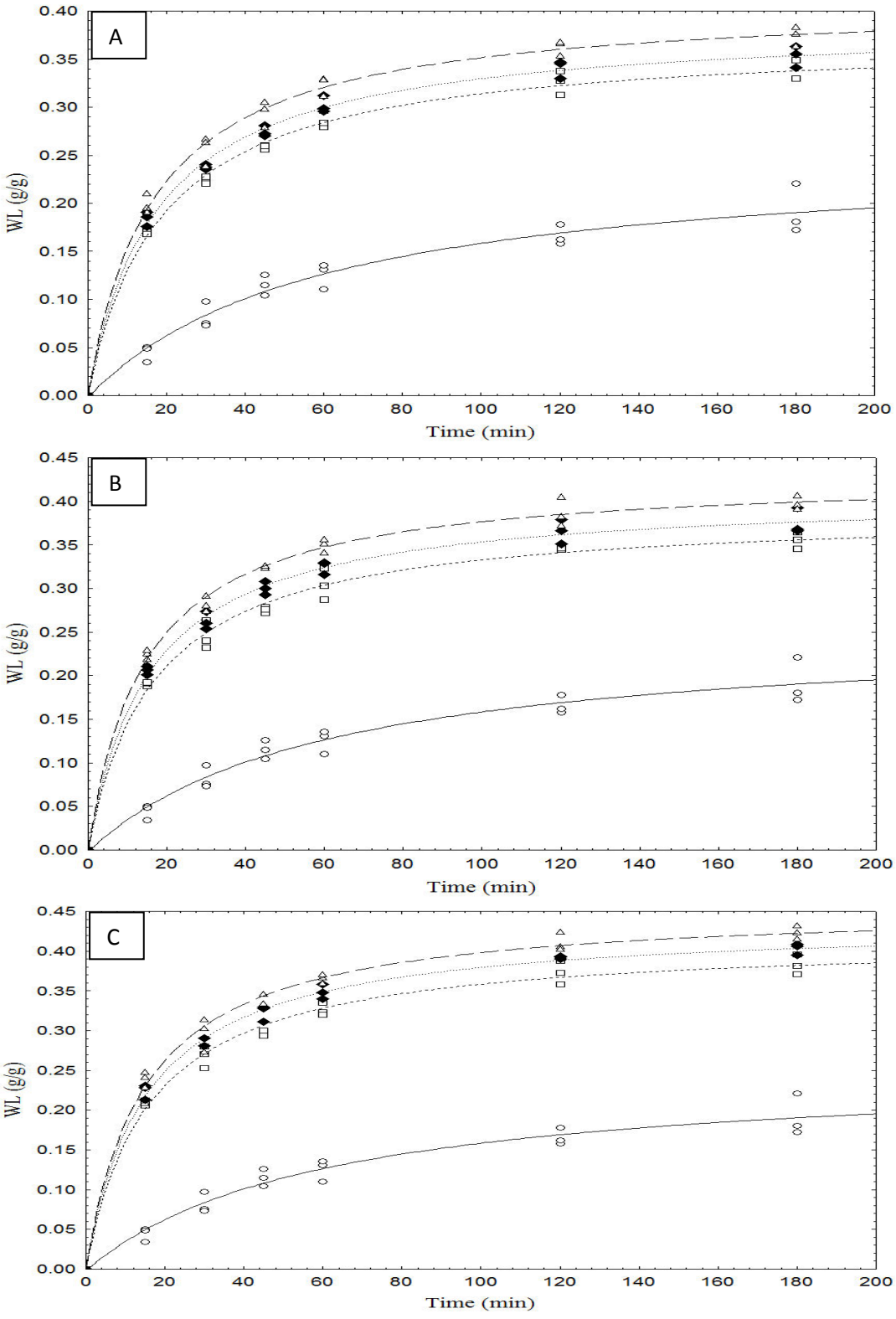


Figure 6.5. Effect of Ultrasound Intensity on WL (A) 25 (B) 50 (C) 75% at Different Time of Exposure (□) 10 (♦) 20 (Δ) 30 min; (○) Control (Lines represent Peleg model)

NMC changes during immersion time are also reflected clearly in Figure 6.6, where a fast initial rate of moisture loss followed by a progressive decrease in the rate regardless of the treatments. A similar trend was found in the previous results on Chilean papayas and apples (Simal *et al*., 1998; Moreno *et al*., 2004). According to Knorr *et al*. (2004) generation of microjets due to asymmetric implosions of the cavitation bubbles close to a solid surface leads to decrease in amount of NMC.

The WL/SG ratio is an adequate index to evaluate the effectiveness of the osmotic process which is permitting estimation of relation between the water and solute transfers inside the food (Alves *et al.,* 2005; Matuska *et al.,* 2006; Moreira *et al.,* 2007). The final WL/SG values were evaluated at each experimental condition and reflected in Table 6.1. Greater dehydration efficiency index was observed in ultrasonically osmosed samples compared with static condition. It is obvious from Table 6.1 that with increase of ultrasound waves amplitude the WL/SG ratio increase, indicating a high efficiency of water removal with minimal solid uptake. Furthermore increase the lengths of sonication decrease the efficiency of the process which reflects the deep structural changes in the fruit structure. It is worthy of note that increae in solid uptake by the product as decrease in membrane selectivity is often considered as undesirable phenomenon (Bonaui *et al*., 1996).

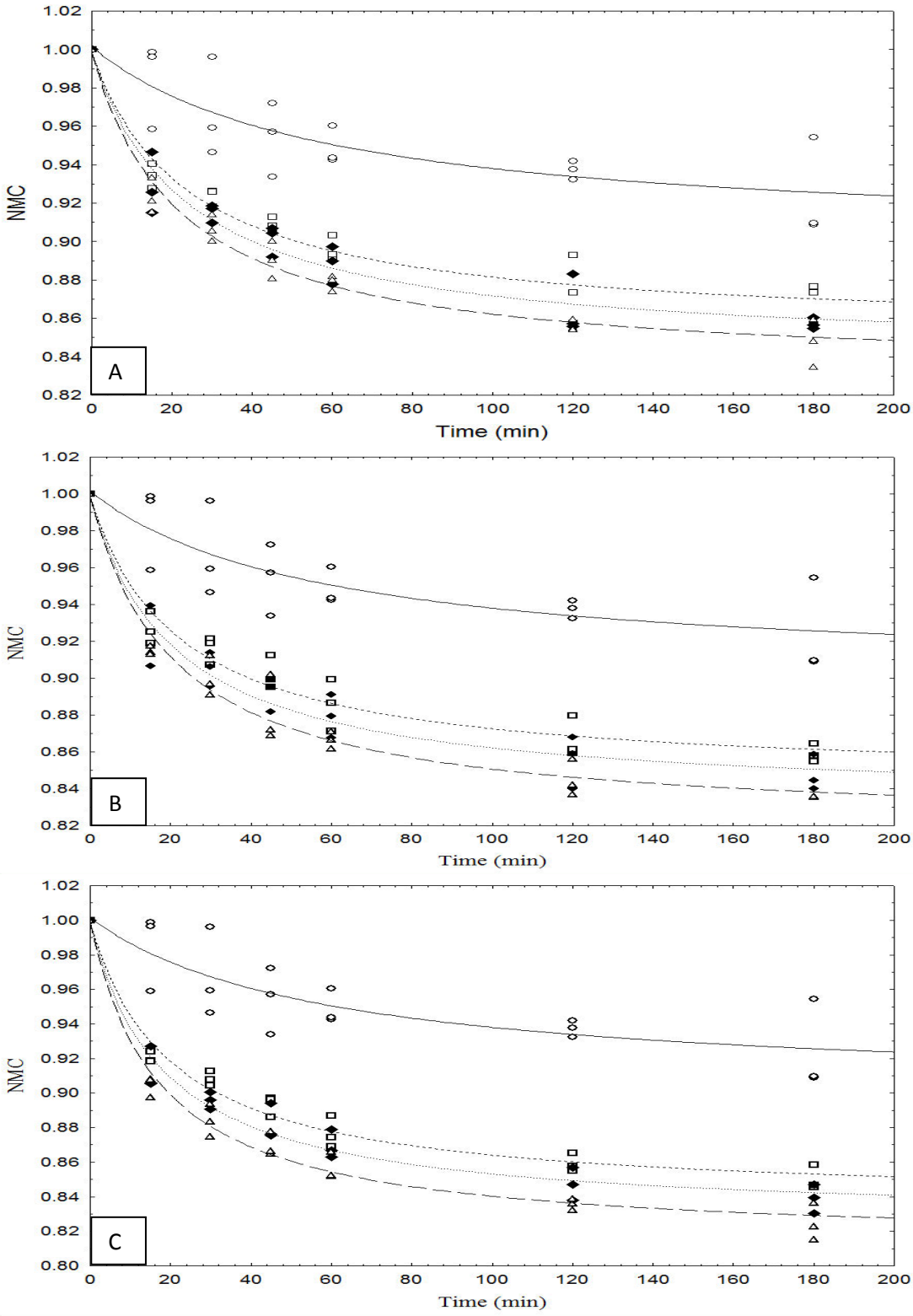


Figure 6.6. Effect of Ultrasound Intensity on NMC (A) 25 (B) 50 (C) 75% at Different Time of Exposure (□) 10 (♦) 20 (Δ) 30 min; (○) Control (Lines represent Peleg model)

Table 6.1. Values of WR, SG, WL and WL/SG after 180 min of ultrasound-assisted Osmotic Dehydration and Untreated Seedless Guava

Ultrasound amplitude (%)	Duration of treatment (min)	WR (g/g)	SG (g/g)	WL (g/g)	NMC (%)	WL/SG
Untreated		0.14±0.010[a]	0.040±0.010[a]	0.19±0.020[a]	0.92±0.020[a]	4.75
25	10	0.27±0.002[b]	0.057±0.010[b]	0.33±0.010[b]	0.86±0.010[b]	5.78
	20	0.29±0.010[c]	0.061±0.007[c]	0.35±0.011[c]	0.85±0.002[c]	5.73
	30	0.30±0.015[c]	0.066±0.011[d]	0.37±0.010[d]	0.83±0.012[d]	5.60
	40	0.30±0.002[c]	0.067±0.003[d]	0.37±0.012[d]	0.83±0.010[d]	5.52
50	10	0.29±0.007[b]	0.060±0.002[b]	0.35±0.009[b]	0.85±0.004[b]	5.83
	20	0.31±0.018[c]	0.064±0.010[c]	0.37±0.014[c]	0.84±0.009[c]	5.78
	30	0.32±0.006[d]	0.069±0.007[d]	0.39±0.007[d]	0.83±0.001[d]	5.65
	40	0.32±0.002[d]	0.070±0.004[d]	0.39±0.002[d]	0.82±0.03[c]	5.57
75	10	0.31±0.010[b]	0.065±0.002[b]	0.38±0.012[b]	0.85±0.007[b]	5.84
	20	0.33±0.008[c]	0.068±0.002[c]	0.40±0.007[c]	0.83±0.008[c]	5.88
	30	0.35±0.004[d]	0.073±0.009[d]	0.42±0.008[d]	0.82±0.010[d]	5.75
	40	0.35±0.003[d]	0.074±0.004[d]	0.42±0.006[d]	0.82±0.003[d]	5.60

Values of WR, SG, WL and NMC were obtained by triplicate and expressed as value±SD.
Homogeneous values, according to a Tukey test, were reflected by the same letter superscript.

Therefore, the influence of ultrasound on mass transfer can be explained by several mechanisms (Haydock and Yeomans, 2003; Knorr *et al*., 2004). These include the degassing effect of sonication (Simal *et al*., 1998; Stojanovic and Silva, 2007), generation of microjets (Cárcel *et al*., 2007), structural affects such as so called ‘‘sponge effect” (Gallego-Juárez *et al*., 1999) and the creation of micro-channels (Muralidhara *et al*., 1985).

Fernandes *et al*. (2009) found that the use of ultrasonic treatment produced micro-channels in the first 10 min of processing. Formation of microscopic channels was also accompanied by a significant degree of cell rupture. The formation of micro-channels in the early stages of the treatment facilitated the mass transfer of water and solid through the tissue. SG increased considerably after 20 min because of the appearance of micro-channels and breakdown of cell, which decreased the resistance of the tissue to the flow of large molecules, such as sucrose molecules. The large micro-channels formed in the fruit tissue explain the lower resistance of the tissue toward the flow of molecules in the fruit and the consequent higher SG and WL observed when this operating condition was applied.

By using the results obtained in the experiments carried out with/without sonication and fitting of the Peleg model to the database, the kinetic parameters (K_1 and K_2) were estimated and presented in Table 6.2. The high R^2 values (> 0.84) and chi-square in a range of 0.000-0.0001, RMSE < 0.001 and E (%) < 2.80%, indicating that Peleg model is suitable and accurate for predicting the kinetics of mass transfer during osmotic dehydration of treated and untreated seedless guava (See Table 6.2).

It can be seen that the K_1 parameter decreased with the increase in the level of ultrasonic wave's amplitude and the duration of ultrasonic treatment except for the 40 min of ultrasonic duration (data not shown). The same behavior was observed for the K_2 parameter. The equilibrium contents were estimated using Eq. (3.7) and the results are presented in Table 6.3. A good agreement (R^2>90) between predicted and experimentally measured data was observed (Figure 6.7).

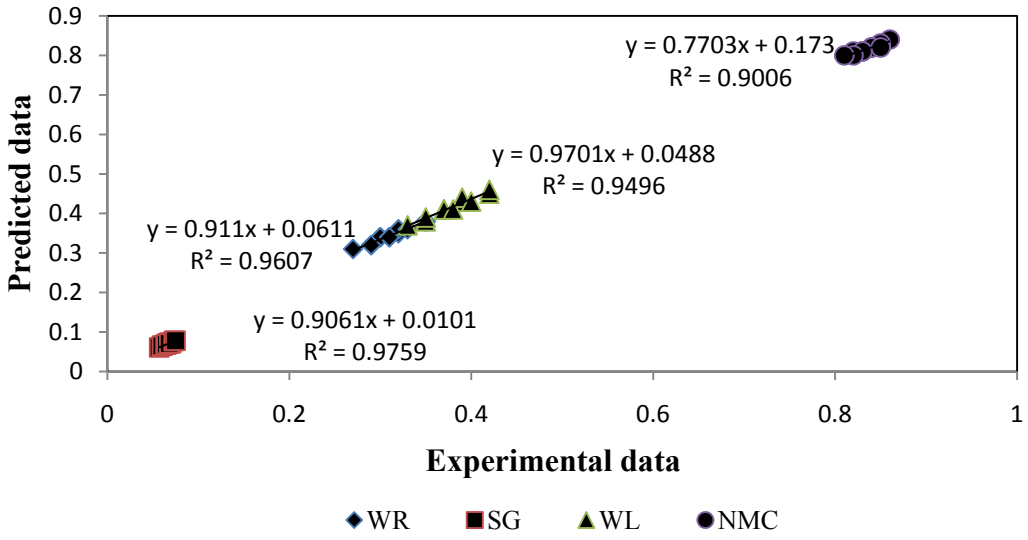


Figure 6.7. Comparisons between the Experimental and Model Predicted Values of Equilibrium WR, SG, WL and NMC of Ultrasound-assisted Osmodehydrated Seedless Guava

Table 6.2. Effect of Ultrasonic Wave's Amplitude and Duration of Treatment on Peleg Parameters and Goodness of Fit

Response	Parameter	Untreated	Treatment								
			25%			50%			75%		
			10	20	30	10	20	30	10	20	30
WR	K_1	305.60 ±52.85	61.37 ±3.30	56.30 ±3.21	51.23 ±4.27	53.05 ±2.98	47.03 ±3.73	41.61 ±2.24	48.23 ±3.07	43.77 ±2.39	41.43 ±2.14
	K_2	5.18 ±0.37	3.21 ±0.05	3.10 ±0.05	2.93 ±0.07	3.10 ±0.05	2.93 ±0.06	2.79 ±0.04	2.88 ±0.05	2.73 ±0.04	2.63 ±0.04
	R^2	0.97	0.99	0.99	0.99	0.99	0.99	0.99	0.99	0.99	0.99
	Chi-square	0.0012	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
	RMSE	0.0010	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.001
	E (%)	3.03	0.79	0.82	0.82	0.89	0.67	0.69	0.86	0.61	0.99
SG	K_1	992.61 ±194.18	285.12 ±80.56	247.99 ±45.69	205.69 ±41.18	238.95 ±68.58	209.32 ±61.15	176.15 ±38.47	196.83 ±23.67	165.32 ±31.50	149.80 ±47.34
	K_2	15.80 ±3.35	16.29 ±1.40	15.18 ±0.83	14.32 ±0.83	15.31 ±1.30	14.71 ±1.25	13.65 ±0.84	14.50 ±0.50	14.04 ±0.74	13.02 ±1.13
	R^2	0.84	0.94	0.97	0.97	0.94	0.94	0.97	0.99	0.97	0.94
	Chi-square	0.0002	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
	RMSE	0.0002	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
	E (%)	1.96	2.67	2.55	2.55	2.47	2.80	2.09	2.33	2.55	1.94
WL	K_1	233.76 ±35.61	50.57 ±2.71	45.98 ±2.43	41.21 ±2.71	43.48 ±2.95	38.50 ±2.34	33.76 ±1.82	43.48 ±2.95	34.98 ±1.62	32.77 ±2.17
	K_2	3.90 ±0.24	2.68 ±0.04	2.57 ±0.04	2.43 ±0.04	2.57 ±0.05	2.44 ±0.04	2.32 ±0.03	2.57 ±0.05	2.29 ±0.03	2.19 ±0.04
	R^2	0.98	0.99	0.99	0.99	0.99	0.99	0.99	0.99	0.99	0.99
	Chi-square	0.001	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
	RMSE	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001
	E (%)	2.41	1.01	1.00	0.96	1.09	0.85	0.83	1.13	0.82	1.14
NMC	K_1	578.04 ±185.73	169.13 ±28.12	153.12 ±25.53	132.85 ±16.44	145.92 ±23.90	128.57 ±22.69	118.50 ±16.58	125.59 ±15.05	106.83 ±14.65	92.3 ±10.25
	K_2	9.99 ±2.01	6.87 ±0.36	6.37 ±0.34	6.00 ±0.23	6.50 ±0.33	6.05 ±0.33	5.58 ±0.24	6.18 ±0.23	5.80 ±0.24	5.38 ±0.18
	R^2	0.84	0.97	0.97	0.98	0.97	0.97	0.98	0.98	0.98	0.99
	Chi-square	4.2E-05	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
	RMSE	0.0007	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001
	E (%)	0.15	0.28	0.30	0.26	0.32	0.33	0.30	0.32	0.32	0.30

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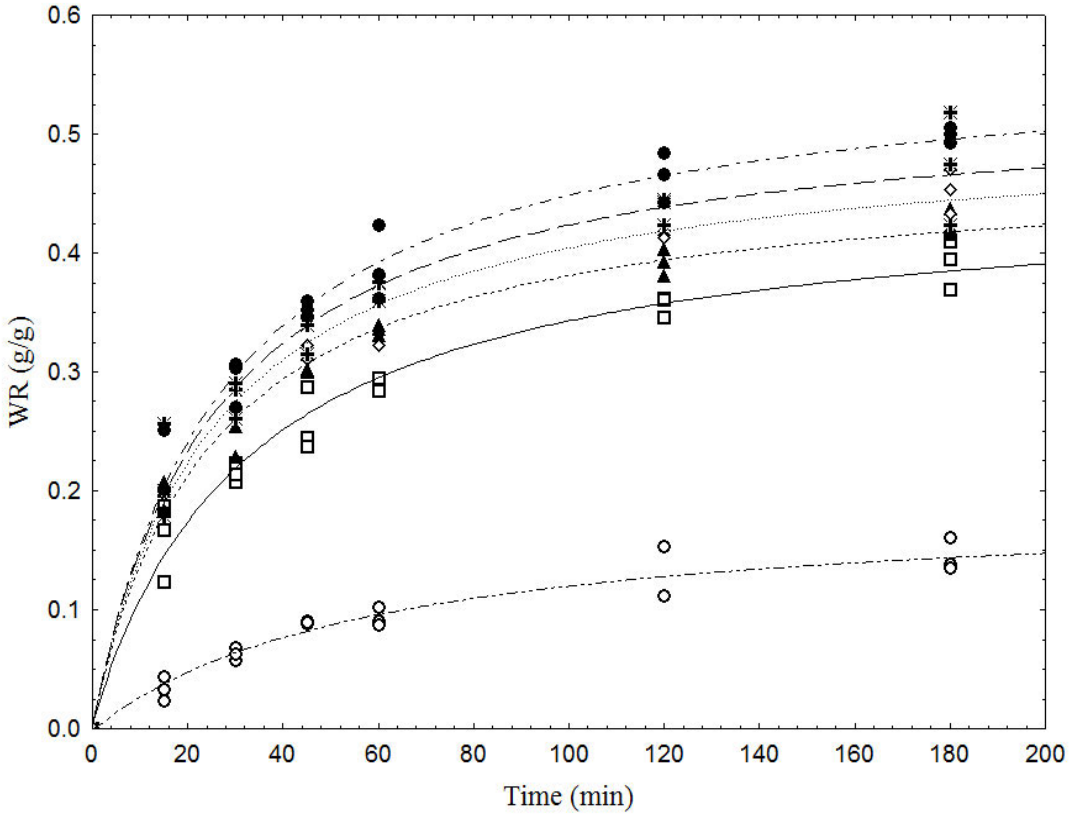
Table 6.3. Equilibrium WR, SG, WL and NMC Predicted Using Peleg Model for Ultrasonic-assisted Osmotic Dehydration

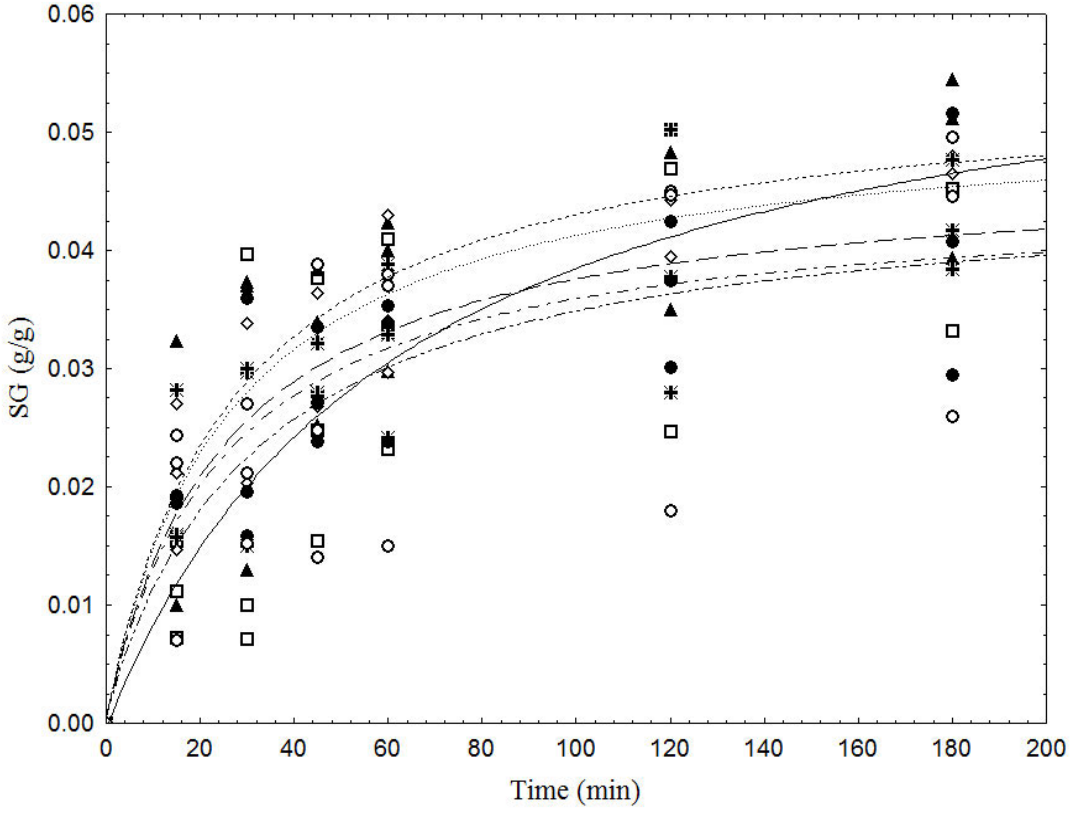
Ultrasound amplitude (%)	Duration of treatment (min)	Equilibrium WR (g/g)	Equilibrium SG (g/g)	Equilibrium WL (g/g)	Equilibrium NMC (%)
25	10	0.31±0.003	0.061±0.003	0.37±0.004	0.84±0.005
	20	0.32±0.004	0.066±0.002	0.38±0.004	0.83±0.005
	30	0.34±0.006	0.070±0.003	0.41±0.005	0.82±0.004
	40	0.34±0.004	0.072±0.004	0.41±0.004	0.82±0.004
50	10	0.32±0.003	0.065±0.004	0.39±0.005	0.83±0.005
	20	0.34±0.005	0.068±0.004	0.41±0.005	0.82±0.006
	30	0.35±0.004	0.073±0.003	0.43±0.005	0.81±0.005
	40	0.36±0.004	0.072±0.004	0.44±0.004	0.81±0.004
75	10	0.34±0.004	0.069±0.001	0.41±0.005	0.82±0.004
	20	0.36±0.004	0.071±0.002	0.43±0.004	0.81±0.005
	30	0.38±0.004	0.077±0.005	0.45±0.006	0.80±0.004
	40	0.38±0.003	0.078±0.001	0.46±0.005	0.80±0.005

All data were obtained by triplicate and expressed as value±SD.

6.3.2 Effect of Centrifugal Force on Mass Transfer during Osmotic Dehydration

The kinetics of WR, SG, WL and NMC during osmotic dehydration of untreated and treated seedless guava cubes using centrifugal force in the sucrose solution of 30 % w/w at 33 °C shown in Figure 6.8. From the figure, there is a clear trend of increasing WR, WL, NMC and decreasing SG with an increase of centrifugal force. For instance, after 180 min of osmotic dehydration, the WL of untreated sample was 0.19±0.02 g/g in absence of centrifugal acceleration, while it attained 0.53±0.01 g/g at 4000 RPM. The SG of untreated sample was 0.046±0.01 g/g and 0.040±0.01g/g in the absence of centrifugal acceleration and at 4000 RPM, respectively. These results are consistent with Azuara's (1996) and Amami's (2007) earlier observations for potato, apple slices and carrot. They reported an increase of WL and lower SG (decreasing of SG) in the centrifugal conditions compared to the static conditions. Furthermore, the rate of WL for treated seedless guava was lower than untreated samples (Figure 6.9) due to the formation of gradients of suspended solids (Azuara *et al*., 1996).


WR (g/g)
Time (min)
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0
20
40
60
80
100
120
140
160
180
200


SG (g/g)
Time (min)
0.00
0.01
0.02
0.03
0.04
0.05
0.06
0
20
40
60
80
100
120
140
160
180
200

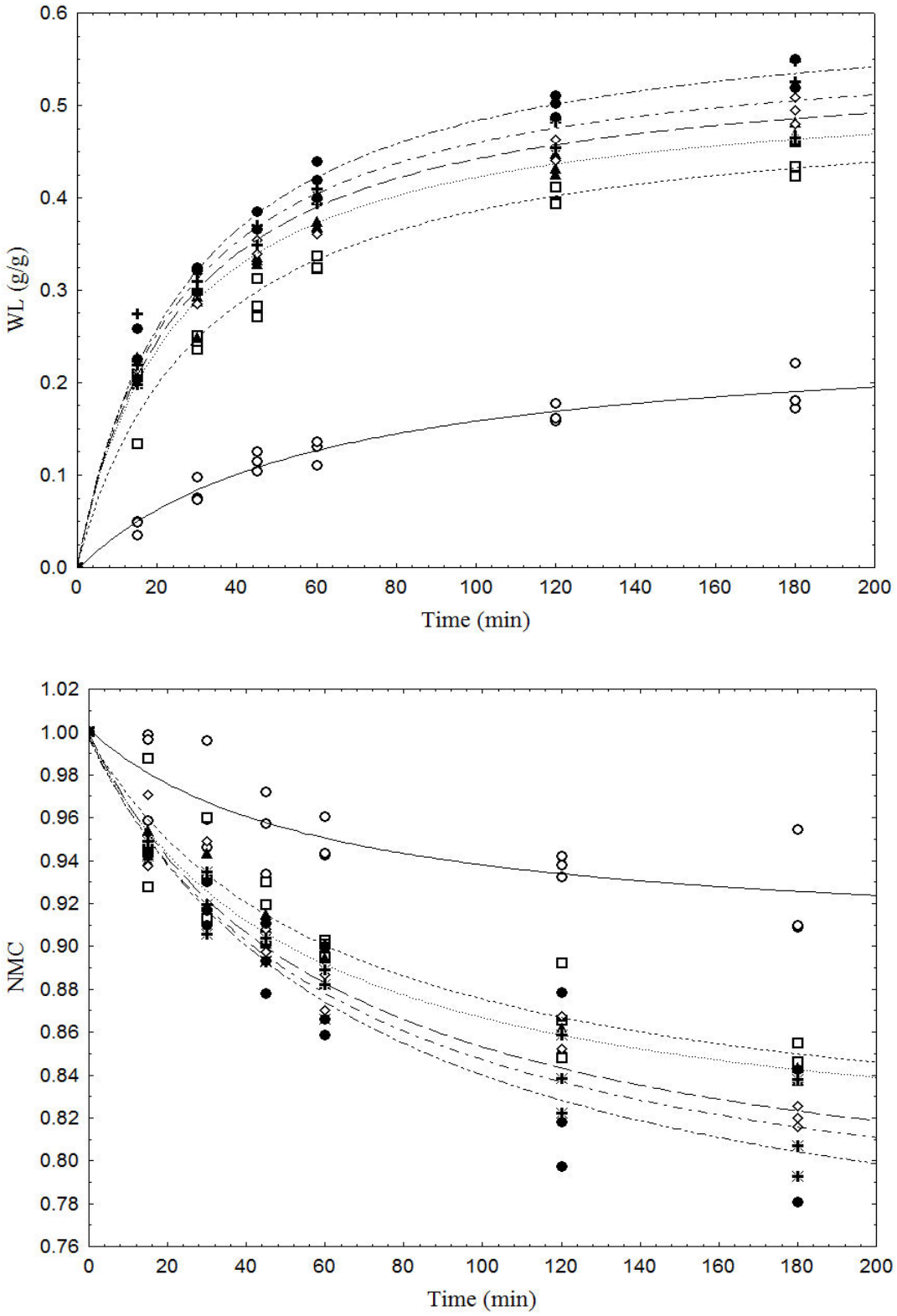


Figure 6.8. Effect of Centrifugal Speed on WR, SG, WL and NMC at (□) 500, (▲) 1000, (◊) 2000, (-) 3000 and (●) 4000 RPM (Lines represent Peleg model)

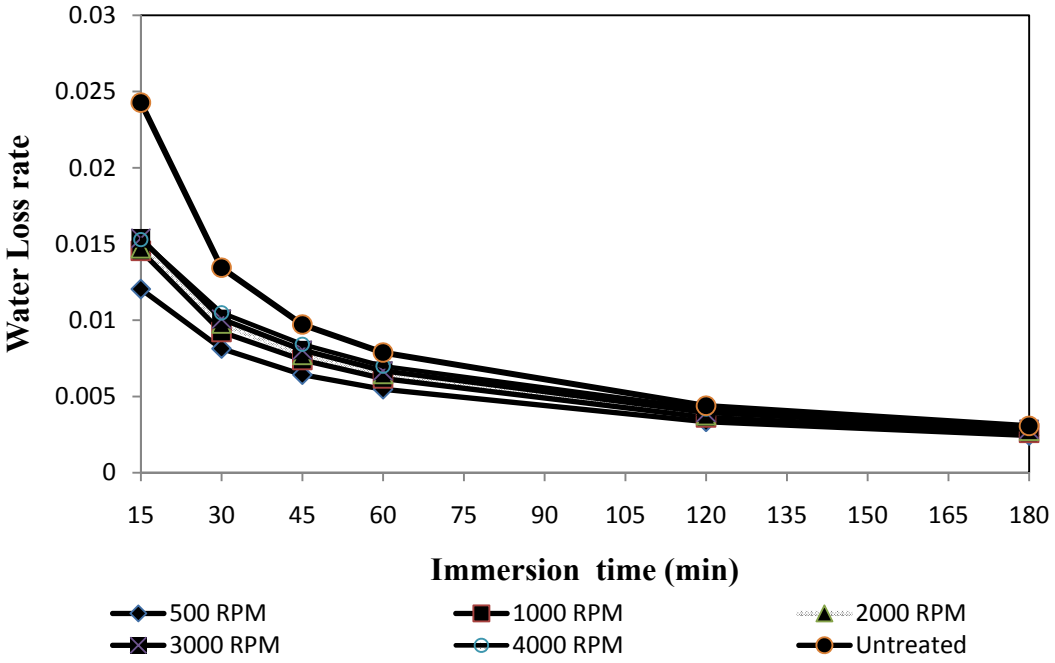


Figure 6.9. Effect of Centrifugal Force on the Rate of Water Loss (ds/dt) with Osmotic Dehydration Time

Table 6.4 presents the amount of WR, SG, WL and NMC of untreated and treated seedless guava using centrifugal force after 180 min of osmotic dehydration. The final WL/SG values have also been reflected in Table 6.4. The WL/SG ratio is much higher for treated seedless guava, than for untreated samples. This finding is in agreement with Azuara's (1996) finding which showed that WL/SG ratio was much higher for apple slices dehydrated by the dynamic system, than for the static system.

Table 6.4. Values of WR, SG, WL and WL/SG after 180 min of Osmotic Dehydration of Centrifugal Treated and Untreated Seedless Guava

Centrifugal Speed(RPM)	WR (g/g)	SG (g/g)	WL (g/g)	NMC (%)	WL/SG
Untreated	0.14±0.01[a]	0.040±0.01[a]	0.19±0.02[a]	0.92±0.02[a]	4.75
500	0.39±0.02[b]	0.048±0.007[b]	0.43±0.01[b]	0.84±0.005[b]	8.95
1000	0.42±0.01[c]	0.046±0.001[b]	0.47±0.009[c]	0.83±0.003[b]	10.21
2000	0.45±0.01[d]	0.042±0.004[c]	0.49±0.01[d]	0.82±0.004[c]	11.66
3000	0.47±0.04[e]	0.040±0.01[d]	0.51±0.04[e]	0.81±0.02[c]	12.75
4000	0.49±0.006[f]	0.040±0.01[d]	0.53±0.01[f]	0.80±0.03[d]	13.25

Values of WR, SG, WL and NMC were obtained by triplicate and expressed as value±SD.
Different superscripts denote significant difference ($p<0.05$) at each column.

The kinetics of WR, SG, WL and NMC using centrifugal force treatment gave good fits with Peleg model. The curve fitting criteria including the coefficient of determination (R^2), chi-square, RMSE and E (%) are summarized in Table 6.5. In all cases, the high values of R^2 (> 0.81) and the lowest values of chi-square (0.000-0.002), RMSE (0.000-0.002) and E (0.11-3.03%) confirm the applicability of Peleg model for the centrifuge-assisted osmotic dehydration of seedless guava cubes. Estimated Peleg equation parameters (K_1 and K_2) for both untreated and treated samples are presented in Table 6.5. It can be seen from the data in Table 6.5 that centrifugal force apparently retarded SG by the foodstuff, but did not interfere with the dehydration process.

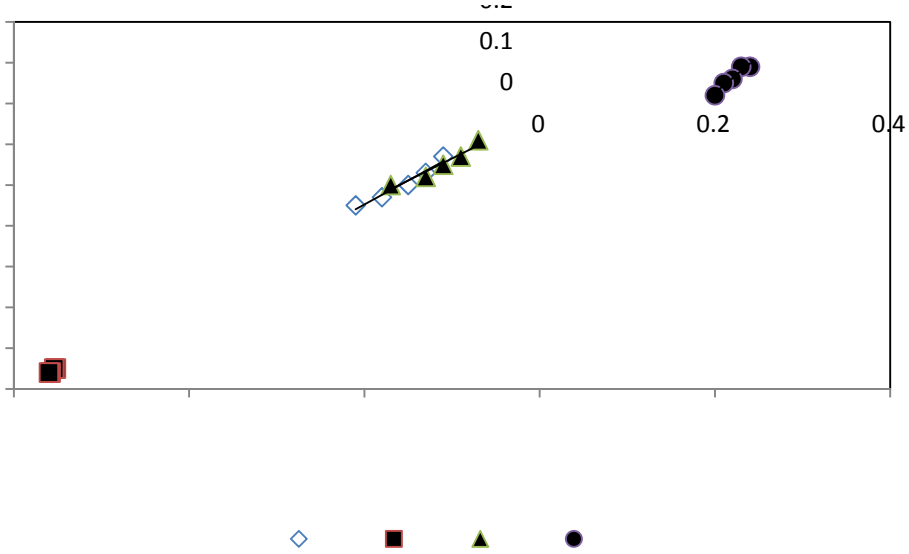
According to Amami *et al*. (2007) two simultaneous counter-current mechanisms can influence the flow of solid include mechanism of diffusion with a difference in concentration and the mechanism of centrifugal force which makes the solid inflows into the food and prevents the solid outflows into the food due to the fact that the solute is heavier than water, respectively. Thus, if the mechanism of centrifugal forces being dominant then a minimal solid is obtained. Therefore, the application of centrifugal osmotic dehydration is favored when the maximal dehydration of product and limited solids uptake is desired.

Table 6.5. Effect of Centrifugal Speed on Peleg's Parameters Constants and Goodness of Fit

		Centrifugal Speed (RPM)					
Response		Untreated	500	1000	2000	3000	4000
WR	K_1	305.60±52.85	73.04±7.85	53.07±3.84	50.65±3.72	49.06±5.82	48.44±4.37
	K_2	5.18±0.37	2.22±0.08	2.11±0.04	1.98±0.04	1.88±0.07	1.75±0.05
	R^2	0.97	0.99	0.99	0.99	0.98	0.99
	Chi-square	0.0012	0.001	0.002	0.001	0.001	0.000
	RMSE	0.0010	0.002	0.002	0.001	0.002	0.001
	E (%)	3.03	1.89	1.96	1.45	1.54	0.90
SG	K_1	992.61±194.18	496.57±190.13	503.75±113.65	551.57±193.86	565.44±209.07	711.01±413.84
	K_2	15.80±3.35	18.49±2.31	19.43±1.42	21.47±2.45	22.53±2.69	22.13±4.36
	R^2	0.84	0.90	0.96	0.91	0.90	0.81
	Chi-square	0.0002	0.000	0.000	0.000	0.000	0.000
	RMSE	0.0002	0.000	0.000	0.000	0.000	0.000
	E (%)	1.96	1.79	1.86	2.76	2.54	3.39
WL	K_1	233.76±35.61	63.79±6.59	48.01±3.50	46.39±3.03	45.15±4.73	45.34±3.10
	K_2	3.90±0.24	1.98±0.06	1.91±0.04	1.81±0.03	1.74±0.05	1.62±0.03
	R^2	0.98	0.99	0.99	0.99	0.99	0.99
	Chi-square	0.001	0.002	0.002	0.001	0.001	0.000
	RMSE	0.001	0.002	0.002	0.002	0.002	0.001
	E (%)	2.41	1.85	1.95	1.54	1.62	1.09
NMC	K_1	578.04±185.73	314.66±77.66	267.64±30.70	260.35±45.22	258.49±40.05	264.23±66.76
	K_2	9.99±2.01	5.00±0.52	4.95±0.22	4.22±0.30	4.06±0.26	3.71±0.42
	R^2	0.84	0.84	0.95	0.98	0.97	0.95
	Chi-square	4.2E-05	4.6E-05	6.1E-05	3.1E-05	6.2E-05	7.8E-05
	RMSE	0.0007	0.000	0.000	0.000	0.000	0.000
	E (%)	0.15	0.16	0.18	0.11	0.19	0.21

K_1 Peleg rate constant (min g/g^{-1}), K_2 Peleg capacity constant ($(g/g)^{-1}$)

agreement (R^2 > 0.91) between predicted and experimentally measured data was observed in Figure 6.10.



ween the Experimental and Model um WR; SG; WL and NMC of odehydrated Seedless Guava

Table 6.6. Equilibrium WR, SG, WL and NMC Predicted Using Peleg Model for Centrifugal-assisted Osmotic Dehydration

Centrifugal Speed (RPM)	Equilibrium WR (g/g)	Equilibrium SG (g/g)	Equilibrium WL (g/g)	Equilibrium NMC (%)
500	0.45±0.010	0.05±0.004	0.50±0.010	0.79±0.008
1000	0.47±0.007	0.05±0.002	0.52±0.008	0.79±0.004
2000	0.50±0.008	0.04±0.003	0.55±0.007	0.76±0.007
3000	0.53±0.013	0.04±0.002	0.57±0.012	0.75±0.006
4000	0.57±0.011	0.04±0.005	0.61±0.008	0.72±0.011

All data were obtained by triplicate and expressed as value±SD.

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6.3.3 Effect of Ultrasound and Centrifugal Force Treatment on Color Properties

Table 6.7 shows the measured lightness (L), redness (a) and yellowness (b) values of an osmotically dehydrated seedless guava with/without ultrasonic and centrifugal force treatments. The L values of the ultrasonic treated samples were significantly higher ($P < 0.05$) than untreated samples which represented the decrease in the degree of browning discoloration. This finding is in agreement with Simal's *et al*. (1998), Gabaldon-Leyva's *et al*. (2007) and Deng and Zhao's (2008) findings which showed that application of ultrasonic waves during osmotic dehydration led to higher ''L'' values of apple cylinders due to inhibition of the oxidant enzymes as a result of greatest water loss, severe cell deformation and tissue collapse. In another study, Simal *et al*. (1998) attributed this result to degassing effect of ultrasonic waves similar to partial vacuum effect. Furthermore, significant differences were found between ultrasonic treatments and untreated seedless guava regarding a, b and ΔE color values.

Surprisingly, application of centrifugal force during osmotic dehydration was found to decrease all color parameters (''L'', ''a'' and ''b'') insignificantly ($p>0.05$). Thus, changes in L, a and b were observed to be minimum in samples obtained by centrifuge-assisted osmotic dehydration at 33 °C with the 30 %w/w sucrose solution (Table 6.7). In addition, the decrease of coordinate ''a'' was higher than that experimented by coordinate b. This decrease of ''a'' and ''b'' can be attributed to the concentration of the liquid phase and the pigments in the cellular tissue as a consequence of water losses during dehydration process.

Treatments			Color Parameters			
			L/L_0	a/a_0	b/b_0	ΔE
Control			0.93±0.008[a]	0.64±0.048 [a]	0.89±0.012 [a]	5.72±0.630 [a]
Amplitude		Duration of Exposure (min)				
Ultrasound	25%	10	0.94±0.007 [a]	0.61±0.022 [a]	0.88±0.006 [a]	5.63±0.402 [a]
		20	0.94±0.010 [a]	0.61±0.024 [a]	0.88±0.014 [a]	5.44±0.570 [b]
		30	0.94±0.009 [a]	0.60±0.300 [a]	0.88±0.008 [a]	5.38±0.386 [c]
	50%	10	0.94±0.006 [a]	0.59±0.002 [a]	0.87±0.001 [a]	5.61±0.333 [b]
		20	0.95±0.009 [b]	0.57±0.004 [b]	0.86±0.011 [b]	5.22±0.427 [c]
		30	0.96±0.005 [b]	0.56±0.017 [b]	0.85±0.013 [b]	4.93±0.393 [d]
	75%	10	0.95±0.010 [b]	0.56±0.004 [b]	0.86±0.001 [b]	5.13±0.369 [b]
		20	0.96±0.005 [b]	0.53±0.014 [b]	0.84±0.003 [b]	5.30±0.330 [c]
		30	0.97±0.008 [b]	0.49±0.019 [b]	0.84±0.009 [b]	5.20±0.565 [d]
Centrifugal Force	Centrifugal Speed (RPM)					
	500		0.93±0.009 [a]	0.61±0.072 [a]	0.88±0.013 [a]	5.90±0.627 [a]
	1000		0.93±0.005 [a]	0.58±0.068 [a]	0.87±0.014 [a]	6.31±0.380 [a]
	2000		0.92±0.003 [a]	0.56±0.019 [a]	0.87±0.002 [a]	6.69±0.387 [a]
	3000		0.92±0.009 [a]	0.53±0.020 [a]	0.86±0.007 [a]	7.13±0.587 [b]
	4000		0.91±0.004 [a]	0.52±0.011 [a]	0.85±0.006 [a]	7.50±0.320 [b]

All data were obtained at least by triplicate and expressed as value±SD.
Different superscripts denote significant difference ($p<0.05$) at each column as compared with control.

the kinetics of color changes. The experimental data of color parameters for ultrasonic and centrifugal force treated seedless guava well fitted to a zero order kinetics model. The R^2 values for each condition of osmotic dehydration were always greater than 0.77 for ultrasonic treated and 0.91 for centrifugal force treated samples. Tables 6.8 and 6.9 show the rate constants (k) were determined from the slopes of normalized curves for changes in ‘‘L’’, ‘‘a’’, ‘‘b’’ and ΔE values of ultrasonic and centrifugal force treated, respectively. It is apparent from Table 6.8 that rate constants for normalized L co-ordinate slightly decreased which confirm for the lighter color of osmo-dehydrated seedless guava treated with ultrasonic wave, whereas the rate constants for ‘‘a’’, ‘‘b’’ co-ordinates increased by ultrasonic treatment. On the other hand, all color co-ordinates of osmo-dehydrated seedless guava treated with centrifugal force increased with increasing centrifugal speed from 500-4000 RPM.

Table 6.8. Rate Constants for Kinetics of Color Changes of Seedless Guava during Ultrasound-assisted Osmotic Dehydration

Treat.	Ultrasound amplitude (%)	Duration of treatment (min)	Color Parameters							
			L		a		b		ΔE	
			k_1	R^2	k_1	R^2	k_1	R^2	k_1	R^2
Control			$3.5\times10^{-4}\pm3.5E-05$	0.91	$2.1\times10^{-3}\pm1.2E-04$	0.96	$5.7\times10^{-4}\pm4.0E-05$	0.94	$3.2\times10^{-2}\pm 0.1E-3$	0.96
Ultrasound	25	10	$3.3\times10^{-4}\pm3.3E-05$	0.91	$2.1\times10^{-3}\pm1.0E-04$	0.97	$6.2\times10^{-4}\pm4.0E-05$	0.95	$3.2\times10^{-2}\pm0.1E-3$	0.97
		20	$3.1\times10^{-4}\pm3.5E-05$	0.90	$2.1\times10^{-3}\pm0.9E-04$	0.98	$6.2\times10^{-4}\pm4.0E-05$	0.95	$3.1\times10^{-2}\pm0.1E-3$	0.96
		30	$3.0\times10^{-4}\pm3.2E-05$	0.90	$2.1\times10^{-3}\pm0.8E-04$	0.98	$6.4\times10^{-4}\pm3.0E-05$	0.96	$3.1\times10^{-2}\pm0.1E-3$	0.97
	50	10	$3.1\times10^{-4}\pm3.5E-05$	0.91	$2.2\times10^{-3}\pm0.9E-04$	0.98	$6.8\times10^{-4}\pm3.0E-05$	0.97	$3.1\times10^{-2}\pm0.1E-3$	0.97
		20	$2.4\times10^{-4}\pm3.0E-05$	0.85	$2.3\times10^{-3}\pm0.7E-04$	0.98	$7.4\times10^{-4}\pm3.0E-05$	0.97	$2.9\times10^{-2}\pm0.1E-3$	0.96
		30	$1.8\times10^{-4}\pm3.5E-05$	0.82	$2.3\times10^{-3}\pm0.8E-04$	0.98	$8.0\times10^{-4}\pm3.0E-05$	0.98	$2.8\times10^{-2}\pm0.1E-3$	0.96
	75	10	$2.4\times10^{-4}\pm3.2E-05$	0.86	$2.3\times10^{-3}\pm0.9E-04$	0.98	$7.2\times10^{-4}\pm2.0E-05$	0.98	$2.9\times10^{-2}\pm0.1E-3$	0.97
		20	$3.5\times10^{-4}\pm3.1E-05$	0.83	$2.5\times10^{-3}\pm0.7E-04$	0.99	$8.3\times10^{-4}\pm3.0E-05$	0.98	$3.0\times10^{-2}\pm0.1E-3$	0.97
		30	$1.5\times10^{-4}\pm3.0E-05$	0.77	$2.7\times10^{-3}\pm0.7E-04$	0.99	$8.6\times10^{-4}\pm2.0E-05$	0.99	$2.9\times10^{-2}\pm0.9E-4$	0.97

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Table 6.9. Rate Constants for Kinetics of Color Changes of Seedless Guava during Centrifugal -assisted Osmotic Dehydration

Treatment	Centrifugal Speed (RPM)	Color Parameters							
		L		a		b		ΔE	
		k_1	R^2	k_1	R^2	k_1	R^2	k_1	R^2
Control		3.5×10^{-4}±3.5E-05	0.91	2.1×10^{-3}±1.2E-04	0.96	5.7×10^{-4}±4.0E-05	0.94	3.2×10^{-2}± 0.1E-3	0.96
Centrifugal Treatment	500	3.5×10^{-4}±3.4E-05	0.92	2.1×10^{-3}±1.4E-04	0.95	6.1×10^{-4}±5.0E-05	0.94	3.3×10^{-2}± 0.1E-3	0.91
	1000	3.8×10^{-4}±3.3E-05	0.93	2.2×10^{-3}±1.2E-04	0.97	6.6×10^{-4}±4.0E-05	0.96	3.5×10^{-2}± 0.1E-3	0.97
	2000	4.1×10^{-4}±3.2E-05	0.94	2.3×10^{-3}±1.1E-04	0.97	7.0×10^{-4}±4.0E-05	0.96	3.7×10^{-2}± 0.1E-3	0.97
	3000	4.3×10^{-4}±3.4E-05	0.94	2.6×10^{-3}±1.4E-04	0.97	7.7×10^{-4}±5.0E-05	0.96	4.0×10^{-2}± 0.1E-3	0.97
	4000	4.6×10^{-4}±4.1E-05	0.93	2.6×10^{-3}±1.0E-04	0.98	7.7×10^{-4}±2.0E-05	0.99	4.2×10^{-2}± 0.2E-3	0.96

6.3.4 Effect of Ultrasound and Centrifugal Force on Texture Properties

Table 6.10 shows the results obtained from osmo-dehydrated seedless guava with/without ultrasonic and centrifugal force treatments for mechanical parameters (hardness, area under the curve and initial modulus). These parameters were significantly ($P<0.05$) affected by the ultrasonic and centrifugal force methods. In general both ultrasonic and centrifugal force treatment caused a decrease in hardness. The area under the curve measured in the ultrasound and centrifugal force treated samples was only slightly lower compared to untreated osmo-dehydrated tissues (Table 6.10). This suggests that the tissue structure remained less intact after treatments. In ultrasonic treatment, no additional benefit could be achieved by 25% ultrasonic wave's amplitude for 10-30 min of ultrasonic exposure. Pohlman *et al*. (1997) also found that no additional benefit could be achieved by 10 min of ultrasonic exposure from the texture properties point of view. The initial modulus of treated samples was lower than that of fresh seedless guava but the extent of reduction of the initial modulus depended on the type of pretreatment. The results on the hardness changes in the osmotic process may be the result of a combination of the leaching of cell constituents and the physical effects of the ultrasonic treatment which damages the tissue and lead to softening (Jen and Robinson, 1984; Floros and Liang, 1994). According to Smith *et al*. (1991) and Got *et al*. (1999) the destructive nature of ultrasonic cavitation and the vibration of the ultrasonic waves themselves can lead to physical weakening of the structure. Deng and Zhao (2008) reported that the cells of the ultrasound treated apples could not be clearly identified and suffered the highest degree of collapse which may be ascribed to the ultrasonic effects.

The samples subjected to osmotic dehydration in the absence of centrifugal force are firmer and more elastic than the samples that are treated using centrifugal force. The hardness loss (Table 6.10) of centrifugal force treated seedless guava may be the result of the great turgor loss due to high amount of WL. The textural study shows that a product obtained with centrifugal treatment is slightly less affected than ultrasonic treatment. Table 6.11 shows the softening rate constants for the osmo-dehydrated seedless guava as a function of ultrasonic and centrifugal force treatments. A good fit of experimental data is obtained, which confirms the applicability of zero-order kinetic model. It can be seen that in both cases the softening rate is increased.

Table 6.10. Values of Normalized Textural Properties after 180 min of Ultrasound- and Centrifugal-assisted Osmotic Dehydration of Seedless Guava

Treatments			Texture Parameters		
			Hardness (g)	Area under curve (g.sec)	Initial Modulus (g/sec)
Control			0.89±0.01 [a]	0.91±0.05 [a]	0.85±0.04 [a]
	Amplitude	Duration of Exposure(min)			
Ultrasound	25%	10	0.89±0.012 [a]	0.91±0.022 [a]	0.78±0.005 [b]
		20	0.89±0.014 [a]	0.91±0.020 [a]	0.78±0.012 [b]
		30	0.88±0.009 [a]	0.90±0.003 [a]	0.78±0.006 [b]
	50%	10	0.88±0.010 [a]	0.89±0.003 [a]	0.76±0.001 [b]
		20	0.87±0.009 [a]	0.87±0.005 [b]	0.75±0.010 [b]
		30	0.86±0.006 [b]	0.86±0.013 [b]	0.75±0.009 [b]
	75%	10	0.86±0.008 [b]	0.86±0.003 [b]	0.76±0.001 [b]
		20	0.84±0.006 [b]	0.83±0.011 [b]	0.74±0.004 [b]
		30	0.82±0.002 [b]	0.79±0.015 [b]	0.73±0.010 [b]
Centrifugal Treatment	Centrifugal Speed (RPM)				
	500		0.89±0.009 [a]	0.88±0.022 [a]	0.82±0.011 [a]
	1000		0.88±0.012 [a]	0.87±0.018 [a]	0.79±0.010 [b]
	2000		0.87±0.005 [a]	0.84±0.009 [b]	0.77±0.005 [b]
	3000		0.85±0.016 [b]	0.83±0.009 [b]	0.76±0.005 [bc]
	4000		0.84±0.012 [b]	0.83±0.011 [b]	0.75±0.003 [c]

All data were obtained at least by triplicate and expressed as value±SD.
Different superscripts denote significant difference ($p<0.05$) at each column as compared with control.

Table 6.11. Rate Constants for Kinetics of Hardness Changes of Seedless Guava during Ultrasound- and Centrifugal-assisted Osmotic Dehydration

Treatments			Hardness	
			k_1 (min^{-1})	R^2
Control			0.00058±0.00003	0.96
Ultrasound	Amplitude	Duration of Exposure(min)		
	25%	10	0.00058±0.00004	0.96
		20	0.00058±0.00003	0.96
		30	0.00061±0.00002	0.98
	50%	10	0.00063±0.00003	0.97
		20	0.00066±0.00003	0.98
		30	0.00073±0.00002	0.98
	75%	10	0.00073±0.00001	0.99
		20	0.00084±0.00002	0.99
		30	0.00095±0.00002	0.99
Centrifugal Treatment	Centrifugal Speed (RPM)			
	500		0.00059±0.00004	0.96
	1000		0.00063±0.00003	0.97
	2000		0.00071±0.00003	0.97
	3000		0.00077±0.00003	0.97
	4000		0.00085±0.00004	0.97

6.3.5 Effect of Ultrasound and Centrifugal Force Treatment on Vitamin C Retention

The vitamin C content in fresh seedless guava was 122.31±3.60 mg/100 g of edible flesh. Table 6.12 shows the values of vitamin C retention for an osmotically dehydrated seedless guava with/without ultrasonic and centrifugal force treatments. It was observed that 7% of vitamin C was lost during osmotic dehydration process at optimized condition (30%w/w sucrose concentration, 33 ºC temperature and 180 min of dehydration time). Analysis of variance procedure was performed to investigate differences in the amount of vitamin C loss due to the ultrasonic and centrifugal force treatments. Loss of vitamin C showed no significant differences ($p > 0.05$) associated to ultrasound and centrifugal force treatments. Furthermore, the vitamin C retention was increased in ultrasonic treatment at high amplitude and sonication time due to degassing effect of ultrasound wave (Simal *et al*., 1998).

Table 6.12. Vitamin C Retention after 180 min of Ultrasound and Centrifugal-assisted Osmotic Dehydration of Seedless Guava

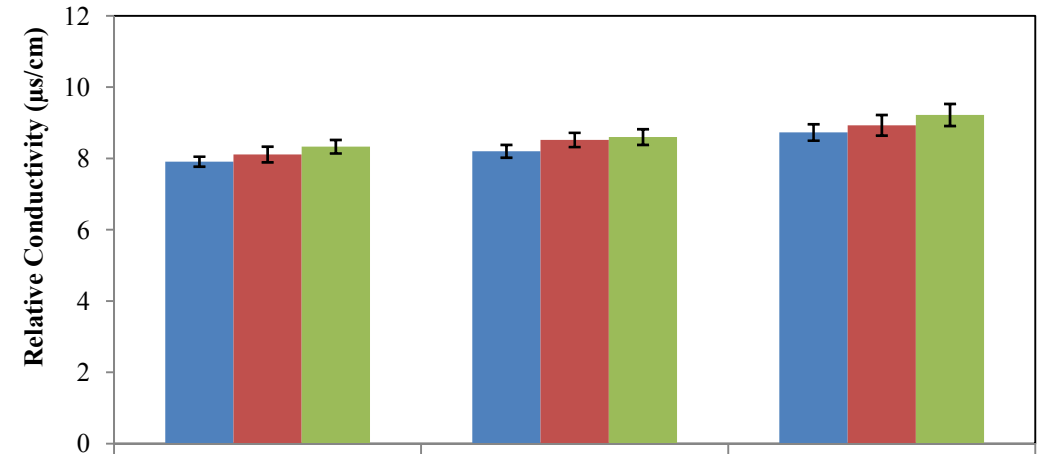
Treatments			Vitamin C retention (%)
Control			97±0.021[a]
Amplitude		Duration of Exposure (min)	
Ultrasound	25%	10	97±0.010[a]
		20	97±0.003[a]
		30	97±0.001[a]
	50%	10	97±0.006[a]
		20	97±0.009[a]
		30	97±0.005[a]
	75%	10	97±0.010[a]
		20	98±0.005[a]
		30	98±0.008[a]
Centrifugal Force	Centrifugal Speed (RPM)		
	500		97±0.002[a]
	1000		96±0.001[a]
	2000		96±0.004[a]
	3000		96±0.002[a]
	4000		96±0.006[a]

All data were obtained at least by triplicate and expressed as value ± SD.
Similar superscripts denote insignificant difference ($p>0.05$) at each column as compared with control.

6.3.6 Influence of the Ultrasound and Centrifugal Force on the Leaching of Cell Constituents into the Osmotic Medium

Figure 6.11 and 6.12 show that solution of treated samples had higher conductivity values than the solution containing untreated samples. The effect of the different pretreatments on the electrolyte release was significant ($P < 0.05$) where centrifugal force treated samples had slightly higher electrolyte release than those treated with ultrasonic wave. After 180 min of osmosis process, the highest conductivity values recorded were 9.2±0.15 µs/cm and 11.0±0.010 µs/cm for ultrasonic and centrifugal force treatments, respectively. With higher ultrasonic wave's amplitude and duration of exposure, the tissue damage becomes more significant, which is indicated by the increase of conductivity of the samples (Figure 6.11). This is also supported by the

data in Table 6.1, which show the lowest selectivity of tissue treated with higher ultrasonic wave's amplitude and duration of exposure. Conductivity values were higher at high centrifugal force indicating that more cell constituents were leached by the samples when treated at high centrifugal forces than at the lower ones (Figure 6.12). This fast leaching of soluble cell constituents probably altered the pressure gradient and chemical balance of the solution thus minimizing solute mass transfer into and out of the treated sample (Taiwo *et al*., 2002). This is also supported by the data in Table 6.4, which show the highest selectivity of tissue treated with centrifugal force. The obtained conductivity at the end of osmotic dehydration process was 2.82±0.15 µs/cm.



	25%	50%	75%
10 min	7.91	8.2	8.73
20 min	8.11	8.52	8.93
30 min	8.33	8.6	9.22

Figure 6.11. Effect of Ultrasonic Treatment on Relative Conductivity

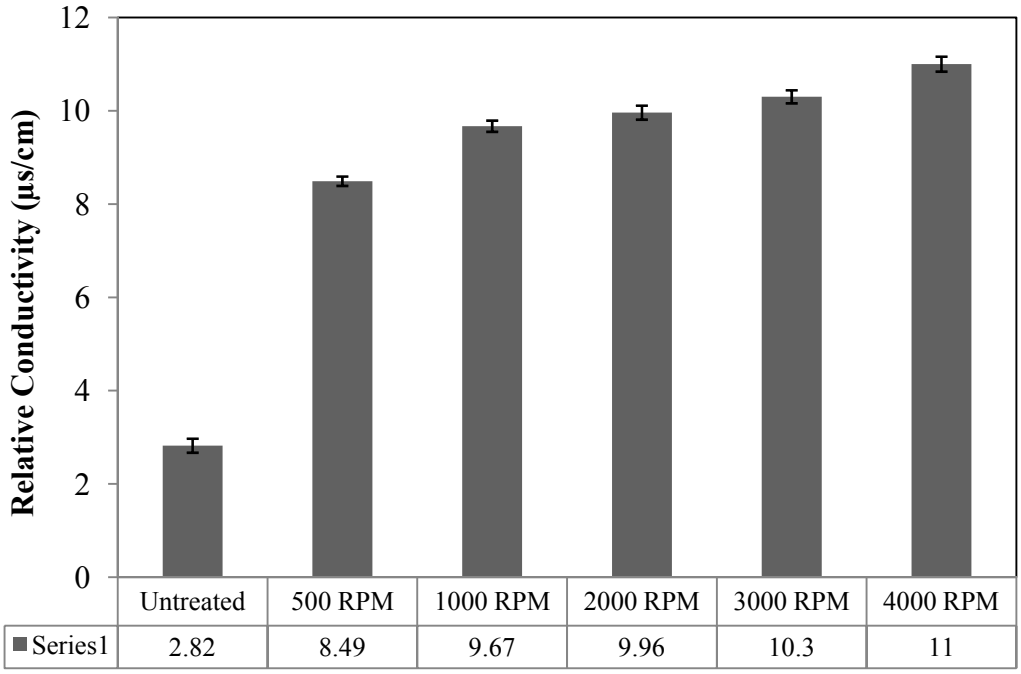


Figure 6.12. Effect of Centrifugal Force Treatment on Relative Conductivity

6.4 Summary

This study has shown that:

1. Ultrasonic wave's amplitude at the range of 25-75% significantly ($p < 0.05$) increased the kinetics of mass transfer during osmotic dehydration in which the duration of ultrasound treatment was only significant after 10 min, and not more than 30 min.
2. The influence of ultrasound on mass transfer can be explained by several mechanism including the degassing effect of sonication, generation of microjets in the direction of the surface, structural effects called ''sponge effect" and creation of micro-channels which caused by acoustic cavitation.

3. Application of centrifugal force significantly ($p < 0.05$) increased WR, WL and NMC but decreased SG compared to the static condition.
4. A good agreement between predicted and experimentally measured data was observed indicating that Peleg model was able to describe precisely the mass transfer during ultrasound- and centrifugal force- assisted osmotic dehydration ($R^2 > 90$).
5. Application of ultrasonic waves during osmotic dehydration led to higher ''L'' and lower ''a'' and ''b'' values which represented the decrease in the degree of browning discoloration during osmotic dehydration. On the other hand, application of centrifugal force during osmotic dehydration was found to decrease all color parameters (''L'', ''a'' and ''b'') insignificantly ($p > 0.05$) compared to untreated osmodehydrated seedless guava.
6. Hardness, area under the curve and initial modulus were significantly ($P < 0.05$) decreased by ultrasonic and centrifugal force treatments. Furthur analysis shows that a product obtained with centrifugal treatment is slightly less affected than ultrasonic treatment.
7. To allow the evaluation, comparisons and to quantify the influence of the treatments during osmotic dehydration on quality attributes, it is useful to estimate the kinetics of quality parameter changes.
8. The experimental data of quality parameters for ultrasonic and centrifugal force treated seedless guava fitted well to a zero order kinetics model ($R^2 > 0.77$).

CHAPTER VII

GENERAL CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE RESEARCH

7.1 Conclusions

Dehydration of seedless guava using osmotic process has been studied. Results obtained in this study demonstrate the effect of concentration and temperature of osmotic solution on dehydration kinetics of seedless guava cubes. Higher values of solution concentration (50%w/w) and temperature (50 ºC) resulted in higher flows of water (0.39±0.001g/g) and solid (0.080±0.006 g/g) through the seedless guava cubes. The comparisons between experimental and predicted values showed a good agreement (high values of R^2 and small values of chi-square, RMSE and E) indicating that Peleg model is adequate for predicting the kinetics of mass transfer during osmotic dehydration. In this way, the Peleg model allows the calculation of the initial and equilibrium mass transfer rates constants during osmotic dehydration, and consequently it can be used as a useful tool in the design and control of the corresponding industrial operation. Osmotic dehydration of seedless guava was optimized with respect to sucrose concentration, temperature and immersion time by using RSM. Second order polynomial reduced models were obtained for predicting WR, SG, and WL. The optimal conditions were temperature of 33 ºC, sucrose concentration of 30% w/w, and 179 min of immersion time to get minimum SG and maximum WR and WL. At these optimum values, WR, WL and SG were found to be 0.15 g/g, 0.20 g/g and 0.03 g/g, respectively. Therefore, response surface

methodology was effective for optimizing process parameters for the osmotic dehydration of seedless guava cubes.

Alteration of fruit's quality is the most drastic for product acceptance. Thus, color and texture of seedless guavas were evaluated during osmotic dehydration. Sucrose concentration and temperature significantly affect the quality attributes evaluated for seedless guava cubes. Color of fresh seedless guava darkened through the osmotic dehydration process. Darkening and loss of greenness and yellowness, as indicated by ''a'' and ''b'' values, accompanied the osmotic dehydration process. High concentration of peroxidase enzyme confirmed the enzymatic browning reaction during osmotic dehydration. Analysis of the mechanical parameters revealed a great decrease in the hardness (32%) and elasticity (55%) of seedless guava samples especially at higher dehydration levels. To quantify the influence of the process variables on the quality changes during osmotic dehydration, it is useful to simulate the kinetics of quality changes. The rates of quality changes followed zero-order kinetics (R^2>0.88) and their temperature dependency were evaluated by using Arrhenius relationship. Rate constants for ''L'', ''a'' and ''b'' as well as hardness value increased with increase in sucrose concentration and temperature. Rate constant for ''b'' value was more temperature sensitive among the other color parameters (E_a=30.03-41.49 KJ/mol). Temperature sensitivity of rate constant for ''b''value decreased with increase in sucrose concentration. Models for rate constants of ''L'', ''a'', ''b'' and hardness as a function of absolute temperature and sucrose concentration were found. These models explain at least 91% of the variability in quality parameters changes. Determination of kinetic parameters can be

useful for optimizing the end-over-end osmotic dehydration of seedless guava to obtain maximum retention of quality attributes.

The results of chapter 3 showed that the obtained values of mass transfer terms (20% WL and 3% SG) at the studied range of process variables were not in accordance with an efficient osmotic dehydration process in which 40–60% WL and <10% SG are mostly aimed (Eren and Kaymak-Ertekin, 2007). In order to improve the rate of mass transfer, a number of enhanced methods such as hot water blanching, thermosonication, ultrasound and centrifugal force were applied. The coupling of hot water blanching with osmotic dehydration under optimized condition makes it possible to obtain higher water (up to 36%) and solid (up to 6%) transfer at a moderate temperature of 33 °C. This can be explained by the ability of heat to permeabilize the cellular membranes efficiently reflected by the increase in the conductivity of osmotic medium and the decrease of osmotic dehydration selectivity. Unfortunately, significant reduction in color parameters of hot water pretreated osmodehydrated samples including lightness (19%), greenness (20%) and yellowness (32%) were found compared with untreated osmotically dehydrated samples. The similar trend was observed for hardness, area under the curve and initial modulus which decreased 38%, 46% and 38%, respectively. Therefore, for the first time, the simultaneous application of heat and ultrasonic waves (thermosonication) at different levels of intensity was investigated to reduce the intensity of heat treatments. The ANOVA result indicated that 50 and 75% of ultrasonic wave's amplitude at 90 ºC had significant ($p < 0.05$) effect on WR, WL and NMC whereas the studied range of ultrasonic wave's amplitude did not have significant ($p > 0.05$) effect on SG. Application of thermosonication treatment

(90ºC+75% amplitude) compared with hot water treatment at 90 ºC lead to increase WR, WL and NMC around 4%, 4% and 10%, respectively. Therefore, slightly higher efficiency of thermosonication seems to be detected without significant ($p > 0.05$) changes of quality attributes when applied before the osmotic dehydration process. The highest diffrences between lightness, greenness, yellowness and hardness of thermosonically and hot water treated osmosed samples were 2%, 5%, 3% and 1%, respectively. Overall, it can be concluded that the thermosonication treatment can be an alternative method to the traditional hot water blanching process. Since heat can impair organoleptical and nutritional properties of foods, there is a great number of studies deal with searching for new techniques able to reduce the intensity of heat treatments. Due to this fact, application of ultrasonic waves and centrifugal force during osmotic dehydration were investigated. The results revealed that ultrasonic technology can be carried out at moderate solution temperature to obtain higher WL (23%) and SG (3.5%) and obtaining the final product less affected by thermal treatment. In agreement with some reports, osmotic dehydration combined with centrifugal force is more favorable for increasing WL (up to 34%), retarding SG, and retaining quality parameters of seedless guava. It was found that combination of centrifugal force with osmotic dehydration leads to decrease in hardness of samples (5%) whereas there is no significant ($p<0.05$) effect on color of samples.

7.2 Recommendations

From the results of this study, it can be recommended that further studies are necessary to develop osmotic dehydration process. Laboratory studies have

demonstrated that osmotic dehydration has the potential to produce high quality products compared to other traditional processes. The industrial application of the process faces engineering problems related to the use of highly concentrated sugar solutions. The highly concentrated solution creates two major problems including high viscosity of solution which needs great agitation to decrease the resistance to the mass transfer and floating of the product because of the great difference in density between the solution and fruit. Microbiological safety of the process, storage ability and sensorial evaluation of final product are important aspects which should be studied thoroughly before further industrial development. Incorporation of fuzzy logic, neural networks, and other novel techniques for process control may enhance development of osmotic dehydration. There are few reports on centrifugal osmotic dehydrtation. To our knowledge, centrifugation is technically possible but further work is needed to investigate the effect of variables such as rotational speed, temperature and osmotic solution concentration on centrifugal osmotic dehydration. Finally, due to this fact that color and texture plays a principle role in designing a new food more studies are necessary to control the darkening and softening process during osmotic dehydration.

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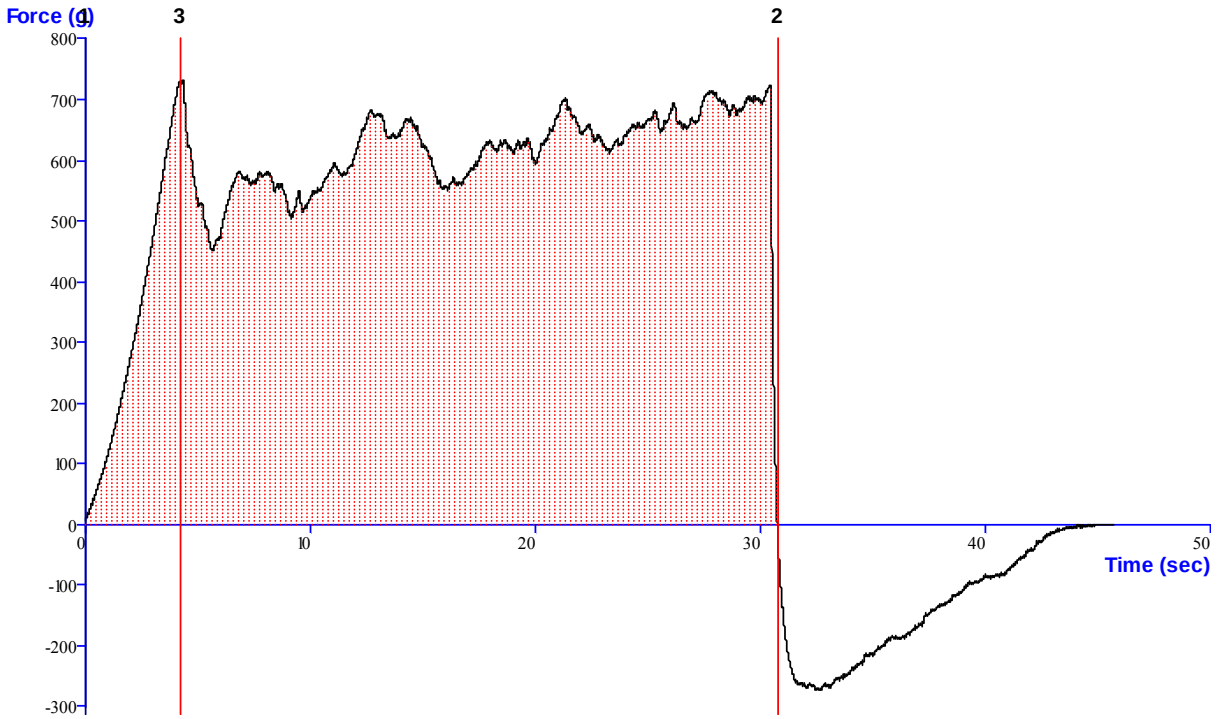
Appendix 1



Typical force-time curve after compression of seedless guava

BIODATA OF STUDENT

Ali Ganjloo was born on January 19th, 1981 in Mashhad city, Khorasan Razavi province, Iran. He completed his primary and high school education in his hometown, Mashhad. He received his Bachelor degree (B.Sc), in field of Food Science and Technology, from the Urima University, Iran, in 2004. He recived his Master of Science (Msc), in field of Food Science and Technology, from the Ferdowsi University of Mashhad, Iran, in 2006 and joined the Faculty of Food Science and Technology, Universiti Putra Malaysia to continue his study to Doctor of Philosophy (PhD) in field of Food Technology.

LIST OF PUBLICATIONS

Scientific Journal Publication:

1. Kinetics of Crude Peroxidase Inactivation and Color Changes of Thermally Treated Seedless Guava (*Psidium guajava* L.). Food Bioprocess Technology. DOI 10.1007/s11947-009-0245-4.

2. Kinetics Modelling of Mass Transfer using Peleg's Equation During Osmotic Dehydration of Seedless Guava (*Psidium guajava* L.): Effect of Process Parameters Food Bioprocess Technology. DOI 10.1007/s11947-011-0546-2.

3. Modelling the Kinetics of Peroxidase Inactivation and Colour Changes of Seedless Guava (*Psidium guajava* L.) During Thermal Treatments. World Applied Sciences Journal. 7 (1): 105-112, 2009

4. Mathematical modelling of mass transfer during osmotic dehydration of seedless guava (*Psidium guajava* L.) cubes. International Food Research Journal 18(2):xx-xx, 2011.

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6. Effect of heat and thermosonication on kinetics of peroxidase inactivation and vitamin C degradation in seedless guava (*Psidium guajava* L.). International Food Research Journal 18(3):xx-xx, 2011.

Proceeding of Conference/Seminar:

1) Feasibility of high-intensity ultrasonic blanching combined with heating for peroxidase inactivation of seedless guava

Ali Ganjloo, *Russly Abdul Rahman, Jamilah Bakar, Azizah Osman, Mandana Bimakr.*

National Conference on Food Science and Technology, Mashhad, Iran.2008

2) **Osmotic dehydration kinetics of thermally treated seedless guava (*Psidium Guajava* L.).**

Ali Ganjloo, *Russly Abdul Rahman, Jamilah Bakar, Azizah Osman, Mandana Bimakr.*

11th Asean Food Conference. 21-23 October 2009. The Rizqun international hotel, Bandar Seri Begawan Brunei Darussalam.

3) **Feasibility of novel methodologies for peroxidase inactivation: effect of kinetics.** ***Ali Ganjloo***, *Russly Abdul Rahman, Jamilah Bakar, Azizah Osman, Mandana Bimakr.*

Agriculture Congress, Tropical Agriculture in a Changing Climate and Energy Scenario. 27-29 October 2009. Palace of the Golden Horses, Seri Kembangan, Selangor, Malaysia.

4) **Kinetics of osmotic dehydration of seedless guava (*Psidium Guajava* L.).**

Ali Ganjloo, *Russly Abdul Rahman, Jamilah Bakar, Azizah Osman, Mandana Bimakr.*

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5) **Feasibility of ultrasonic wave's amplitude combined with heat for enhancing mass transfer during osmotic dehydration of seedless guava.**

Ali Ganjloo, *Russly Abdul Rahman, Jamilah Bakar, Azizah Osman*, *Mandana Bimakr.*

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6) **Modelling the kinetics of mass transfer and color changes during osmotic dehydration of thermosonically pretreated seedless guava (*Psidium Guajava* L.).**

Ali Ganjloo, *Russly Abdul Rahman, Jamilah Bakar, Azizah Osman*, *Mandana Bimakr.*

1st International congress on Food Technology, 3-6th November 2010. Antalya, Turkey.

7) **Mathematical Modelling of Mass Transfer during Osmotic Dehydration of Seedless Guava Cubes**

Ali Ganjloo, Russly Abdul Rahman, Jamilah Bakar and Azizah Osman

International Conference on Food Research. 22-24 November 2010. Putrajaya, Malaysia.

8) **Effect of Temperature on Mechanical and Optical Changes Kinetics of Seedless Guava Subjected to Osmotic Dehydration**

Ali Ganjloo, *Russly Abdul Rahman, Jamilah Bakar, Azizah Osman and Mandana Bimakr.*

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