

**MICROWAVE OSMOTIC DEHYDRATION OF MANGO CUBES UNDER
CONTINUOUS FLOW MEDIUM SPRAY CONDITIONS USING SOLUTE MIXTURES**

By

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MICROWAVE-OSMOTIC DEHYDRATION USING SOLUTE MIXTURES

ABSTRACT

Microwave osmotic dehydration (MWOD) has ability to enhance moisture loss (ML) and limit solids gain (SG) as compared with conventional OD treatments. MWOD has been shown to be effective both in medium immersion mode (MWODI) and medium spray mode (MWODS) with MWODS showing better performance than MWODI. The current study was focused on MWODS for further enhancing SG reduction by using selected solute mixtures.

The first study was focused on comparing the effectiveness of sucrose (S) solute with sucrose+dextrose (S:D) and sucrose+maltodextrin (grade 10DE) (S:MD) combinations for MWODS process with mango cube using three input variables (temperature, concentration and composition of solute mixtures) on mass transfer kinetics and post-MWODS product quality. The study demonstrated an enhanced ML/SG ratio with S:MD (85:15). This combination also gave a product with better texture and appearance factors. In further study, different grades of MD (10, 15 and 18DE) were compared for performance evaluation with MWODS, where MD 10DE was found to perform better than other two grades in facilitating better ML, limit SG and improve ML/SG ratio. The Azuara model was also shown to fit the experimental data for mass transfer kinetics ($R^2 > 0.92$).

The next study was to optimize MWODS process using sucrose+maltodextrin (10DE) (S:MD) using CCRD design, which included different proportions of S:MD (sucrose: maltodextrin= 100:0 to 80:20), temperatures (33-66.7°C) and concentrations (33-66.7%). Overall, it was found that S:MD in 84:16 proportion showed better mass transfer characteristics as well as better product quality. Osmotically dried products need to be further dried to a lower moisture content using a finish drying technique to provide stability to the product. Finish air drying was applied after MWODS pretreatment using S:MD (85:15) evaluate the influence on the quality of the dry product. A CCRD was used with variables temperature, concentration, flow rate and contact time as an input variable and the process was optimized based on mass transfer, quality characteristics and physical properties of the finished dried product. The optimized conditions were: temperature, 51.7°C; solute concentration, 58.5%; contact time, 30.6 min and solution flow rate of 1.8 L/min.

Microwave vacuum drying (MWVD) was also evaluated for finish drying to see if it would give a quality advantage with mango cubes MWODS pretreated as before. In addition to other quality parameters, a scanning electron microscope was also used for observing structural changes at the surface of the treated product. It was found that the MWODS pre-treated mangoes with S:MD followed by MWVD gave the shortest overall drying time and better structural stability and quality characteristics positioning itself next to the freeze-drying method (considered the best).

Overall, this research contributes to a better understanding of mass transfer behavior of MWODS employed with different solute mixtures, and for comparing finish drying under conventional air drying, MWVD and freeze-drying conditions. Sucrose and maltodextrin mixtures yielded much higher ML, limited SG and improved ML/SG ratio and product quality in dried mango cubes than has been reported in earlier studies not using MW treatment.

RÉSUMÉ

La déshydratation (OD) par micro-ondes (MWOD) a la capacité d'accroître la perte d'humidité (ML) et de limiter le gain en solides (SG) par rapport aux traitements OD classiques. MWOD s'est avéré efficace tant en mode d'immersion moyenne (MWODI) qu'en mode de pulvérisation moyen (MWODS), avec une performance supérieure à celle de MWODI. La présente étude était axée sur les MWODS afin d'améliorer encore la réduction de la SG en utilisant des mélanges de solutés sélectionnés.

La première étude s'est concentrée sur la comparaison de l'efficacité du soluté de saccharose (S) avec le saccharose+dextrose (S:D) et le saccharose+maltodextrine (grade 10DE) (S:MD) pour le procédé MWODS avec le cube de mangue en utilisant trois variables d'entrée (température, concentration et composition des mélanges de solutés) sur la cinétique de transfert de masse et la qualité des produits post-MWODS. L'étude a démontré un rapport ML/SG amélioré avec S:MD (85:15). Cette combinaison a également donné un produit avec de meilleurs facteurs de texture et d'aspect. Dans une étude plus approfondie, différentes qualités de MD (10, 15 et 18DE) ont été comparées pour l'évaluation des performances avec MWODS, où MD 10DE s'est avéré plus performant que les deux autres grades pour faciliter une meilleure ML, limiter la SG et améliorer le rapport ML/SG. Il a également été montré que le modèle Azuara correspond aux données expérimentales pour la cinétique de transfert de masse ($R^2 > 0,92$).

L'étude suivante consistait à optimiser l'application de MWODS sur des cubes de mangue utilisant du saccharose+maltodextrine (10DE) (S:MD) selon le schéma CCRD, qui comprenait différentes proportions de S: MD (saccharose: maltodextrine = 100: 0 à 80:20).), les températures (33.3⁰C à 66.7⁰C) et les concentrations (33% à 66,7%). Dans l'ensemble, il a été constaté que le rapport S:MD dans la proportion de 84:16 présentait de meilleures caractéristiques de transfert de masse ainsi qu'une meilleure qualité du produit. Les produits séchés par osmose doivent être davantage séchés jusqu'à une teneur en humidité inférieure en utilisant une technique de séchage de finition pour assurer la stabilité du produit. Le séchage à l'air fini a été appliqué après le prétraitement MWODS à l'aide de S: MD (85:15) pour évaluer l'influence sur la qualité du produit sec. Un CCRD a été utilisé avec des variables de température, de concentration, de débit et de temps de contact comme variable d'entrée et le processus a été optimisé en fonction du transfert de masse, des caractéristiques de qualité ainsi que des propriétés physiques du produit séché fini. Les conditions optimisées étaient les

suivantes: température 51.7⁰C; concentration en soluté, 58.5%; temps de contact, 30.6 min et débit de la solution de 1.8 L/min.

On a également évalué le séchage de finition par séchage sous vide à micro-ondes (MWVD) afin de déterminer si cela donnerait un avantage qualitatif avec les cubes de mangue MWODS prétraités comme auparavant. Outre d'autres paramètres de qualité, un microscope électronique à balayage a également été utilisé pour observer les changements structurels dans le produit traité. Il a été constaté que les mangues prétraitées MWODS avec S:MD suivies par MWVD donnaient le temps de séchage global le plus court et une meilleure stabilité structurelle ainsi que des caractéristiques de qualité se positionnant au niveau de la méthode de lyophilisation (considérée comme la meilleure).

Globalement, cette thèse de recherche contribue à une meilleure compréhension du comportement de transfert de masse des MWODS utilisés avec différents mélanges de solutés et à la comparaison du séchage de finition dans des conditions de séchage à l'air conventionnel, de MWVD et de lyophilisation. Les mélanges de saccharose et de maltodextrine ont produit une ML beaucoup plus élevée, une SG limitée et un rapport ML/SG amélioré ainsi que la qualité du produit dans les cubes de mangue séchés par rapport aux études menées, sans utilisation du traitement de MW.

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CONTRIBUTIONS OF AUTHORS

Some parts of this thesis research have been presented at scientific conferences and submitted/prepared as manuscripts for publication. The author of this thesis was responsible for the design of experiments, experimental work, data analysis and manuscripts preparation under the supervision of Dr. Ramaswamy who helped in defining the problem and providing direct advisory input as the research work progressed.

Part of this thesis has been published or submitted as follows

Peer-Reviewed Publications

Shinde, B., and Ramaswamy, H.S., 2019. Evaluation of mass transfer kinetics and quality of microwave-osmotic dehydrated mango cubes under continuous flow medium spray (MWODS) conditions in sucrose syrup as moderated by dextrose and maltodextrin supplements. *Drying Technology*, 1-15.

Shinde, B. and Ramaswamy, H.S. 2020. Kinetic modeling of sucrose and maltodextrin (10-18 DE) moderated mass transfer rates in mango cubes during microwave osmotic dehydration under continuous medium spray conditions. *Drying Technology*, 1-13.

Shinde, B. and Ramaswamy, H.S. Optimization of maltodextrin (10DE) - sucrose moderated microwave osmotic dehydration of mango cubes under continuous flow spray mode (MWODS) conditions. Paper submitted to LWT journal. (*Paper in review*)

Shinde, B. and Ramaswamy, H.S. 2020. Evaluation and optimization of air drying quality of MWODS treated mango cubes during using maltodextrin moderated sucrose solution. (*Ready to be submitted*)

Shinde, B. and Ramaswamy, H.S. 2020. Effect of microwave vacuum drying on mangoes (*Mangifera indica*) pretreated with microwave osmotic dehydration under continuous flow maltodextrin moderated spray condition (*In preparation*)

Conference Presentations

Shinde, B., and Ramaswamy, H.S., 2016. Evaluation of the influence of osmotic solutes on microwave-osmotic dehydration of mango under continuous flow medium spray (MWODS) conditions. Northeast Agricultural and Biological Engineering Conference (NABEC) 2016. July 31- August 3, 2016, Orono, ME, USA. (Poster presentation)

Shinde, B., and Ramaswamy, H.S., 2017. Optimization of microwave-osmotic dehydration under spray condition (MWODS) of mango (*Mangifera indica*) using combinations of sucrose and maltodextrin as the osmotic agents. Northeast Agricultural and Biological Engineering Conference (NABEC) 2017. Jul 30– Aug 2, Groton Connecticut, USA. (Poster presentation)

Shinde, B., and Ramaswamy, H.S., 2018. Microwave osmotic dehydration of mango cubes: Influence of osmotic solute mixtures. American society of agricultural and biological engineering, (ASABE) 2018. July 29 to August 01, 2018, Detroit, Michigan, USA. (Paper presentation).

Shinde, B., and Ramaswamy, H.S., 2018. Effect of microwave osmotic pre-treatment with sucrose and maltodextrin solute mixture on finished air drying of mangoes by using CCRD. Northeast Agricultural and Biological Engineering Conference (NABEC) 2018. July 15-18, 2018 in Morgantown, WV, USA. (Poster presentation)

Shinde, B., and Ramaswamy, H.S., 2018. Effect of the osmotic solute mixture (sucrose: maltodextrin) on microwave osmotic dehydration under continuous flow medium spray-microwave vacuum process using ccrd. Canadian Institute of food science and technology (CIFST) 2018. May 27 to 29, 2018 in Niagara-on-the-lake, ON, Canada. (poster presentation)

Contribution to the knowledge

The significant contributions of this research are enriching scientific knowledge on microwave-osmotic and microwave-vacuum drying while applying a mixture of osmotic agents and understanding their effects on product quality and mass transfer kinetics of mangoes. The specific contributions to the knowledge of this thesis work are described below:

1. Microwave osmotic dehydration under continuous flow medium spray (MWODS) processing conditions was recognized before. During this research series the study was focused, *for the first time*, on the effect of different solute mixtures on MWODS, where osmotic agents were selected based on their molecular weights and studied for mass transfer kinetics as well as quality characteristics. The solute mixture sucrose+maltodextrin (10DE) was proven to deliver lower SG and better ML/SG performance amongst others.
2. Next the study involved the comparison between a higher molecular solute mixture (sucrose+maltodextrin (10DE), (sucrose+maltodextrin (15DE) and (sucrose+maltodextrin (18DE)) and their behavior on mass transfer kinetics, *again a first-time comparative study*. The solute mixture sucrose+maltodextrin (10DE) was shown to be most effective amongst all. It was also concluded that the solute mixture S:MD10DE produced satisfactory results by increasing K_w and reducing K_s when compared with other solute mixtures. It was also established that the Azura model can fit satisfactorily in MWODS treatment, which concludes that the two-parameter Azuara model can be used to describe transient mass transfer kinetics.
3. Response surface methodology was used to evaluate the effects of process variables, especially the proportion of two osmotic solutes such as sucrose and maltodextrin, on the mass transfer kinetics of MWODS process and the desirability function along with conventional graphical methods were used to identify a range of optimum processing conditions based on selected optimization criteria. The CCRD models give a limited range of experimental runs over the factorial design, which made it desirable and very advantageous in this study. *Such studies using MWODS is a first-time research contribution.*

4. The effect of sucrose + maltodextrin solute mixture during MWODS pretreatments, on the subsequent air-drying method and the quality of finished dried product, was investigated using response surface methodology. The CCRD models were analyzed to understand the optimized processing conditions of MWODS pretreatment based on the mass transfer data along with the quality characteristics of the product. For a better understanding of the solute behavior, the mangoes were pretreated with sucrose, sucrose + dextrose and fresh (without pretreatment) and values were compared, where sucrose + maltodextrin presented superior results.
5. The MWODS pretreatment using sucrose+maltodextrin solute mixture was employed in tandem with MWV finished drying method to demonstrate the combined effect of two processes (MWODS and MWV). The parametric optimization of the process using CCRD models indicated that the temperature and concentration during MWODS along with magnetron power level of MWV tended to increase dehydration parameters (ML and SG) and to decrease quality parameters respectively.
6. Overall, the entire study of the influence of solute combination on mass transfer characteristics under microwave osmotic drying conditions had never been studied before and therefore contributes a significant addition to our state of knowledge in this area.

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NOMENCLATURE

a_w	Water Activity (dimensionless)
a_0^*, a^*	Chromaticity coordinates (red (+) to green (-)) of standard and sample, respectively (dimensionless)
b_0^*, b^*	Chromaticity coordinates (yellow (+) to blue (-)) of standard and sample, respectively (dimensionless)
C	Concentration (w/w)
Chewiness	mJ or grams (g)
E	Strength of the electric field (in V/m).
f	Frequency of the electromagnetic wave in Hertz (Hz)
F	The rate of transfer per unit area of cross-section (kg/m^2)
D	The diffusion coefficient (m^2/s)
Hardness	Grams (g) or Newtons (N)
J	$\sqrt{-1}$
k_w, k_s	Overall mass transfer coefficients for water and solute, respectively
L_0^*, L^*	Lightness of the standard and sample, respectively (dimensionless)
M_0, M_t	Sample mass (kg) at time zero, time t, respectively
M_{glucose}	Molecular weight of glucose
Mn	The number average molecular weight of the sample
MLe and SGe	Equilibrium values of moisture loss and solids gain
ML_t and SG_t	Moisture and solids fraction at time t
P	Energy developed per unit volume (in W/m^3)
S_0, S_t	Solids content (kg/kg) at time zero, time t, respectively
T	Absolute Temperature (Kelvin)
t	Time
V	Volume (m^3)
W	The water content ($\text{g H}_2\text{O}/\text{m}^3$)
Wr, Wd	Masses of the rehydrated and dry material (g), respectively.
X_0, X_t	Water fractions (kg/kg) at time zero, time t, respectively
x	The space coordinate measured normal to the section (m)

ΔE	Total color change
ϵ^*	Complex relative permittivity
ϵ'	Dielectric constant
ϵ''	Loss factor
ϵ_0	Absolute permittivity of vacuum (8.854188 x10-12 F m-1)

ABBREVIATIONS

AD	Air Dried
ANOVA	Analysis of Variance
ANN	Artificial neural networks
CCRD	Central Composite Rotatable Design
CI	Confidence Interval
COD	Conventional Osmotic Dehydration
D	Dextrose
DE	Dextrose equivalent
De	Effective diffusivity
DP	Degree of polymerization
DISP	Dewatering Impregnation Soaking Process
EM	Electromagnetic
FB	Fluidized bed
FD	Freeze Dried
HFCS	High Fructose Corn Syrup
HDM	Hydrodynamic Mechanism
HELP	High-Intensity Electric Field Pulses
HHP	High Hydrostatic Pressure
LOX	Lipoxygenase
MD	Maltodextrin
MFB	Microwave Fluidized Bed
MFD	Microwave Freeze Drying
ML	Moisture Loss
MPSVD	Microwave Vacuum Spouted Bed Process
MSB	Microwave Spouted Bed
MW	Microwave
MWODI	Microwave Osmotic Dehydration under Continuous Flow Medium Immersion

MWODS	Microwave Osmotic Dehydration under Continuous Flow Medium Spray
MWV	Microwave-Vacuum
MVD	Microwave-vacuum drying
NMC	Normalized Moisture Content
NSC	Normalized Solids Content
OD	Osmotic Dehydration
PEF	Pulsed electric field
POD	Peroxidase
Pred-R²	Predicted Coefficient of Determination
PSMFD	Pulse Spouted Bed Microwave Freeze Drying
PVOD	Pulsed Vacuum Osmotic Dehydration
R²	Coefficient of Determination
RH	Relative Humidity
RHC	Rehydration Capacity
RMS	Root Mean Squared
RSM	Response Surface Methodology
SG	Solids Gain
VI	Vacuum Impregnation
VOD	Vacuum Osmotic Dehydration
VD	Vacuum Dried
WL	Water Loss

Chapter 1

Introduction

Osmotic dehydration (OD) is one of the most popular pretreatments prior to drying operations which have been described as a treatment to allow the creation of novel food products through the incorporation of solutes (Ramaswamy & Tola, 2011). Osmotic dehydration (OD) is a water removal technique, in which horticultural products such as fruits and vegetables are immersed in a hypertonic solution or solids such as granular sugar or salt, for a physical separation of moisture from the fruit to the concentration solution due to osmotic forces. Osmotic dehydration has potential to reduce quality losses by using a relatively low temperature and to reduce energy needs, as water does not go through phase change (Bolin et al., 1983). Osmotic dehydration of fruits before drying can enhance taste and maintain their structural characteristics (Udomkun et al., 2015). The osmotic dewatering partly removes water and produces an intermediate moisture product with lower water activity, at which most of the chemical, physical and biological activities, which deteriorate the foods, are ceased (Piasecka et al., 2013). Osmotic pretreatment reduces enzyme activity and product water activity along with minor changes in product characteristics (Giraldo et al., 2003). It also reduces enzymatic browning (Giraldo et al., 2003) and often retains or improves color (Conway et al., 1983; Giannakourou & Taoukis, 2003; Giraldo et al., 2003) and texture (Huxsoll, 1982; Robbers et al., 1997; Talens et al., 2002) of the food product. In recent years, the development of intermediate moisture food using osmotic dehydration has received many appraisals among consumers due to minimal processing (Ahmed et al., 2016; da Silva et al., 2014; Raoult-Wack, 1994; Sutar et al., 2012). Osmotic dehydration is one of the most employed techniques to improve the organoleptic and nutritional properties of foods, and it has been utilized to improve fruit quality (e.g., color, texture, flavor, and nutrients (Azam et al., 2013; Chakraborty & Samanta, 2015).

Furthermore, the osmotic agent is one of the critical factors in OD, which accelerates the process when used in proper proportion. Most commonly, sucrose, fructose, and sodium chloride are the major dehydrating agents for fruits. Along with these solutes, other solutes of interest such as antioxidants or preservatives could be added to the osmotic solution (Nagai et al., 2015). In the similar context, various osmotic agents such as sucrose, glucose, fructose, corn syrup, sodium chloride, sorbitol, in their combination, have been studied during osmotic dehydration process (İspir & Toğrul, 2009b). Any solute or solvent that is soluble in water can be used (e.g.,

starch, ethanol, polyols, and dextrose) as dewatering and impregnating agents (Lenart & Flink, 1984). Some authors have reported the use of ternary solutions (mixtures of sugars and salts) as an advantageous method, leading to higher water losses with lower solids gains and also providing an increase in the total solution concentration, without reaching the saturation limits (Bohuon et al., 1998; Medina-Vivanco et al., 2002; Qi et al., 1998). The application of non-conventional carbohydrates such as oligofructose and maltodextrin showed better protection when concerning color and sensory attributes than conventional osmotic agents (glucose) (Dermesonlouoglou et al., 2016). The osmotic agents and their mixtures have a substantial influence of osmotic dehydration efficiency.

In addition, the research is gaining interest in developing new techniques to improve the traditional method of osmotic dehydration, especially to enhance the osmotic dehydration rates. Li and Ramaswamy investigated a novel process of ‘Microwave Osmotic Dehydration under Continuous Immersion medium flow’ (MWODI)” which was further modified by Azarpazhooh and Ramaswamy and introduced as ‘Microwave Osmotic Dehydration under medium flow continuous Spray condition’ (MWODS) (Azarpazhooh & Ramaswamy, 2009b; Li & Ramaswamy, 2006c). These methods were aimed to enhance one-way mass transfer, i.e., improving moisture loss and restricting the solids gain. The application of microwave energy successfully results in rapid moisture reduction within a short time. The principle behind MWODS method is, it generates heat by exciting dipolar (water molecules) and polarizing (ionic salts) components which ultimately produces heat within the samples and forces water to move out from the food product (Wray & Ramaswamy, 2013). Li and Ramaswamy (2006) investigated the mass transport coefficients under microwave osmotic dehydration (MWOD, immersion medium) and reported that the osmotic dehydration under microwave heating made it possible to obtain a higher diffusion rate of moisture transfer at lower solution temperatures (Orsat et al., 2007). Azarpazhooh and Ramaswamy (Azarpazhooh & Ramaswamy, 2009a, 2009b) studied diffusion model for MWOD (spray condition) and concluded that higher equilibrium moisture loss and lower solids gain were recorded in MWODS method when compared with other osmotic dehydration methods (Sosa-Morales et al., 2010).

Further, since the moisture content of osmotically dehydrated products is still generally high, they are usually coupled with other finishing drying processes such as air drying, vacuum

drying or freeze-drying to reduce the moisture content to the final target level. The main objective of dehydration is to remove moisture to a point where a product is microbiologically and enzymatically stable in order to limit product deterioration during storage and allow its incorporation into different food products (Nijhuis et al., 1998; Van Nieuwenhuijzen et al., 2001). Water removal also lowers transportation and storage costs by reducing the weight of the product (Bolin et al., 1983; Rastogi et al., 2002). Generally, the combination osmotic drying and a second stage air drying has been demonstrating to yield a better-quality product and energy savings (Azuara et al., 2002). Hence, the combination of OD followed by finished drying is of gaining importance. Traditional hot air drying (Mandala et al., 2005; Piotrowski et al., 2004), vacuum drying (Dixon & Jen, 1977), freeze-drying (Hawkes & Flink, 1978), microwave heating and processing applications (Nelson & Datta, 2001), microwave-vacuum drying (MVD) (Wray & Ramaswamy, 2015b) were also found to produce better quality product when applied to OD treated samples. The microwave vacuum drying is gaining attraction due to its benefits over other drying methods. In MVD method, the boiling point of water is significantly reduced under vacuum, and therefore, the sample can be maintained at a lower temperature while still evaporating moisture (Beaudry et al., 2004). The microwave energy involved in MVD heats up the product directly instead of relying on air as a heat transfer medium and there is no energy wasted in pre-heating the oven cavity and air to the requisite temperature. The most difficult part to remove the moisture from core of the sample can be achieved quickly using microwave vacuum drying (Bouraoui et al., 1993). Microwave-vacuum drying limits product degradation that would take place due to exposure to oxygen and high temperatures by drying in a reduced oxygen atmosphere (Drouzas & Schubert, 1996).

Research Objectives

Therefore, the overall objective of this thesis was to evaluate the influence of osmotic agents on mangoes (*Mangifera indica*) during microwave-osmotic dehydration under continuous flow medium spray (MWODS) conditions as well as finished dried product using microwave-vacuum drying method. The specific objectives were as:

- I. To understand the influence of dextrose and maltodextrin addition to sucrose solution in different proportions on the mass transfer and quality factors of mango cube under MWODS processing conditions
- II. To investigate moisture and solids transfer rate and to evaluate the Azuara model (both moisture loss and solids gain) during MWODS to compare the performance of four different solute mixtures- sucrose, sucrose+maltodextrin 10DE, sucrose + maltodextrin 15 DE and sucrose + maltodextrin 18 DE.
- III. To optimize the maltodextrin moderated sucrose solution during microwave osmotic dehydration of mango cubes under continuous flow spray mode (MWODS) conditions.
- IV. To evaluate the MWODS behavior using complex solute on the finished air drying and to optimize the processing parameters using central composite rotatable design.
- V. To evaluate the effect of microwave vacuum drying on the quality of mango cubes pretreated with microwave osmotic dehydration under continuous flow maltodextrin moderated spray conditions and compare them with other drying methods.

PREFACE TO CHAPTER 2

The following chapter aims to provide the background information on dehydration, as related to osmotic dehydration and microwave-assisted techniques. This Chapter 2 "Literature Review" presents an overview of dehydration by highlighting the factors influencing the process. Hence, this chapter is sectioned into several parts which will explain about the fruit mango, osmotic dehydration process, factors influencing OD with emphasis on osmotic agents and finished drying methods with a particular focus on the application microwave energy.

CHAPTER 2

LITERATURE REVIEW

2.1 Mango (*Mangifera indica*)

Mango is the most heavily produced and popular tropical fruit in the world after banana (Mitcham & McDonald, 1992; Ribeiro & Schieber, 2010). Nevertheless, mango is less commercialized, when compared to the quantity produced due to the lack of awareness in developed countries. Furthermore, the tropical mango fruit has a typical concern while preserving the quality attributes and physical characteristics specifically with flavor degradation, as this fruit is widely popular due to its taste, color, and overall sensory attribute (Bernardi et al., 2009; Torezan et al., 2002). Apart from the popularity of the mango fruit, it was chosen for this study concerning that it is an essential source of an important source of macro-, micronutrients, and a broad range of phytochemicals which might get destroyed during the harsh drying process. To maintain the fruit quality for reasonably long periods, controlled temperatures and modified storage atmospheres are required, which, in many cases, is not fully exploited in producing countries. However, the demand for healthy, natural and tasty processed food, available throughout the year, is continuously increasing, not only for finished products, but also for ingredients to be included in complex foods such as ice-creams, cereals, bakery, and confectionery.

Mango is the most heavily produced tropical fruit in the world. The largest mango producing countries are India (40%), China (32%) and Mexico; the leaders in the international trade being Mexico, the Philippines, and India. Global production of mango was about 30 million tons (MT) in 2010, and it is the second-largest tropical fruit crop in the world, after banana. According to FAO 2010 statistics, mango imports/exports were about 1.5 MT in 2010, and are expected to grow with time (Galán Saúco, 2013). Nevertheless, mango processing is lightly commercialized, when compared to other fruits or in relation to the quantity produced. The Dried mango is the commonly preserved form of the fruit in Asia, and it has also become increasingly popular in Europe (Berardini et al., 2005; Tedjo et al., 2002). However, conventionally dried mango fruit has undesirable tough texture, poor color and cooked flavor with a loss of nutritive value, which reduces its economic importance (Durance et al., 1999). Since dehydration prone to reduce the overall acceptability of the product, increased demand for

minimally processed mango products has been increased with a prolonged shelf-life, through maintaining a healthy and tasty experience. Therefore, pretreatment such as osmotic dehydration can be an asset to retain quality and to reduce nutrient losses of processed fruits and vegetable (Heng et al., 1990; Mandala et al., 2005; Riva et al., 2005; Torreggiani & Bertolo, 2001). Aiming to better explore the mango fruit, the microwave osmotic dehydration was studied as a pretreatment prior to drying to obtain minimally processed products.

2.2 Osmotic dehydration:

Osmotic dehydration has been generally recognized as a dehydration pre-treatment which can result in better quality retention, better energy conservation, and a better acid to sugar ratio (Raoult-Wack, 1994) in dried fruits with superior sensory properties and such products have been used as useful ingredients in many processed food formulations or complex foods (Serenio et al., 2001; Torreggiani, 1993). Osmotic dehydration has the potential to remove water at low temperatures; in addition, it is an energy-efficient method, as water does not go through a phase change (Bolin et al., 1983). Osmotic dehydration, also called a “dewatering impregnation soaking process” (DISP), which involves the soaking of foods—mostly fruits and vegetables—in hypertonic salt or sugar or in a combined solution, to reduce the water content while increasing the solid soluble content (Torreggiani, 1993). The principle underlying OD is that water diffuses from dilute solution to concentrated solution (hypertonic solution) through a semi-permeable membrane till equilibrium is established. The raw material is placed into concentrated solutions of soluble solids having higher osmotic pressure, which causes the water and solute activity gradients across the cell membrane, the cell wall, and the surface of the tissue. The diffusion of water is associated with the simultaneous counter diffusion of solutes from the osmotic solution into the tissue. This contributes to a net opposite flux of water and solutes that allow the tissue to become concentrated with a determined ratio of solute gain/water loss (SG/WL) depending on process conditions (Chiralt & Fito, 2003). However, it is difficult to obtain a perfect semi-permeable membrane in food systems due to their complex internal structure, and there is always some solid diffusion into the food. Hence this process gives rise to, at least, two major simultaneous counter-current flows: an important water flow out of the food into the solution and a simultaneous transfer of solute from the solution into the food, which is both due to the water and solute activity gradients across the cell’s membrane (Kumar & Sagar, 2014).

The water transfer is generally accompanied by natural substances such as vitamins, flavors, fruit acids, pigments, saccharides, and mineral (Falade & Adedokun, 2007). As a consequence of this exchange, the product loses weight and shrinks. The cellular membrane of fruits and vegetables are composed mainly of parenchyma cells, freely allow the solvent molecules to pass through, but they also allow, to a lesser degree, the passage of some of the solute molecules, however, they are important in terms of final product quality (Dixon & Jen, 1977). Therefore, it is difficult to establish general rules about them. However, the most important terms to understand are osmotic pressure, plant tissue structure, and mass transport relationships (Islam & Flink, 1982b; Lerici et al., 1985).

2.2.1 Role of osmotic dehydration

Osmotic dehydration has been used in a sequence with other processing steps to achieve controlled changes of the original properties of the raw material (Torreggiani, 1993). While some treatments such as freezing have primarily a stabilizing effect, other steps such as partial dehydration, particularly osmotic dehydration allow structural, nutritional, sensory and other functional properties of the raw material to be modified. The differentiating feature of osmotic dehydration, compared to other dehydration processes, is the penetration of solutes into the food material (İspir & Toğrul, 2009a). So it is possible, to a certain extent, to change the food system formulation, making it applicable to further processing by, first adjusting the physicochemical composition of food by reducing water content, or adding water activity lowering agents; second incorporating ingredients or additives with antioxidant, or other preservative properties into the food; third, adding solutes of nutritional or sensory interest and fourth Providing a larger range of food consistency. According to temperature, time, type of syrup, surface, and the mass ratio of product to solution, osmotic dehydration can induce a different effect in the same raw materials.

2.2.2 Mass transfer phenomenon in osmotic drying

The semi-permeable membranes present in biological materials are the dominant resistance to mass transfer during osmotic dehydration. The cell membrane can change from being partial to totally permeable, leading to significant changes in tissue architecture (Rastogi et al., 2002). In fruits or vegetables, the cell wall membranes are living biological units, which can

stretch and expand under the influence of growth and turgor pressure generated inside the cells. A method of partial dehydration of fruits and vegetables by osmosis in sugar or salt solution take place due to the phenomenon of mass transfer. The kinetics of mass transfer in OD is a two-way exchange of solutes and soluble components. When plant cells are placed in a hypertonic solution, water removal starts from the surface that is in contact with the osmotic solution, resulting in cell disintegration (Rastogi et al., 2000). During osmosis, mass reduction is occurred due to the diffusion of a water molecule from sample to osmotic solution and infusion of sugar or salt from osmotic solution to the sample. It is reported that sugars penetrate to a depth of 2-3 mm into the plant tissue while changes in water content are observed up to 5 mm (Bolin et al., 1983; Lenart & Flink, 1984; Salvatori et al., 1999). The cell vacuole and the rest of the protoplasm will shrink, and plasmolysis occurs due to water diffusion from cell surface during osmosis. However, the interior surface of the material can remain in full turgor pressure. Whereas the turgor pressure gradient results in the detaching of the plasma membrane and the middle lamella due to the degradation or denaturation of the components of the middle lamella.

The cell debonding will occur during osmotic dehydration due to mass transport, as discussed by Lewicki and Porzecka-Pawlak (2005) (Lewicki & Pozecka-Pawlak, 2005). Consequently, the cell is damaged and reduces in size by the loss of water and contact between the outer cell membrane and the cell wall (Rastogi et al., 2000; Rastogi et al., 2002). On the other hand, an Extensive uptake of osmoactive substance results in the development of a concentrated solids surface layer posing an additional resistance to mass transfer (Lenart & Lewicki, 1987). Consequently, the porosity of the product will increase (Mayor et al., 2008), and the tissue shrinks because the amount of water flowing out is generally greater than the solutes diffusing in. Therefore, the weight of the foods will decrease, as will the water activity. Singh et al. (1998) conducted studies on mass transfer changes during OD of pear and different factors affecting on the mass transfer phenomenon (Singh et al., 1998). The effects of the process variables, such as sucrose concentration, processing the time and temperature, slice thickness, fruit to syrup ratio, and agitation of osmotic solution on percent weight reduction and total soluble solids were determined. It was observed that high sucrose concentration and temperature increased the percent weight reduction and total soluble solids. The best resume lights were achieved using 60% sucrose solution at 50⁰C. A thickness of 10 mm with fruit to syrup ratio of 1:4 was found to be suitable. Chandra and Kumari (2015) studied that the percent moisture loss, weight loss, and

solid gain increased with increase in sugar syrup concentration from 40–60⁰Brix while decrease with sample to sugar syrup ratio and then increased (Chandra & Kumari, 2015). Moisture content decreased more rapidly during initial stage of drying as compared to later part of drying in both drying conditions. Therefore, the weight of the foods will decrease, as will the water activity. It is reported that up to a 50% reduction in the fresh weight of fruits or vegetables may be brought about by osmosis (Kar, 2001; Rastogi & Raghavarao, 1997; Uddin et al., 2004).

2.2.3 Advantages of osmotic dehydration

It has been shown that osmotic pre-treatment improves the quality of dried products in terms of color, flavor, or aroma and texture as the process takes place under mild heat treatment (<50⁰C) resulting in products with superior sensory characteristics (Ponting, 1973) such as reduced heat damage to texture and color (Torreggiani, 1993) and increased retention of volatiles due to flavor retention property when sugar or sugar syrup is used (Dixon & Jen, 1977; Flink, 1975). Reduced discoloration of the fruit from enzymatic browning can be achieved during OD, since a product continuously immersed in the osmotic solution, making the process oxygen-free (Contreras & Smyrl, 1981; Ponting et al., 1966). This ultimately reduces the chances of enzymatic browning. Therefore, no need to use sulfur dioxide and blanching for protection against oxidative and enzymatic discoloration (Islam & Flink, 1982b; Rahman, 1992). OD is now considered a valuable tool in minimal processing of foods. It can be applied either as an autonomous process or as a processing step in alternative processing schemes leading to a variety of end products.

Osmotic dehydration is energy efficient as moisture is efficiently removed from a food product without a phase change (Bolin et al., 1983; Uddin et al., 2004). It also requires low operating cost as it can be conducted at low or ambient temperatures (Bolin et al., 1983). In addition, the product is processed in the liquid phase, generally giving good heat- and mass-transfer coefficients (Raoult-Wack, 1994). OD has been successfully used to reduce water activity of fruits and vegetables to about 0.9, keeping much of original quality. OD has been combined with conventional drying methods such as hot air drying to produce shelf-stable fruit products (Islam & Flink, 1982b; Rahman, 1992). OD has been combined with conventional drying methods such as freezing (Maestrelli et al., 2001; Tregunno & Goff, 1996), freeze-drying (Donsi et al., 2001), vacuum drying (Rahman & Mujumdar, 2007), air drying, osmo-convective

drying (Corzo et al., 2008; Islam & Flink, 1982b) and microwave drying (Orsat et al., 2007) are necessary in order to provide shelf stability to the product.

2.2.4 Impact of osmotic dehydration

Certain defects and difficulties are associated with osmotic dehydration that needs to be studied to improve its efficiency. These include soluble solid leaching, extensive solids uptake, and rapid water loss rates. Solute uptake and leaching of valuable product constituents often lead to substantial modification of the original product composition, with a negative impact on sensory characteristics and nutritional profile (Torreggiani, 1993). In natural food systems also, there is some leakage of solute (sugars, organic acids, minerals, salts, etc.) across the membrane. Though quantitatively negligible, it may be essential as far as organoleptic or nutritional qualities are concerned (Khin et al., 2006). Osmotic dewatering is a process that enables water removal and modification of the chemical composition of the material without changing its integrity. However, it is possible to a certain extent, to change its nutritional and functional properties, achieving a specific formulation of the product without modifying its integrity (Torreggiani, 1993).

Besides chemical changes, osmotic dehydration causes an alteration of physical properties of the plant tissue. Shrinkage, decreased water holding capacity, changes in porosity and resistance to deformation are usually observed during osmotic dehydration. The alteration of physical properties reflects the deleterious influence of osmotic dehydration on the structure of plant tissue and morphology of the cell (Lewicki & Pozecka-Pawlak, 2005). The texture of the plant tissue is mainly determined by properties of the cell and middle lamella, as well as the turgor pressure (Jin et al., 2014). Moreover, the volume of intercellular spaces, presence of starch granules, and the chemical composition of the cell sap all affect the plant tissue (Ilker & Szczesniak, 1990). Mass transport phenomena and their effect on physical, textural, and chemical properties of osmotically dehydrated products are increasingly being studying. It is important to link microstructural investigations with processing parameters, and the compositional and mechanical characteristics of the tissues during osmotic dehydration (Shi & Le Maguer, 2002).

As compared to single drying processes, osmotic dehydration achieves a twofold transformation of the food item, by both a decrease in water content and solute incorporation, which may result in a subsequent weight reduction. Also, the ‘direct formulation,’ together with partial dehydration, is the distinctive aspect of the process when compared to other dehydration methods. Old techniques such as candying or salting are based on the same principles of osmotic dehydration, but generally, they are long-term processes, and they favor the solute penetration and limit the water removal. Recently, wider prospects for osmotic dehydration have arisen as a pre-step to further processing. The large solute uptake causes additional resistance to the mass transfer of water and leads to a lower dehydration rate in complementary drying (Wang & Sastry, 2000). However, using osmotic dehydration as a pre-treatment before air drying can be practical only when the osmotic agent that is gained by the food is required for the product. Further, the pre-treatment is time-consuming. Again, osmotic dehydration was noted to cause a reduction in anthocyanin in cranberries because of leaching in the syrup (Matuska et al., 2006).

2.3 Factors affecting osmotic dehydration

The osmotic dehydration process deals with mass transport in the presence of hypertonic solution, where the quantity and the rate of water diffusion rely on several variables and processing parameters. Many research studies have been conducted to study the influence of variables such as solute concentration, temperature, immersion time, osmotic solution and food mass ratio, agitation of osmotic solution around the sample and specific surface area of the food and by using a low pressure system etc. (Corzo & Gomez, 2004; Lerici et al., 1985; Raoult-Wack, 1994; Raoult et al., 1989; Rastogi et al., 1999; Rastogi & Raghavarao, 1997; Rastogi & Niranjana, 1998). Following is the detailed discussion about factors which can affect on OD process.

2.3.1 Type of solutes

The type of osmotic agent used and its molecular weight or ionic behavior strongly affects the kinetics of water removal, solid gain, and equilibrium water content (Alibas, 2007a). In the most published literature, the osmotic dehydration process is applied with common carbohydrates (sucrose, sorbitol, corn syrup, glucose, and fructose) or salts (NaCl, CaCl₂) or their mixtures. The most commonly used osmotic agents are sucrose and sodium chloride (Telis et al.,

2003). Many studies have pointed out the effectiveness of combining both solutes (Lerici et al., 1985). Any solute or solvent that is soluble in water can be used (e.g., starch, ethanol, polyols, and dextrose) as dewatering and impregnating agents (Lenart & Flink, 1984). Low molar mass saccharides (glucose, fructose, and sorbitol) favors moisture loss, and higher molar mass solutes reduce solids gain (Raoult-Wack, 1994).

Maltodextrin can also be used as a partial substitute for sucrose, due to their low levels of sweetness, are desirable osmotic agents for food materials requiring less sweetening. The rate of penetration into the fruit pieces was faster with high fructose corn syrup (HFCS) than sucrose. However, taste panel evaluations indicated that overall sucrose solution was preferred as an osmotic medium over HFCS (Le Maguer, 1988). The effect of osmotic pretreatment on the quality and functional properties of frozen cucumber tissue was demonstrated by Dermesonlouoglou et al. (2007). The dehydrofrozen samples showed improved firmness for prolong storage period and better quality (Dermesonlouoglou et al., 2007). The differences observed in osmotic dehydration behavior between salts and sugars presumably arise from the differences in size and molar concentration of the ionized salts versus the larger, unionized sugars. The smaller salt ions can more easily diffuse through the cell membrane, resulting in a gain in higher solids. This solid uptake also reduces water loss as the potential osmotic gradient is reduced. Sugars that are larger cannot easily diffuse, and equilibrium is achieved primarily through the flow of water from the cells (Ponting et al., 1966).

The water activity (a_w) lowering capacity increased as the dextrose equivalent value increased. Logarithmic relationships were found between molecular weight and dextrose equivalent (Argaiz et al., 1995). However, the diffusion coefficient decreases with increasing solid contents (Welti et al., 1995). Saurel (1994), found that this was closely related to the formation of a barrier layer, which was promoted by high molecular weight solutes, which prevents loss of natural fruit solutes (Saurel et al., 1994). Hawkes and Flink (1978), investigated the possibility of using binary mixtures of solutes with sucrose as a means to reduce solute cost and/or improve osmotic effectiveness, and reported that sucrose alone or in combination with salt can give a high level of water loss from the fruit piece, and result in high solids content prior to air- or vacuum-dehydration (Hawkes & Flink, 1978). Moreover, osmotic solutions that contain sucrose with either salt, maltodextrin, or lactose have been shown to be more effective than

sucrose alone at the same total concentration. However, the ultimate choice of the blend will depend on factors such as solute cost.

2.3.2 Concentration of solutes

The kind of sugar and its concentration as osmotic substance strongly affect the kinetics of water removal, the solids gain, and the equilibrium water content. A research study showed that by increasing the molar mass of solute, weight loss and dehydration aspects of the process are favored and both equilibrium and drying rate increases with the increase of osmotic syrup concentration (Islam & Flink, 1982a).

Invert sugar should theoretically be more effective than sucrose of the same concentration because, when completely inverted, invert sugar has twice as many molecules per unit volume (Ponting et al., 1966). Moreover, acids act as a catalyst in hydrolyzing disaccharides to the monosaccharides. Again, during osmotic dehydration the fruit acids leach out of the fruit into the syrup, accelerating sucrose hydrolysis, and leading to an increase in glucose and fructose followed by a similar decrease in sucrose.

2.3.3 Temperature of osmotic solution

The rate of osmosis is markedly affected by temperature. This is the most important parameter affecting the kinetics. The temperature of the osmotic process has been extensively studied by many researchers. The water loss increases with the increase in temperature, whereas solid gain is less affected by temperature. The rate of osmosis increases the mass exchange and diffusion coefficient, but above 50⁰C, enzymatic browning and flavor deterioration takes place (Videv et al., 1990). Ascorbic acid and chlorophyll retention are also markedly affected. High temperature over 60⁰C modifies the tissue characteristics, favoring impregnation phenomenon, and thus solid gain (Lenart & Flink, 1984). Initially, the water loss and solid gain increases in temperature up to 50⁰C depending upon the fruit and variety and later on falls sharply becoming nearly constant at 60⁰C which indicated a negligible increase in the rate of sucrose diffusion above 60⁰C (Rahman, 1992). Since water loss is higher at a higher temperature, the osmotic equilibrium is achieved by the flow of water from the cell rather than by solid diffusion. Also, acceleration of water loss without modification of sugar gain when the temperature is increased has been observed by many authors.

2.3.4 Contact time

The loss in water content and gain in soluble solids content is a function of time. In general, as the time of treatment increases, the weight loss increased, but the rate at which this occurs decreases. In other words, the rate of mass transfer is generally found to decrease with an increase in immersion time during the osmotic process. Increase in immersion time will increase weight loss in osmosed fruit. Most changes in weight and dry matter content occur during the first 30 min of processing, while macroscopic properties such as cell shape, cell size, and shape of intercellular spaces change during the whole investigated time of osmotic dehydration of an apple (Lewicki & Pozecka-Pawlak, 2005). Further dewatering leads to maximum water loss, such as 50% reduction takes place within the first two to three hours depending upon the type of fruits (Hawkes & Flink, 1978).

2.3.5 Geometry of sample

Osmotic concentration behavior will depend on the geometry of the sample piece. This is due to the variation of surface area per unit volume or mass and diffusion length of the component involved in mass transport. There is an increase in the mass reduction of about 1.3 times when apple slice thickness decreased from 10 to 5 mm (Contreras et al., 2007). It was also found that solids gain increased as the ratio of the surface area to diffusion length (A/L) increased while water loss increased to a maximum (depending on shape) and then decreased. The low water loss corresponding to higher A/L value was probably due to a reduction of diffusion caused by high solid gain.

2.3.6 Agitation of osmotic solution

Agitation of the osmotic solution is an important aspect of osmotic treatment. The agitation ensures that the concentrated solutions are restored around the particle surface and that a concentration difference favorable to mass transfer is recreated. In early works (Ponting et al., 1966), the effect of agitation was studied by comparison of agitated and non-agitated treatments. It was reported that agitated samples exhibited greater weight loss than non-agitated ones, and thus, agitation was found to be another process parameter. Raoult-Wack et al. (1994) studied the effect of agitation on both water loss and solid gain and reported: agitation of the osmotic solution resulted in higher mass transfer coefficient values for solutions of higher concentration

and higher viscosity (Raoult-Wack, 1994). The agitation has a good influence on weight loss (especially for the concentrated solutions) and on the exchange speed. The agitation ensures that the concentrated solutions are renewed around the particle and therefore, a concentration difference favorable to mass transfer is recreated. As a corollary, dilution of the boundary layer.

2.3.7 Osmotic solution and food mass ratio

The mass ratio of osmotic agent to fruit also has a significant influence on osmotic dehydration. Higher osmotic solution/fruit ratio favored higher moisture removal and higher sugar uptake. It means an increase of osmotic solution to sample mass ratio increased both the solid gain and water loss in OD (Lenart & Flink, 1984). To avoid significant dilution of the medium and subsequent decrease of the (osmotic) driving force during the process a large ratio (at least 30:1) was used by most workers whereas some investigators used a much lower solution to product ratio (4:1 or 3:1) in order to monitor mass transfer by following changes in the concentration of the sugar solution (Conway et al., 1983). It has been shown that an appreciable degree of dehydration can be achieved by contacting the fruit pieces with a sufficient level of osmotic syrup to prevent dilution and to effect steady water-solute transfer. It has been recommended that a 20:1 solution/fruit ratio to avoid changes in the syrup concentration (Saurel et al., 1994). Assumption of constant solution concentration can be satisfied by maintaining a high solution/fruit ratio. In research, a ratio of 30:1, or as high as 50:1, are used to maintain equilibrium during experimentation. Although the assumption of high osmotic solution/fruit ratio can be satisfied on a laboratory scale, the industrial application requires handling of tons of fruits, and many problems can be faced when high volumes of concentrated solutions are circulated through the equipment in an industrial application.

2.4 Maltodextrin

Maltodextrin is a long chain polysaccharide widely used in food industries, which can be produced by acid and/or enzymatic hydrolysis of starch. It contains linear amylose and branched amylopectin degradation products, joined by α -(1,4) and α -(1,6) linkages. Hence, the maltodextrins are considered as D-glucose polymers of variable length and therefore different molecular weight (Dokic et al., 1998; Wang & Wang, 2000). They represent a mixture of saccharides with a broad molecular weight distribution, depending on dextrose equivalent (DE),

which reflects the degree of hydrolysis. General formula for maltodextrins are $[(C_6H_{10}O_5)_n H_{20}]$. A wide range of maltodextrins are commercially available which is categorized based on the dextrose equivalent (DE) value (Fitton, 1979), where the different DE value maltodextrins have different physicochemical properties like solubility, freezing temperature, viscosity, gel forming capacity etc (Dokic et al., 1998; Wang & Wang, 2000). Hygroscopicity, solubility, osmolality, and their effectiveness to reduce the freezing point increase with increasing DE, while viscosity, cohesiveness, and coarse-crystal prevention increases with decreasing DE (Chronakis, 1998).

Dextrose equivalent (DE value) is a measure of reducing sugar present which can be expressed as percentage of d-glucose on dry matter of hydrolysate. The dextrose equivalent value is a common parameters to measure the molecular weight of the maltodextrin (Rong et al., 2009), which is inversely proportional to the molecular weight of the compound, as shown in equation 1

$$\text{Dextrose equivalent (DE)} = \frac{M_{\text{glucose}}}{M_n} \times 100 \quad (2.1)$$

M_{glucose} is the molecular weight of glucose and M_n is the number average molecular weight of the sample. This could explain that the maltodextrin 10DE, which was used in current study, has highest molecular weight amongst other solutes. Therefore, the range of maltodextrins used in the present work based on the molecular weight of the solute is: maltodextrin 10DE > MD 15DE > MD 18DE. Therefore, the properties of each maltodextrin grade is different than other and hence the functionality is different.

Generally, the high DE maltodextrins possess high viscosity, better solubility, bulking and bodying characteristics, whereas the low DE maltodextrin such as maltodextrin 10DE have better binding properties and can function more effectively as fat binders than high DE maltodextrin (Kennedy et al., 1995). Commercial maltodextrins are available from three botanical sources: corn, potato, and rice starches. Rice maltodextrin had a significantly higher content of saccharides with degree of polymerization (DP) less than 10 whereas, for potatoes the DP is greater than 20. It is believed that the longer average chain lengths of potato maltodextrin resulted in greater retrogradation, and the shorter chains in rice maltodextrin resulted in lower maximum freeze concentrated glass transition temperature (Wang & Wang, 2000). Since their parent starches are different, it is possible that maltodextrins from different botanical sources also exhibit different properties due to inherent differences in their chemical structures.

Carbohydrate profile and microstructure organization of maltodextrin. Following is the table indicating the difference between the maltodextrin 10DE, 15DE and 18DE, which was obtained from Univar Pvt. Ltd, Canada.

Table 2.1: Carbohydrate profile of maltodextrins (10DE/15DE/18DE)

Carbohydrate profile	Maltodextrin 10DE	Maltodextrin 15DE	Maltodextrin 18DE
Dextrose	<1	1	2
Maltose	2	5	6
Maltotriose	4	7	9
High saccharides (DP4+)	93	87	83

It has been recently explored that the molecular weight could be one of the tools to predict the fundamental properties of maltodextrin and to understand its effect on various processes, but it has been applied to a particular issues so far (Wang & Wang, 2000; White Jr et al., 2003). Therefore, the current work is an attempt to understand the relation between three different molecular weight maltodextrins and the mass transport mechanism during osmotic dehydration pretreatment. Following are some properties of maltodextrins.

2.4.1 Physicochemical characteristics of Maltodextrins

The properties of maltodextrins are determined by their respective dextrose equivalent value, their carbohydrate profile, and the method by which they were physically processed for use.

2.4.1.1 Viscosity, solubility and sweetness

The solubility, osmolality, and their effectiveness to reduce the freezing point increase with increasing DE, while viscosity, cohesiveness and coarse crystal prevention increase as DE decreases (Chronakis, 1998). It is possible, however, to have a similar DE values but different proportions of high-and low-molecular-weight saccharides by altering the temperature of hydrolysis during maltodextrins production (Griffin & Brooks, 1989). Therefore, solubility and

solution stability will be influenced by high-molecular-weight components, while viscosity, crystallization, and sweetness will depend on the amount of low molecular weight components.

2.4.1.2 Hygroscopicity

The presence of compounds like maltotriose and maltotetraose in the maltodextrins, imparts high on its hygroscopicity. On the other hand, studies also suggested that moisture absorption increases smoothly with decreasing molecular weight, while sugars containing a high-molecular-weight fraction achieved equilibrium moisture sooner than the corresponding low-molecular-weight fraction (Kearsley & Birch, 1975).

2.4.1.3 Turbidity

Turbidity is an important characteristic which can influence on film forming property of maltodextrin during encapsulation of flavor, volatile component etc. The low DE values display a relatively low turbidity (Raja et al., 1989). Mainly, the greater tendency of amylose to retrograde comparing with amylopectin led to the formation of haze or precipitation at higher concentrations.

2.4.1.4 Free and bound water

The mean molecular mass composition and distribution of molecular masses in maltodextrins are also responsible for the physical properties of water. At high concentrations the interactions between high molecular mass polysaccharides and water dominate, while at more diluted and liquid systems the interaction between oligosaccharides and water increase. The higher the degree of polymerization the higher the bound water at high polysaccharide concentrations. In low molecular mass maltodextrin fractions this relation is reversed with decreasing polysaccharide concentrations. Thus, the oligosaccharides stabilize the water interactions in solutions and gels, whereas the polysaccharides increase the polymer interactions. However, high molecular maltodextrin fractions contain amounts of bound water that are independent on concentration (Chronakis, 1998).

2.4.1.5 Gel forming properties

Gel forming capacity of maltodextrin is temperature and concentration dependent. A sharp increase in concentration decreases the degree of polymerization which emphasized the development of continuous structure with long and linear macromolecular chain in maltodextrin (Kasapis et al., 1993). The rheological properties of maltodextrins are also concentration dependent where, high molecular weight chain are capable of forming ordered nuclei for the development of three dimensional network (Clark et al., 1989).

2.4.2 Food applications of maltodextrin

Maltodextrins are used in the food industry due to its ability to form gels and retain water, as a texture modifier, either for gelation, retention of water, and to a certain extent substitution of fat (Alexander, 1995). They perform multifaceted functions in food systems, including bulking, providing resistance to caking, adding texture and body, forming films, binding flavor and fat, serving as oxygen barriers, giving surface sheen, aiding dispersibility and solubility, freezing control and preventing crystallization, and as product extenders (Setser & Racette, 1992). Lower DE maltodextrins result in a better fat binding. Setser and Racette (1992) found thick, leathery crusts in cakes containing high levels of an 18DE corn maltodextrin and also in the combinations of maltodextrins with polyols. Maltodextrins have been proven useful to reduce Maillard reactions and used in microencapsulation of food components such as fat and oils, vitamins, minerals, and colorants (Kenyon & Anderson, 1988; Sheu & Rosenberg, 1995; Whorton & Reineccius, 1995). High DE molecules protected encapsulated orange peel oil against oxidation which suggest the importance of DE to the functionality of the wall system (Anandaraman & Reineccius, 1986). Bangs and Reineccius (1982), reported that retention of volatile flavor compounds decreases as maltodextrin DE increased (Bangs & Reineccius, 1982).

2.5 Modeling in osmotic dehydration

In recent years, despite increased research into osmotic dehydration, the fundamental knowledge about predicting mass transport is still a gray area (Raoult-Wack, 1994; Spiazzi & Mascheroni, 1997). Therefore, one of the major approaches of modern research is to understand and develop the model for osmotic dehydration to optimize the process along with subsequent drying processes and to achieve the highest possible quality at minimum energy costs (Saguy et

al., 2005). The modeling will also lead to predicting the influence of process parameters and sample properties on moisture loss and solids gain. The process parameters such as temperature, solution concentration, and contact time, should be taken into account while modeling an osmotic dehydration process. Also, it is one of the important factors in minimizing the energy cost while achieving the highest possible quality product (Saguy et al., 2005). Since during osmotic dehydration of fruits and vegetables, the plant cells are involved in the process does not act an ideal membrane for the process and hence it increases the complexity of the process during mass transfer (Fitton, 1979). Therefore, the mass transfer across the cell membrane takes place as a combination of osmosis, diffusion, and hydrodynamic mechanism, or HDM (Fito & Pastor, 1994). Two basic approaches can be used to model conventional osmotic dehydration (COD) (Ramaswamy et al., 1982; Salvatori et al., 1998). The first one is the macroscopic approach, which assumes that the food tissue is homogenous, and properties of the cell wall, membrane, and vacuole are lumped to model. Whereas, in microscopic, the heterogeneity of plant tissues and complex cellular structure is accountable, which can represent them in a simplified conceptual model (Fito et al., 1996).

2.5.1 Macroscopic approach

The pseudo-diffusion, square root of time, irreversible thermodynamic, and other approaches can be carried out in a macroscopic approach (Fito et al., 1996). However, the macroscopic approach used for modeling osmotic dehydration often ignores internal resistance to mass transfer (Fasina et al., 2002). The concentration profile can be developed through mass transfer process using a macroscopic approach which can clarify the mass transfer mechanisms and their coupling, as well as cell wall deformation and relaxation changes, can be determined when combined with microscopic features (Alzamora et al., 1997; Lenart, 1984). Several mathematical models have been proposed based on Fick's unsteady state laws of diffusion (Fito et al., 1996; Nsonzi & Ramaswamy, 1998; Ochoa-Martinez et al., 2007b) with estimation of diffusion coefficients for both water loss and sugar gain also including hydrodynamic mechanisms (Fito et al., 1996; Salvatori et al., 1998). Also, empirical and semi-empirical models are often applied (Barat, Fito, et al., 2001; Panagiotou et al., 1999). HDM is a mass transfer mechanism initially developed to be able to better predict the effect of vacuum on solute impregnation in order to obtain novel products (Fito & Pastor, 1994). The Hydrodynamic

mechanisms were first conceived due to the complete effects of vacuum during vacuum osmotic dehydration (VOD), which could not be explained using only diffusional and osmotic transport models. Hence the HDM was investigated to complete the model of the process (Fito & Pastor, 1994).

The Fick's unsteady state law of diffusion has been used in many research to estimate the water or solute diffusivity, simulating the experiments with boundary conditions to overcome the assumptions involved in Fick's law (Barat, Fito, et al., 2001; Fasina et al., 2002; Telis et al., 2003). Sample dimensions and the effective diffusion coefficient are the two parameters required in Fick's law. There are few ways to estimate the coefficient such as finding numerical or analytical solutions to experimental data (Nguyen et al., 2006), calculating the relation between the slope of theoretical diffusion curve and the slope of experimental mass transfer ratio (Ade-Omowaye et al., 2002; Rastogi et al., 2002), and applying linear and nonlinear regressions (Akpınar, 2006). Of these studies, only a few have considered unsteady-state mass transfer during osmotic dehydration (Ade-Omowaye et al., 2002; Escriche et al., 2000; Kayacier & Singh, 2004; Rastogi & Raghavarao, 2004; Roberts et al., 2002). To solve Fick's law, there are few things that need to assume such as, solute concentration is constant; mass transfer is restricted to moisture out of and solids into the fruit; diffusion rate is infinite, allowing for instantaneous equilibration between fruit and syrup; samples are homogenous, of ideal shape and of a uniform size; no product shrinkage; and that the effective diffusivity depends on the conditions of osmotic dehydration alone (Nsonzi & Ramaswamy, 1998).

Since the Fick's laws can be solved by using various idealized geometric sample shapes, such as infinite slabs and cylinders, spheres, among others (Crank, 1975; Nsonzi & Ramaswamy, 1998; Ochoa-Martinez et al., 2007b; Rastogi et al., 2002). Also, Fick's first law explains the linear relationship between the flux of a component and the concentration gradient of that component (Crank, 1975). The Fick's first and second law express as:

$$F = -D \frac{\partial c}{\partial x} \quad (2.2)$$

$$V \frac{\partial w}{\partial t} = D \frac{\partial^2 y}{\partial x^2} \quad (2.3)$$

Where F is the rate of transfer per unit area of cross-section (kg/m^2), c is the concentration of diffusing substance (kg/m^3), x is the space coordinate measured normal to the section (m) and D is the diffusion coefficient (m^2/s) (Ramaswamy & Van Nieuwenhuijzen, 2002). w is moisture content ($\text{g H}_2\text{O/m}^3$), x is the spatial coordinate (m); t is a time in s; D is the diffusion coefficient (m^2/s), and V is volume (m^3). Fick's second law of diffusion can be used to determine diffusion coefficients for either water loss or solids gain, where a mass transfer is assumed to be one-directional, and interactions of other components on the diffusion of the solute are considered negligible (Rastogi et al., 2002).

The estimation of the diffusion coefficients for both water loss and solids gain can be achieved using the second unsteady Fick's law assuming that the mass transfer during the process is unidirectional and the interactions of the other components on the diffusion of the solute are negligible. A rate parameter was used by Biswal et al. (1991) and Ramaswamy and van Nieuwenhuijzen (2002) to model osmotic dehydration of green beans as a function of solution concentration and process temperature (Biswal et al., 1991; Ramaswamy & Van Nieuwenhuijzen, 2002). The parameter was calculated from the slope of the straight line obtained from bean moisture loss, and solids gain vs. the square root of time. Also, Azuara et al. (1992) developed a model based on Fick's second law of unsteady-state one-dimensional diffusion to predict the kinetics of water loss and solids gain during osmotic dehydration (Azuara et al., 1992). In addition, this model has been proposed to compute the time required for a given weight reduction as a function of the processing temperature and of the solution concentration or to estimate the dehydration parameters.

Furthermore, the models for moisture diffusivity and soluble solids diffusivity was studied by Nsonzi and Ramaswamy (1998) to understand the osmotic dehydration kinetics of the blueberry (Nsonzi & Ramaswamy, 1998). Azarpazhooh & Ramaswamy (2010a) demonstrated that the Azuara model provided a good fit for both moisture loss and solids gain of apple pieces during microwave osmotic dehydration and allowed for the calculation of equilibrium values of both moisture loss and solids gain without waiting for real equilibration to occur (Azarpazhooh & Ramaswamy, 2009a). The same authors noted that the actual values deviated from predicted values at short treatment times (less than 30 min), so the model may fit better with longer treatments.

The application of artificial neural networks (ANN) in modeling osmotic dehydration is gaining importance in recent years (Ochoa-Martinez et al., 2007a). The optimal configuration of ANN is obtained by varying the main parameters, namely: transfer function, learning rule, number of neurons and layers, and number of learning runs (Chen et al., 2001). ANN is a powerful tool to model the data to learn linear and non-linear relationships between variables directly from a data set and allows for these relationships to be applied to unlearned data. Ochoa-Martinez et al. (2007b) aimed to develop useful models in order to predict effective diffusivity (D_e) and moisture loss at a given time as well as at equilibrium during osmotic dehydration of fruits by combining Crank's solution and ANN as a function of the following ten process variables: temperature, concentration, contact time, water and solids content of the sample, porosity, surface area, characteristic length, solution ratio, and agitation level (Ochoa-Martinez et al., 2007a). Chen et al. (2001) also found that using ANN to predict quality changes during osmo-convective drying of blueberries yielded much better performance than traditional mathematical models indicating that it is feasible to use ANN for prediction and optimization of this process (Chen et al., 2001). Overall, it can be very useful in practice for a wide variety of products (Ochoa-Martinez et al., 2007a) considering the fact that it fit well and were built upon a wide variety of data.

2.5.2 Microscopic approach

Many of the macroscopic changes seen in osmotically dried food materials are based on changes at the microscopic level, such as cell shrinkage or lysis, which in turn affects sample porosity and texture which is also considered as the microscopic approach to study the mass transfer inside the cellular material. During osmotic dehydration depends on both processing variables and micro-structural properties of the biological tissue (Barat et al., 1999). When biological cellular material undergoes osmotic dehydration, mass fluxes in the system imply changes in structural and transport properties (volume, dimension, viscosity, density, porosity, etc.). As a result, these changes affect the mass transfer fluxes. Therefore, the processed product might face some changes such as color changes are caused by compositional changes through degradation or loss of pigments (Chiralt & Talens, 2005; Krokida et al., 2000), changes in mechanical properties (Telis et al., 2005) due to detachment of middle lamella from the cell wall, leading to loss of cell turgor, increased intercellular space, and change in sample size and shape

(Chiralt & Talens, 2005). In addition, an increase in the porosity during osmotic dehydration process due to cell shrinkage, for instance, will affect mass transfer due to hydrodynamic mechanisms and capillary action which pulls osmotic solution into the sample, representing an increase in non-diffusional driving forces that will contribute to mass transfer (Chiralt & Talens, 2005; Fito & Pastor, 1994). These changes should be taken into account while understanding the OD process because they are related to quality factors and some aspects of food processing, such as food classification, process modeling and design of equipment . Therefore, the study of the micro-structural changes during dehydration can help to understand and predict the changes occurring in the physical-chemical properties at higher levels of structure.

2.6 Development in osmotic dehydration

The osmotic dehydration has been successfully adapted prior to finished drying process due to its advantage over the conventional drying process. However, osmotic dehydration is a time-consuming process. Therefore, supplementary methods are needed to enhance the mass transfer with minimum effect on the product quality (Rastogi et al., 2002). They include: ultrasound (Rodrigues & Fernandes, 2007), pulsed vacuum (Ito et al., 2007; Chafer et al., 2003), high-intensity electric field (Rastogi et al., 1999), high hydrostatic pressure (Akyol et al., 2006) and microwave (Azarpazhooh & Ramaswamy, 2009a, 2009b; Li & Ramaswamy, 2006c). Apart from the supplementary techniques, the other treatments have also been studied such as treating the skin of waxy fruits (Grabowski et al., 2007), determining optimal solution temperature (Li & Ramaswamy, 2006a). The selection of any treatment or the supplementary techniques depends on the product requirements such as the expected water loss, soluble solids gain, and the sensory properties of the food products, etc. Few of these techniques are discussed below.

2.6.1 Ultrasound application

A recently developed technique of ultrasound has not been explored fully until recent (De Gennaro et al., 1999). It is the process where acoustic cavitation is generated by ultrasonic waves which can generate minute vapor-filled bubbles that collapse rapidly or generate voids in liquids. This process can be carried out at ambient temperature as no heating is required, thus reducing thermal degradation (Rodrigues & Fernandes, 2007). The use of ultrasound has been known to improve mass transfer for various products and processes in liquid-solid systems, such as

osmotic dehydration (Deng & Zhao, 2008; Stojanovic & Silva, 2007). Applying ultrasound during osmotic treatment has a significant effect on the kinetics of water loss, sugar gain, and firmness loss, as well as on the microstructure of osmotically dehydrated different products and processes in the liquid-solid system, such as osmotic dehydration of apples (Cárcel et al., 2007). When high-intensity ultrasound was applied during the OD of apple, the accelerated rate of osmotic dehydration was recorded (Gallego-Juarez et al., 1999). A similar outcome as reported by Rodrigues and Fernandes (2007) when melon cubes were subjected to ultrasound under a water bath before OD and determined that this method removes sugar and alters tissue structure and enhances the water diffusion during subsequent drying. It was also denoted during the similar study that the ultrasound enabled faster drying along with lesser sugar content dried product.

Duan et al. (2008) used ultrasound pretreatment to improve the freeze-drying rate of apple samples to ultrasound during OD and showed enhanced water loss (Duan et al., 2008). However, there was the cell structure was significantly affected by the process, thereby cell collapse, which resulted in lower product firmness (Deng & Zhao, 2008). However, this effect can vary according to target tissue and intensity, as when applied to melon there was no cell collapse observed, there was only the creation of micro channels between the cells (Fernandes et al., 2008). These effects depend upon the intensity of the ultrasound along with the type of cell structure etc. However, there is more research necessary to determine the ideal ultrasonic frequency to be used in this process (Cárcel et al., 2007).

2.6.2 High pressure

The high-pressure techniques can be applied to liquid and solid foods, with or without packaging, at pressures between 100 and 800 MPa (Eshtiaghi et al., 1994) and it has been tested for their effectiveness as an alternate to thermal blanching (Eshtiaghi & Knorr, 1993). It has been proven that high hydrostatic pressure (HHP) with the combination of mild heat treatment can be used for blanching purposes to inactive peroxidase (POD) and lipoxygenase (LOX) in carrots, green beans, and green peas (Akyol et al., 2006). On the other hand, it is notified that an application of high hydrostatic pressure for 5 min enhanced moisture loss and solids gain in pineapple pieces during the subsequent osmotic treatment when 400MPa was applied (Rastogi & Niranjana, 1998). However, in the further microscopic analysis, it was confirmed that there was

extensive cell rupturing, leaving the cells more permeable with a reduction in intercellular material, where the severity of the damage was directly correlated to the pressure of the HP treatment. Overall the cell collapse contributed negatively to sample texture and also limited rehydration capacity (Rastogi & Niranjana, 1998). On the other hand, it was also concluded that the high-pressure increase moisture loss during osmotic treatment, but it made even more of an impact on solids (sugar) uptake, and can be essential when sugar uptake in the product is desired (Taiwo, 2001).

2.6.3 Pulsed electric field

Pulsed electric field (PEF) has recently gained importance due to its nonthermal processing advantages. In PEF treatment the short bursts of the electric field are applied to the sample which creates small holes into the cell wall of the sample and ultimately increases the permeability and water diffusivity with minor alteration in the food matrix (Ade-Omowaye et al., 2002). The effectiveness of PEF during osmotic dehydration was firstly introduced by Rastogi et al. (1999) (Rastogi et al., 1999). PEF has a positive influence on mass transfer in further processes due to its ability to increase the permeability of plant cells in a short time (μs to ms range) while keeping the product matrix unaltered, thereby positively accelerating mass transfer during osmotic dehydration (Ade-Omowaye, Angersbach, et al., 2001). Rastogi et al. (1999) subjected carrot slices to high-intensity electric field pulses (HELP) prior to osmotic dehydration where the study reported an exponential increase in diffusion coefficient with increased intensity of electrical field until 1.09kV/cm beyond which increased field strength had little effect (Rastogi et al., 1999).

Taiwo et al., 2003, subjected apple to high-intensity electric field pulses (HELP), which enhanced moisture removal while limiting solids gain when subjected to OD treatment (Taiwo et al., 2003). The conductive testing in the same study determined that the application of HELP was attributed to the good structural integrity in apple. This work also concluded that the samples pretreated with HELP prior to OD were brighter in color, better retained ascorbic acid, and provided a firmer texture. Therefore, many research studies suggested that PEF pre-treatment might be a better alternative than processing at high temperatures (Ade-Omowaye, Rastogi, et al., 2001; Lazarides & Mavroudis, 1996; Taiwo, 2001). However, HELP contributes to cellular disintegration and loss of compressive strength in texture, which is similar to the high-pressure

treatment (Rastogi et al., 1999). On the other hand, it also has been proven that membrane breakdown is not the main factor determining solute uptake (Taiwo et al., 2003). The hence pulsed electric field can be considered as good pretreatment for promoting moisture loss over solids gain.

2.6.4 Application of vacuum

The application of vacuum during osmotic dehydration has been studied widely to enhance mass transfer with homogeneous concentration profile in the product (Fito et al., 2001). In vacuum osmotic dehydration technique, the extended time under high vacuum during osmotic dehydration can cause an irreversible deformation of the food tissue and ultimately decreases the free volume available for impregnation of solute. Hence the application of pulsed vacuum is gaining importance while subjecting to biological commodities (Ito et al., 2007). An enhanced mass transfer kinetics under vacuum osmotic dehydration (VOD) can be achieved due to its twofold mechanism as described by Fito and Pastor (1994), is firstly the hydrodynamic mechanism (HDM) contributes for faster mass transport in short time when compared with atmospheric pressure treatment; secondly pseudo-fiction mechanism promotes a larger interphase surface which allows for higher mass transport (Fito & Pastor, 1994).

During vacuum impregnation (VI), the mass loss is reduced, and the process yield is increased due to lower mass loss in comparison with atmospheric pressure. Moreover, the sensorial properties of the product are improved as the products are enriched with nutrients, vitamins, minerals, and other additives (Chiralt et al., 2001). Therefore, it was concluded in many studies that the Vacuum impregnation has a huge influence on the characteristics of the product such as the internal ratio, water loss and solids gain (Barat, Chiralt, et al., 2001; Chafer et al., 2003). The degasification of the outer layer of cells in the sample was caused due to the PVOD treatment. As a result, the lightness of the samples decreased (Chafer et al., 2003; Talens et al., 2002). On the other hand, an increase in lightness (L^*) was also reported when samples are treated with PVOD (Deng & Zhao, 2008). The similar studies also found that the application of pulsed vacuum resulted in higher solids gain and lowest loss of firmness of Fuji apple samples. The results also concluded that in PVOD samples, moderate cell deformation and collapse were examined using scanning electron microscopy. Chafer et al. (2003) reported that

PVOD preserves fruit color as, it excludes any contact of oxygen with the internal part of the tissue in the intercellular spaces, and limits browning (Chafer et al., 2003).

2.6.5 Blanching and skin pretreatment

Blanching has been applied prior to drying of fruits and vegetables such as bananas (Dandamrongrak et al., 2002), red paprika (Ade-Omowaye, Rastogi, et al., 2001), figs (Piga et al., 2004), potatoes (Eshtiaghi & Knorr, 1993), strawberries (Alvarez et al., 1995). The steam or hot water blanching used before osmotic dehydration with the purpose of enzyme inactivation, to prevent oxidation, discoloration, and off-flavor development, microbial growth and to promote gas removal from surfaces and intercellular spaces. However, Water blanching (85–100⁰ C) usually results in loss of nutrients such as minerals and vitamins (Akyol et al., 2006). Blanching can cause some changes in the chemical and physical state of nutrients and vitamins as well as having an adverse environmental impact from the large water and energy usage. Few studies have found that blanching the berries in boiling water for 120s had no positive effect on moisture removal, but rather lowered the dry basis moisture content by increasing sugar uptake (Chafer et al., 2003; Rennie et al., 2009).

Fruits having a waxy outer skin to protect it from weather, insects, and parasites generally require a skin pre-treatment prior to osmotic dehydration to avoid the need for much longer osmotic drying times which would lead to high energy consumption and low product quality (Grabowski et al., 2007; Saravacos & Charm, 1962). The treatments can be classified as chemical, thermal, mechanical, or combination in nature (Grabowski et al., 2007; Lewicki, 1998). Overall, the treatment deemed to be most effective was a mechanical halving of the cranberries as this technique allowed for the osmotic solution to come into full contact with the inner flesh of the fruit, resulting in a 100-fold increase in the mass diffusion (Grabowski et al., 2007).

2.6.6 Application of microwave during osmotic dehydration

Microwave energy is widely used to heat the product as well as for finished drying purpose due to the ability of electromagnetic energy to penetrate into the sample and enhance moisture removal from the inner core of the food product (Orsat et al., 2007). More recently, the microwave energy was employed during the OD process and this novel process was named after

"Microwave osmotic dehydration " (MWOD) either in immersion (Li & Ramaswamy, 2006c) or in spray based configurations (Azarpazhooh & Ramaswamy, 2011; Azarpazhooh & Ramaswamy, 2009a, 2009b, 2011, 2012a, 2012b; Wray & Ramaswamy, 2013; Wray & Ramaswamy, 2015a, 2015b; Wray & Ramaswamy, 2015c; Wray & Ramaswamy, 2015d). The MWOD technique first introduced by Li and Ramaswamy, 2006 and they observed that the apple cylinders when subjected to microwave energy while immersed into the sucrose solution in a continuous flow of osmotic had improved moisture transfer rates when compared to conventional osmotic dehydration (COD), while solids gain was reduced compared to prior studies using both traditional static batch (Li & Ramaswamy, 2006a) and continuous flow (Li & Ramaswamy, 2006b) conditions in the same experimental setup. During the MWOD process, the microwave energy enhances moisture removal from the food along with the osmotic potential developed by sucrose solution during the process. An increased moisture flux as detected due to the selective absorption of microwave energy by the water molecules in food, which also tends to limit the simultaneous transfer of a solute from the solution into the food (Li & Ramaswamy, 2006c).

In the microwave osmotic dehydration immersion (MWODI) technique, the microwave energy was predominantly absorbed by the osmotic solution in which the fruit sample was immersed, and therefore the microwave heating effect was compromised during the dehydration of sample. Hence, the method of MWODI was modified into spray mode by Azarpazhooh & Ramaswamy, where the apple pieces were subjected for osmotic solute spray under microwave heating condition (Azarpazhooh & Ramaswamy, 2009b). The microwave osmotic dehydration under continuous flow medium spray condition (MWODS) delivered high moisture reduction along with minimum solids uptake when compared with MWODI, COD, and conventional osmotic dehydration immersion (CODI) method. Also, the spray technique eliminates the possibility the samples floating on the surface of the osmotic solution when subjected to MWODI process, which was also discussed in past studies (Raoult-Wack, 1994). The reason being said that more microwave energy was absorbed by fruit pieces rather than osmotic solution would, in turn, promote the driving of moisture out of the samples (Azarpazhooh & Ramaswamy, 2009b). It was also investigated during the research study that the osmotic dehydration under microwave heating made it possible to obtain a higher diffusion rate of moisture transfer at lower solution temperatures (Azarpazhooh & Ramaswamy, 2009a).

Microwave heating has the specific advantage of rapid and uniform heating due to the penetration of microwaves into the body of the product (Alibas, 2007a; Bilbao-Sáinz et al., 2006). Therefore, a research study also mentioned that for the fruit with waxy skin, which will need a skin pretreatment when subjected to a conventional osmotic dehydration (COD) method, won't need any pretreatment prior to MWODS process (Wray & Ramaswamy, 2013), process (Wray, 2013), as the intense microwave energy can maintain almost same moisture diffusion with or without skin pretreatment. This attributes to the most important fact that microwave heating is volumetric heating, which refers to the material absorbing microwave energy directly and internally and converting it into heat. As a result, heat is generated from within the fruit pieces leading to faster heating rates (compared to conventional heating, where heat is usually transferred from the surface to the interior) and producing rapid and uniform heating (Gowen et al., 2006).

2.7 Microwave heating mechanism

Microwave heating is based on electromagnetic waves (EM) in the frequency range of 300MHz to 300GHz. However, the band of 915MHz and 2450MHz is reserved for microwave heating applications, which extensively used on industrial and domestic purpose, respectively (Nijhuis et al., 1998; Orsat et al., 2007). Microwave causes volumetric heating of the targeted material, which certainly depends upon the dielectric properties of the food material (Beaudry et al., 2004; Nijhuis et al., 1998; Orsat et al., 2007). The food material heats within the samples when subjected to microwave and the heat product follows two mechanisms: (1) molecular friction due to the rapid movement of molecules with permanent dipole moments in response to the changing direction of microwaves (on the order of millions of times per second) as the waves pass through the food product, or (2) charge drift of ionic species (such as Na^+ or Cl^-) under the action of the microwaves, which leads to collisions between ions, increasingly disordered kinetic energy throughout the sample and subsequent heat generation (Nijhuis et al., 1998; Orsat et al., 2007).

The first mechanism mentioned above is responsible for the heating of food sample under the microwave, as the food samples are typically made up largely of water (a molecule with a permanent dipole) (Nijhuis et al., 1998). The heat generated within the sample, due to the penetration of microwaves into the food samples, and this mechanism distributed the heat

uniformly inside the food and heated the food samples quickly (Alibas, 2007a). Furthermore, the energy absorbed by the sample is given by the following equation (Orsat et al., 2007):

$$P = 2\pi f \epsilon_0 \epsilon'' |E|^2 \quad (2.4)$$

where P (in W /m³) is the energy developed per unit volume, f is the frequency of the EM wave in Hertz (Hz), ϵ_0 is the absolute permittivity of vacuum (8.854188x10⁻¹² F/m), ϵ'' is the loss factor, and |E| is the strength of the electric field (in V/m).

Overall temperature, as well as temperature uniformity of the sample under microwave, can be controlled by modifying either power density (watts of EM energy applied per gram of sample) or duty cycle (magnetron on/off periods) (Orsat et al., 2007).

2.7.1 Dielectric properties of food materials

The heating behavior of the food samples under EM radiation or microwave is based on the dielectric properties of food material (Orsat et al., 2007; Sosa-Morales et al., 2010). The dielectric properties such as dielectric constant (ϵ') and the loss factor (ϵ'') have an extensive impact on microwave heating of the food samples. The dielectric constant (ϵ') dictates the EM-field distribution in the sample material and indicates the ability of a material to couple with microwave energy, while the loss factor (ϵ'') expresses the loss interactions or the materials ability to dissipate electrical energy (Orsat et al., 2007). Moreover, these parameters vary with the frequency of the applied field, temperature, moisture content, composition and particle density of the sample material (Orsat et al., 2007). The dielectric loss factor (ϵ'') expresses the ability of a material to dissipate electric energy and is described as (Venkatesh & Raghavan, 2004):

$$\epsilon = \epsilon' - j\epsilon'' \quad (2.5)$$

$$\tan(\delta) = (\epsilon''/\epsilon') \quad (2.6)$$

Where $\tan(\delta)$ is the loss tangent, $j = \sqrt{-1}$ which indicates a phase shift between the real (ϵ') and imaginary (ϵ'') parts of the dielectric constant. The penetration depth (dp) is defined as the distance traveled by a microwave into a sample when the power has dropped to 1/e or 36.8% of its incident power and expressed it as-

$$dp = \frac{\lambda_0 \sqrt{\epsilon'}}{2\pi \epsilon''} \quad (2.7)$$

Where λ_0 is the wavelength of the microwave in free space. Factors that affect dielectric properties include (i) sample composition (especially water content in foods as it makes up the bulk of the food product), (ii) sample density, as the amount of mass per unit volume, will determine how much material is present to interact with the electromagnetic (EM) field, (iii) temperature, where at low frequencies the loss factor (ϵ'') will increase with temperature and vice versa at high frequencies, (iv) frequency of the applied EM field, as molecules tend to react more or less vigorously to different wavelengths depending on their dipole moment and finally (v) storage time, as compositional changes that take place when a fruit is in storage may affect its dielectric properties (Sosa-Morales et al., 2010). The dominant factor influencing dielectric properties in food products is water content, as this molecule accounts for the majority of absorbance of microwave energy in foods (Sosa-Morales et al., 2010; Venkatesh & Raghavan, 2004). As a result, as the moisture content of a food increases, so does the dielectric constant and loss factor, resulting in better microwave heating properties for that product (Komarov et al., 2005; Sosa-Morales et al., 2010).

2.8 Finished drying methods

Osmotic dehydration is a pre-drying process where partial drying of the product occurs, followed by a secondary (finish) drying process. The post-osmosed product always subjected to finished drying in order to obtain the required 15-20% moisture content of dried fruits. Drying is one of the oldest, versatile, wide spread technique which is the most frequently used method for food preservation. The primary focus of the drying is to remove moisture to reduce the water activity and hence the associated microbial and enzymatic activity and product quality deterioration. The stability of dehydrated fruit products can be ensured by maintaining a_w lower than 0.7 (Ramaswamy & Marcotte, 2005), which minimizes the quality deterioration during storage and make the product available throughout the season without resorting to the more expensive refrigerated and frozen storage practices. There are several dehydration techniques available in a commercial practice which can be used for a wide variety of food products. In most cases, these techniques are carried out higher temperatures and lower humidity conditions in order to accelerate the drying process which is often unable to protect natural taste, nutritional quality, rehydration characteristics and physicochemical and sensory characteristics of the dehydrated food (Lenart, 1996; Lin et al., 1998).

The drying methods such as sun drying, convective hot air drying has traditionally been performed, whereas a recently developed new ways of drying arises extensively for improved quality of dried product such as, drying under vacuum so that lower temperatures could be used, use of rapid drying techniques which would reduce the drying time, use of freeze-drying which is done under conditions below the triple point of water facilitating sublimation; thereby protecting the product texture and other quality factors, use of novel heating sources like microwave and radio frequency heating (significant reduction in drying time), use of various treatments which promote better mass transport phenomena, etc.

2.8.1 Freeze-drying

Freeze drying operates below the triple point of water and therefore allows for the removal of water by sublimation, wherein water transforms directly from its solid form (ice crystals) to a gas, bypassing the liquid state. This process therefore allows for low drying temperatures and high product quality, where freeze-dried products are often regarded as a 'best case' scenario in terms of quality and are therefore often used as a reference for other drying procedures (Azarpahzoo & Ramaswamy, 2011; Lenart, 1996; Nijhuis et al., 1998). Freeze-dried products generally possess very good structural rigidity which prevents structural collapse and maintains the porosity of the food matrix, enabling the excellent rehydration capacity that is typical of these products (Beaudry et al., 2004). However, this technique is also very expensive, where at an industrial scale the cost of freeze-drying has been estimated to be 5-10 times more than hot air drying, limiting its use to high-value products (Duan et al., 2007). Along with installation costs, the expensive nature of freeze-drying can be mainly attributed to energy, which is very high due to the high level of vacuum that must be maintained throughout in order to allow sublimation as well as the fact that the product must first be frozen. As the process can take several days to achieve the desired moisture content, enhancing the rate of moisture removal during freeze drying would reduce the amount of time required, increase throughput, and as a result likely reduce its energy consumption.

2.8.2 Hot air drying

Hot air drying is one of the traditional methods of finished drying using ovens with the hot air of at least 60°C+ for 10-12 hours. This finished drying process can be employed with or

without moving air to improve the process efficiency. Such as, cranberries were dried to 15% moisture content in a research study using hot air drying method at 62⁰C and 10m/s air speed in 12.6 hours (Beaudry et al., 2004). Hot air oven drying can be used as a reference method for moisture content determination and AOAC methods typically require that fruit samples be dried for periods of 16-20 hours at temperatures at or above 100⁰C. Drying time can be decreased when forced-air dryers are used to better remove moisture from the samples (Yongsawatdigul & Gunasekaran, 1996a).

The major concern of hot air drying method is high power consumption, heat damage due to high temperature, degradation of an essential component, loss of quality and nutritional content (Alibas, 2007b; Drouzas & Schubert, 1996; Yongsawatdigul & Gunasekaran, 1996a; Yongsawatdigul & Gunasekaran, 1996b). Surface hardening is another concern during hot air drying due to the high rate of water evaporation from the surface of the sample than its core (Yongsawatdigul & Gunasekaran, 1996a). Therefore, other methods of drying can be used to overcome the disadvantages of air drying, such as slow drying rates and low quality of the resulting product (Beaudry et al., 2004).

2.8.3 Microwave-vacuum drying

Microwave-vacuum drying has been successfully applied to various food products in order to reduce volatile loss, accelerate moisture removal and slow heat transfer to the solid phase due to the absence of convection (Drouzas & Schubert, 1996). Combined microwave vacuum drying of cranberries was suggested as one of two potential solutions for alleviating physical damage caused during microwave dryings such as scorching, off-color production, and uneven heat distribution (Gunasekaran, 1990). Typically, a vacuum is applied to reduce absolute pressure to a level close to 7kPa, where the boiling point of water is reduced to 39⁰C (Beaudry et al., 2004). Vacuum drying is particularly advantageous because it allows water to vaporize at lower temperatures than under atmospheric conditions; therefore the drying operation can be maintained at much lower temperatures, reducing the impact on the food product (Beaudry et al., 2004; Yongsawatdigul & Gunasekaran, 1996a). Moreover, because air is excluded during drying, oxidation reactions are minimized (Gunasekaran, 1990). These characteristics all contribute towards minimizing the loss of color, flavor, lowering the damage to the texture despite high installation and operating costs (Yongsawatdigul & Gunasekaran, 1996a).

Wray and Ramaswamy studied the effect of microwave vacuum drying of microwave-based osmotically pre-treated cranberry halves on energy efficiency and quality characteristics of the final product (Wray & Ramaswamy, 2015a, 2015d). During this study, the microwave vacuum was applied for 12 to 95 min on MWODS (microwave osmotic dehydration under spray condition) of cranberries. The initial power density was determined to be approximately 10.2 W/g, and magnetron duty cycles ranged from 3 s on/27 s off for the 10% setting up to 15 s on/15 s off for the 50% setting. Cranberries were also dried using conventional hot air drying, freeze-drying and vacuum drying for comparison (Wray & Ramaswamy, 2015b). Results showed that the performance of MWV (microwave vacuum)-produced samples were next to freeze-dried sample and was far exceeded TP levels of the MWODS-AD sample, indicating that the process was much more proficient at maintaining phenolic compounds than air drying. Also, the study confirmed that MWV process is significantly better than the air-drying process for maintaining anthocyanin content. Overall the study concluded that the MWODS–MWV combination process was found to be suitable as a rapid method for the production of high-quality dried cranberries, by enhancing color, texture, and retention of health-positive compounds that are of increasing concern to consumers (Wray & Ramaswamy, 2015d).

2.8.4 Other microwave-based finished drying methods

2.8.4.1 Microwave freeze-drying

There are many methods available for finished drying process after the application of OD to achieve a desired moisture content level to make the product shelf-stable. The microwave freeze-drying (MFD) is one of the effective methods for drying of the food material. The freeze-drying allows for the removal of water by sublimation. Since the process operates below the triple point of water which transforms the water directly from its solid form (ice crystals) to a gas, bypassing the liquid state. Therefore, this process gives dried food with better quality characteristics than any other drying method. However, the process is very expensive, time-consuming, and consumes a high amount of energy. Therefore, these factors are addressed in the microwave freeze drying (MFD) technique. The technique was firstly introduced due to its time-saving advantage. However, some technical problems restricted the use of MFD on the industrial basis. moreover, arcing due to high vacuum, non-uniform distribution of the microwave field

which can in some cases lead to overheating and quality deterioration (Duan et al., 2007), are few of the issues with MFD.

Regardless of these issues, the studies are still focused on MFD technique, where one of the study observed 35.7% of energy-saving along with 40% shorter drying times when compared with FD (Jiang et al., 2013). During this study, it was also determined that banana chips when a process at the highest applied power density (2W/g) produced acceptable to consumers (Jiang et al., 2013). When the beef was subjected to MFD, it was concluded that the MFD process was particularly beneficial to large beef pieces, where drying times were most significantly reduced from traditional freeze-drying. Also been applied in the production of instant vegetable soup mixes, where it was observed that drying time and product quality decreased with increasing microwave power, while too low a power led to excessively long drying times (Wang et al., 2010; Wang et al., 2009).

2.8.4.2 Microwave Fluidized/Spouted Bed

Application of microwave energy during spouted or fluidized bed drying is an emerging field of research to overcome the issues of microwave heating such as thermal runaway. Microwave assisted fluidized-bed (MFB) ensures the proper mixing of the sample and more even distribution of the applied energy throughout the sample load and therefore, enhances product quality as well as the rate of moisture removal. The MFB was found to be 2-5 times faster than fluidized bed (FB) (Stanisławski, 2005). For macaroni beans, the drying time was reduced by 50% whereas for peppercorns it was 80-90% (Goksu et al., 2005; Kaensup & Wongwises, 2004). The 50% drying time reduction was also recorded with shelled corn and results were able to be accurately modelled using artificial neural network based approach (Momenzadeh et al., 2011). A decreased drying time by 83.39-98.07% and energy consumption by 82.07-95.22% was reported with soybeans (Zare & Ranjbaran, 2012).

The microwave-spouted bed (MSB) technique recently explored by many researchers on various food samples such as sweet potato (Liu et al., 2015; Liu et al., 2014), lettuce cubes (Feng et al., 2012), bulgur and wheat (Kahyaoglu et al., 2010), wheat (Jumah & Raghavan, 2001; Kahyaoglu et al., 2012), potato (Yan et al., 2010). The method was also employed with potato cubes in order to create a product similar to deep-fried potato chips as a healthier snack

alternative, where the resulting product had acceptable breaking force, expansion ratio, and rehydration ratio (Yan et al., 2010). A further derivatization has been the development of pulse-spouted bed microwave freeze drying (PSMFD). In the case of lettuce slices, PSMFD was found to reduce color changes, shrinkage, and final moisture content as compared to static MFD, while also reducing the drying time by 20% (Wang et al., 2013). Additionally, once rehydrated the samples were harder and more elastic than those dried via either static MFD or traditional FD (Wang et al., 2013). In the case of banana cubes, it was found the PSMFD was able to uniformly dry the sample than static MFD and maintain ascorbic acid values at those found in traditional FD while reducing drying time by 50% (Jiang et al., 2014). PSMFD was also shown to produce desalted duck egg white powders with better color, lower apparent density, and shorter drying times as compared to conventional FD (Wang et al., 2013).

PREFACE TO CHAPTER 3

The brief overview about the process presented in chapter 2 explained that, there are many ways to accomplish new outcomes and ways to improve the osmotic dehydration process. The previous publications in this series of research, highlighted the effectiveness of microwave osmotic dehydration in both the immersion and spray based microwave-osmotic processes (MWODS and MWODI, respectively) with cut fruit particles (apple cylinders) as well as whole fruit (cranberry) with sucrose as the osmotic agent. However, there was no emphasis on the application of other osmotic solutes to understand the efficiency of the process. In addition, the new outcome with a tropical fruit product was achieved which might be of interest in the process of MWODS. Therefore, in this chapter the enhancement of effectiveness of the MWODS treatment was studied using various osmotic solute mixtures based on the molecular weight of the solute and the results were compared with commonly used sucrose solution, on the mass transfer and quality of MWODS product.

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

Part of this study has been used in the following presentations and publications:

Shinde, B. and Ramaswamy, H.S., 2016. Evaluation of the influence of osmotic solutes on microwave-osmotic dehydration of mango under continuous flow medium spray (mwods) conditions. Presented as a poster at the Northeast Agricultural and Biological Engineering Conference (NABEC) 2016. July 31- August 3, 2016, Orono, ME, USA.

Shinde, B. and Ramaswamy, H.S., 2019. Evaluation of mass transfer kinetics and quality of microwave-osmotic dehydrated mango cubes under continuous flow medium spray (MWODS) conditions in sucrose syrup as moderated by dextrose and maltodextrin supplements. *Drying Technology*, 1-15.

CHAPTER 3

Evaluation of mass transfer kinetics and quality of microwave-osmotic dehydrated mango cubes under continuous flow medium spray (MWODS) conditions in sucrose as moderated by dextrose and maltodextrin supplements

Abstract

Microwave-osmotic dehydration under continuous spray condition (MWODS) has been demonstrated as an efficient method to enhance moisture loss using sucrose syrups under various process and product related factors. Product dependent factors such as solute type, solute mixtures, concentration, and solutes combinations can also influence the mass transfer properties. This study was specifically focused on evaluating the influence of added dextrose and maltodextrin to sucrose solution on the MWODS drying of mango cubes which has never been studied. Process variables were: different combinations of sucrose, dextrose, and maltodextrin at selected temperature and concentration of the osmotic solution. Results demonstrated that dextrose enhanced the moisture loss (ML) to 61.7% while maltodextrins helped to limit the solid gain (SG) to 4.8%. The superior performance of sucrose:maltodextrin (85:15) combination was demonstrated resulting in higher ($P<0.05$) ML/SG ratio of 11 which was indicative of better quality retention in the osmotically dehydrated mango cubes. This combination also gave a product with better texture and appearance factors.

3.1 Introduction

Mango is a very popular fruit worldwide and is second highest in terms of production of tropical fruits after banana (Mitcham & McDonald, 1992). Apart from being used in the fresh state, a significant amount is processed into a variety of products such as canned fruit slices, juice, puree, beverage, jams, etc. A considerable portion is also preserved as frozen and dehydrated products. The fruit is highly perishable because it is often harvested during rainy seasons. New avenues for preservation and marketing to reduce its postharvest losses and improve utilization.

Dehydration is the process used to preserve food by reducing moisture content in the food. The basic philosophy of the dehydration process is to reduce the water activity (a_w) in food which ultimately helps to reduce the microbial and enzymatic activity and make the product shelf stable. The stability of dehydrated fruit products can be ensured by maintaining a_w lower than 0.7 (Ramaswamy & Marcotte, 2005) which minimizes the quality deterioration during storage and makes the product available throughout the season without resorting to the more expensive refrigerated and frozen storage practices. There are several dehydration techniques available in a commercial practice which can be used for a wide variety of food products. In most cases, these techniques are carried at higher temperatures and lower humidity conditions in order to accelerate the drying process which is often unable to protect natural taste, nutritional quality, rehydration characteristics and physicochemical and sensory characteristics of the dehydrated food.

Osmotic dehydration (OD) has been generally recognized as a dehydration pre-treatment which can result in better quality retention, better energy conservation, and a better acid to sugar ratio in dried fruits with superior sensory properties and such products have been used as useful ingredients in many processed food formulations or complex foods. It has been demonstrated in previous work that osmotic pretreatment reduces enzyme activity and product water activity along with minor changes in product characteristics (Giraldo et al., 2003). It also reduces enzymatic browning (Giraldo et al., 2003) and often retains or improves color (Conway et al., 1983; Giannakourou & Taoukis, 2003; Giraldo et al., 2003) and texture (Huxsoll, 1982; Robbers et al., 1997; Talens et al., 2002) of the food product. Osmotic dehydration is a water removal technique, in which horticultural products such as fruits and vegetables are immersed in a hypertonic solution for a physical separation of moisture from the fruit to the concentration

solution due to osmotic forces. In general, in osmotic dehydration there is a significant amount of moisture loss but also the process is associated with a simultaneous impregnation of solids from the osmotic solution (Azuara et al., 2002; Kaymak-Ertekin & Sultanoglu, 2000; Raoult-Wack, 1994). Because this moisture loss is facilitated without causing the moisture to be vaporized as in traditional dehydration processes, significantly lower energy is required for accomplishing the moisture removal. Further, since the moisture content of osmotically dehydrated products is still generally high, they are usually coupled with other finished drying processes such as air drying, vacuum drying or freeze drying to reduce the moisture content to the final target level. Generally, the combination osmotic drying and a second stage air drying has been demonstrated to yield a better quality product and energy savings (Azuara et al., 2002; Raoult-Wack, 1994). However, one of the serious problems with osmotic dehydration is that the process is inherently very slow often requiring several hours for reducing the initial moisture content by about 50%.

Several techniques have been employed to enhance the osmotic dehydration rate of fruits and vegetables. The traditional osmotic dehydration process is a considerably slow and time consuming process and therefore, number of techniques such as pulsed electrical field (Andrés et al., 2007), pulsed vacuum (Ito et al., 2007), ultrasound (Rodrigues & Fernandes, 2007), high pressure (Rastogi & Niranjan, 1998), and microwave (MW) drying (Krokida et al., 2000; Krokida & Maroulis, 1997) have been studied to accelerate the process of dehydration. Most of the osmotic dehydration studies were focused on enhancing one-way mass transfer such as improving moisture loss but restricting the other by limiting the solids gain. The application of microwave energy has proven to be effective in improving moisture loss (Azarpazhooh & Ramaswamy, 2009b; Li & Ramaswamy, 2006c; Wray & Ramaswamy, 2013). Since the microwave process can effectively dry the food products. Microwave energy has proven to be effective in drying for several reasons. One of the reasons is that it generates heat by exciting dipolar components such as water molecules and polarizing ionic salts. The orientation of excited molecules under the microwave field produces heat within the samples which is known as volumetric heating (Orsat et al., 2007). This builds up pressure within the food sample and forces water to move out from the food product (Sosa-Morales et al., 2010). Therefore, heat produced due to microwave energy is generated throughout the food product which is uniform and rapid heating (Beaudry et al., 2004).

In microwave-assisted osmotic dehydration, the increased rate of moisture loss under microwave conditions results in reduced solids gain (Wray & Ramaswamy, 2013). The effective application of microwave-assisted osmotic drying was demonstrated by Li and Ramaswamy (Li & Ramaswamy, 2006c) under immersion condition and by Azarpazhooh and Ramaswamy (Azarpazhooh & Ramaswamy, 2009b) using solute spray conditions. During these studies, apple cylinders were treated with sucrose solution under microwave-osmotic dehydration immersion (MWODI) and spray (MWODS) and found to produce high moisture loss (ML) and reduced solids gain (SG) than the conventional osmotic drying. The first MWOD study made use of an immersion technique (MWODI) with a continuous flow of osmotic solution showing significant improvements over the conventional osmotic dehydration (COD) (Li & Ramaswamy, 2006c). This was further improved in a later study replacing the immersion unit with a spray system (MWODS) providing more contact between MW and fruit pieces. In these studies, the concept of ML/SG was introduced as an index of product quality, with higher ML/SG promoting better quality.

Another aspect which has been well explored in COD is the use of alternate solutes or solute combinations for enhancing osmotic dehydration kinetics. Hawkes and Flink (Hawkes & Flink, 1978) investigated the cost effectiveness of osmotic dehydration process using binary mixtures of solutes and reported that sucrose alone or in combination with other solutes could give a high level of water loss from the fruit piece and result in high solids content prior to air- or vacuum-dehydration. Moreover, osmotic solutions that contain sucrose with either salt, maltodextrin, or lactose are more effective than sucrose alone at the same total concentration (Hawkes & Flink, 1978). It was also studied that the binary and ternary osmotic solutions can reduce total aerobic viable microbial counts of meat during storage at 4⁰ C for 9 days (Dimakopoulou-Papazoglou & Katsanidis, 2017). The application of non-conventional carbohydrates such as oligofructose and maltodextrin showed better protection when concerning color and sensory attributes than conventional osmotic agents (glucose) (Dermesonlouoglou et al., 2016). The behavior of various osmotic agents such as sorbitol, sucrose, maltodextrin, fructose was studied and reported that lowest and highest solids gain was achieved with maltodextrin and fructose respectively (İspir & Toğrul, 2009b). In addition the vitamin C in tomatoes was retained when osmotically pretreated with maltodextrin solute mixture and stored at -20⁰ C for 12 months (Dermesonlouoglou et al., 2016). Also it was found that the higher

maltodextrin concentration favors volume loss, enhances water loss as well as achieves better sensory attributes (Azuara et al., 2002). The study on osmotic dehydration of blueberries using maltodextrin concluded that the solute maltodextrin enhanced palatability, good color, and preferable texture of processed blueberries (Chun et al., 2012). However, the ultimate choice of the blend will depend on factors such as solute cost, type of product, the effect of the solutes on the processed product. However, such concepts have never been tested in MW environment. Since solutes and water can influence microwave heating, their role in MWOD application should be of interest. The proposed study is based on the concept of using more than one osmotic agent to improve the efficiency of MWODS process.

The aim of this study was therefore to evaluate the influence of dextrose and maltodextrin addition to sucrose solution in different proportions on the mass transfer and quality factors of mango cube under MWODS processing conditions. The molecular weight (M_w) of dextrose (180.2 g/mol) is lower than sucrose (342.3 g/mol) on the contrary maltodextrin has a higher range of molecular weight than sucrose (9000-155000 g/mol) (Avaltroni et al., 2004), which may react differently during MWODS process. From OD kinetics point of view, the purpose was to evaluate their potential to enhance moisture loss (ML) and limit solids gain (SG) (increase ML/SG ratio) during the MW osmotic dehydration process.

3.2 Materials and methods

3.2.1 Raw materials

Commercial grade sucrose (Lantic Sugar Ltd., Montreal, Qc, Canada), dextrose and maltodextrin-10DE (Univar Pvt. Ltd, Canada) were used in conjunction with tap water for preparing the osmotic solution, and the concentrations were maintained on a wet basis (w_b). Frozen mango pieces were obtained from a local food freezing company (Nature's Touch, Canada) and kept frozen (-21°C to -27°C) until use. Prior to use, the mango pieces were thawed overnight (8-10 h) in a refrigerator (4°C - 7°C). The dimensions of the mango pieces were measured as approximately 1.9 cm X1.7 cm X1.3 cm, which is a cuboid shaped mango piece. The quality parameters of frozen-thawed mangoes were measured as L^* 36.8 (± 3.4), a^* 9.9 (± 2.0), b^* 37.1 (± 3.1), hardness 600g (± 50), and chewiness 197g mm (± 19). Also, the similar quality parameters of fresh raw mangoes (obtained from local store) were measured to have a clear view of the total processing effect on quality characteristics of the mango fruit. The color

and texture parameters of fresh mangoes are: L^* 42.8 (± 7.68) a^* 13.1 (± 1.99), b^* 42.3 (± 8.64), Hardness 1568g (± 219) and Chewiness 628g mm (± 192). The moisture content of the frozen-thawed (untreated) mango cube was measured using AOAC method (AOAC, 1975), by keeping the cubes in an oven at 105°C for approximately 24h (until a constant weight was achieved). The moisture content of the mango cube was determined as 86.1% (wb) on average.

3.2.2 Microwave Setup

The schematic of the experimental setup is illustrated in Figure 3.1 and the details of which can be obtained from Azarpazhooh and Ramaswamy (Azarpazhooh & Ramaswamy, 2009b) or Wray and Ramaswamy (Wray & Ramaswamy, 2015b). Briefly, MWODS assembly consisted of a domestic microwave (Danby DMW1153BL 0.031 m³, Guelph, ON, Canada) with a nominal power output of 1100W and 2450MHz, which contained a spray head (Waterpik, Model RPB-173C, 12.5cm diameter, Waterpik Technology Inc., Markham, ON, Canada) attached to the custom-made glass sample chamber (12.5cm diameter). The tested samples were placed in a nylon mesh on the porous acrylic plate “stage” inside the glass sample chamber. The acrylic stage allowed to drip down the sprayed osmotic solution, while keeping the samples in contact with the microwave. The osmotic solution collected at the bottom of the sample chamber was recycled through series of coils- immersed inside a steam jacketed water bath (Model TDB/4 Groen division, dover corp, IL) and then pumped through the spray head, using a peristaltic pump (Model 75211-30 Digital gear pump, Barnant company IN).

The temperature of the water bath was set to the inlet temperature of the osmotic solution, and the osmotic solution was then circulated through the assembly to equilibrate the temperature before putting the samples into the system. The temperature of the osmotic solution was monitored using a pair of in-line Type-T thermocouples connected to a digital thermometer (Omega DP-462, Omega technology, Laval, QC). The thermocouples were placed immediately before and after the microwave cavity to measure the temperature of the solution before going inside the microwave and immediately after coming out of the microwave. The increase in the temperature of the osmotic solution after residing the microwave cavity was about 4-5°C, which was further passed through the series of coils under the water bath to obtain required temperature before coming back into the microwave. The series of coils were made with a sufficient length to allow a solution to sample of 30:1 ratio. The large amount of osmotic solution in the closed

system allowed to maintain a constant solute concentration throughout the experiments for each solute type, which was measured using a handheld refractometer.

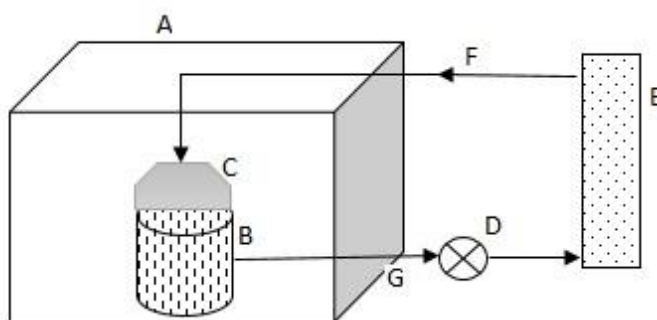


Figure 3.1: Schematic diagram of MWODS (A: microwave oven cavity, B: microwave transparent sample chamber, C: spray head, D: digital gear pump, E: water bath (containing heat exchanging coils, not pictured), and F and G are thermocouple measuring points immediately before and after the solution enters and leaves the microwave cavity, respectively.)

3.2.3 Experimental procedures

3.2.3.1 MWODS experiments

The system was setup, and the osmotic solution was preheated according to the selected temperature of the run type. The osmotic solution at a fixed flow rate was allowed to flow through the system with the microwave turned fully on (100% power level) during the experiments. Each batch consisted of two nylon mesh bags, each filled with 50 ± 1 g mango pieces which means each test run consisted of 100 ± 2 g mango samples in total. These mangoes were kept in a single layer in two small Nylon mesh bags and placed on the perforated acrylic stage in the sample chamber. The test runs were carried out for 30 min after which the pump was stopped, and the samples were removed. The excess osmotic solution adhering to the product surface was shaken off and wiped with a wet paper towel, and the mango cubes were then weighed again. The treated samples in one of the bag was then used for moisture determination and the other for quality evaluation. Moisture content was determined in a dry oven set at 105°C for approximately 24 h (AOAC, 1975) (dried to constant weight).

3.2.3.2 Experimental design

Experimental design for this research work was based on three variable parameters such as concentration, temperature and solute mixture proportions. The concentration of solute mixture (S+D, S+MD and S) was studied at 40% and 60% levels. Since the concentration less than 40%

will not be effective enough for heavy moisture reduction in a short time; whereas, osmotic solute concentration more than 60% will strongly affect the solute flow through the spray head due to its high viscosity. In addition, the temperatures were studied at 40°C, 50°C, and 60°C. The temperature less than 40°C will not be effective for active movement of water molecules, whereas temperatures more than 60°C will damage the quality of the product by initiating the browning process. Hence, the temperature range was kept between 40°C to 60°C.

The range of secondary solute (dextrose and maltodextrin) was kept between 10% to 15% by keeping sucrose as a primary or major solute (90% to 85%). These proportions were adopted from previous research, where the researcher used this proportion for conventional osmotic dehydration (Azuara et al., 2002). It would be an interesting aspect to see the effect of these solute with a given proportion under MWODS. It would be difficult to understand the effect of secondary solute when used below 10% concentration level. On the other hand, the concentration of secondary solute more than 15% will affect the mass transfer parameters, and it will increase the number of experiments to study. It was reported in the previous study that the flow rate has a minor effect on the MWODS process in comparison with the other processing factors. Therefore, the flow rate (1050mL/min) was also kept constant. Hence to minimize the experimental runs and to obtain a desirable number of experiments, it was decided to keep flow rate and contact time(30min) as a constant parameter.

Experimental design for MWODS using two solute combinations, by keeping sucrose as one of the primary solutes, is described in Table 3.1. This experimental design was used with three variables, such as solute concentration, fraction of solutes in osmotic solution (sucrose to dextrose or sucrose to maltodextrin fraction) and temperature while keeping contact time (30 min) and flow rate (1050mL/min) respectively at constant levels.

3.2.4 Mass transfer kinetics

Moisture loss (ML), solids gain (SG), moisture loss to solids gain ratio (ML/SG) and weight reduction (WR) were obtained using the following equations:

$$\% \text{ Moisture Loss (ML)} = 100 \frac{M_0 X_0 - M_t X_t}{M_0} \quad (3.1)$$

$$\% \text{ Solids Gain (SG)} = 100 \frac{M_t S_t - M_0 S_0}{M_0} \quad (3.2)$$

$$\text{ML: SG ratio} = \frac{\%ML}{\%SG} \quad (3.3)$$

$$\% \text{ Weight Reduction (WR)} = 100 \frac{M_0 - M_t}{M_0} \quad (3.4)$$

where M_0 and M_t are the total mass of the fruit sample at time 0 and time t , respectively; X_0 and X_t are the moisture fractions (kg/kg, wet basis) at time 0 and time t , respectively; S_0 and S_t are the solid fractions (kg/kg, wet basis) at time 0 and time t , respectively.

The above Eqs. (3.1-3.4) were used assuming a uniform mass transfer of solids into the product and considering that there is no significant loss of solids from the sample into the solution. In brief, the mass transfer parameters were calculated by weighing the samples before MWODS treatment (M_0), after treatment (M_t) and by using oven drying method to measure the post-MWODS solids contents (S_t). The initial solids fraction (S_0) was calculated as the difference between the mass and the moisture content of fresh (frozen thawed) mango cubes. For each experimental run, the four duplicates with average values were used. Osmotic dehydration kinetic data gathering is somewhat different compared to other dehydration tests in that separate experiments have to be run for each treatment temperature-time combinations.

3.2.5 Quality analysis

For each run type, the quality parameters were measured as elaborated below. Since the process of MWODS is new in drying technologies, so this will be the matter of interest to investigate the effect of the processing factors, especially different types of solutes, on quality characteristics of the samples.

3.2.5.1 Texture evaluation

Texture profile analysis (TPA) of both the MWODS-treated samples and frozen thawed mango cubes were obtained from a TA.XT Plus Texture Analyzer (Stable Microsystems, Surrey, UK). To obtain TPA, a two-cycle compression test was performed using a flat bottom probe of 25 mm diameter, with pretest speed of 5 mm/sec, the test speed of 5 mm s⁻¹ and post-test speed of 5 mm s⁻¹. The target compression was about a distance of 3 mm into the sample during two consecutive cycles to target a 25% deformation from the average height of samples. These settings with minor modifications were used with guidance from Banjonginsiri et al.

(Banjongsinsiri et al., 2004), who used TPA on mango cubes. The analysis was performed with six replicates, and the average values (with standard deviation) were used.

Table 3.1: Experimental design for MWODS of mango cubes

Run	Solute	Solute Fraction	Solute Concentration	Solute Temperature
1	S (Sucrose)	100:0	40%	40 ⁰ C
2				50 ⁰ C
3				60 ⁰ C
4			60%	40 ⁰ C
5				50 ⁰ C
6				60 ⁰ C
7	S:D (Sucrose:Dextrose)	90:10	40%	40 ⁰ C
8				50 ⁰ C
9				60 ⁰ C
10			60%	40 ⁰ C
11				50 ⁰ C
12				60 ⁰ C
13	S:D (Sucrose:Dextrose)	85:15	40%	40 ⁰ C
14				50 ⁰ C
15				60 ⁰ C
16			60%	40 ⁰ C
17				50 ⁰ C
18				60 ⁰ C
19	S:MD (Sucrose:Maltodextrin)	90:10	40%	40 ⁰ C
20				50 ⁰ C
21				60 ⁰ C
22			60%	40 ⁰ C
23				50 ⁰ C
24				60 ⁰ C
25	S:MD (Sucrose:Maltodextrin)	85:15	40%	40 ⁰ C
26				50 ⁰ C
27				60 ⁰ C
28			60%	40 ⁰ C
29				50 ⁰ C
30				60 ⁰ C

Note: S=Sucrose, S:D= Sucrose: Dextrose, S:MD= Sucrose: Maltodextrin

A wide range of responses such as hardness, chewiness, adhesiveness, cohesiveness, factorability, gumminess, and springiness can be obtained from TPA analysis (Bourne, 2002). Hardness and chewiness were selected in this study as parameters to determine the texture influence of osmotically processed mango cubes. Hardness was defined by the peak force during the first compression cycle and chewiness was obtained from the product of gumminess and springiness (Bourne, 2002). Chewiness was calculated using Equation 3.5.

$$\text{Chewiness} = \text{Gumminess} \times \text{Springiness} \quad (3.5)$$

3.2.5.2 Color

Color values of the MWODS treated samples were evaluated in the L^* , a^* , b^* system using a tristimulus Minolta Chroma Meter (Minolta Corp., Ramsey, NJ, USA). The Chroma Meter was warmed up 20 min prior to use and the color was calibrated against a white standard. Six measurements were made with each sample, and the values were averaged to obtain the L^* (lightness), a^* (green (-) to red (+)), and b^* (blue (-) to yellow (+)) values of the individual trials. The ΔE (total color change) was also determined as per the Equation 3.6 (Maftoonazad & Ramaswamy, 2008).

$$\Delta E = \sqrt{(L_0 - L)^2 + (a_0 - a)^2 + (b_0 - b)^2} \quad (3.6)$$

3.2.6 Data Analysis

Experiments were performed in four replicates and mean values with standard deviations are presented in graphs. The analysis of variance was performed using JMP® v-13 (SAS Institute Inc., Cary, NC., U.S.A) to compare the results at 95% confidence level. The paired t-test was performed to understand the significant difference between the overall performance of each solute mixtures based on mass transfer and quality parameters.

3.3 Results and discussion

3.3.1 Mass transfer kinetics during MWODS

3.3.1.1 Moisture loss

The calculated values along with percent standard deviations of the moisture loss (ML), solids gain (SG), weight reduction (WR) and moisture loss to solids gain ratio (ML/SG) are presented in Figure 3.2. It can be observed from Figure 3.2 (A) and (B) that, more than 35% ML

occurred during 30 min MWODS process, under all experimental conditions. This ML is within the range of the moisture loss observed in previous studies of MWODS (Azarpazhooh & Ramaswamy, 2009b; Li & Ramaswamy, 2006c; Wray & Ramaswamy, 2015b). The reason behind heavy moisture loss is the rapid heating of water molecules in the presence of microwave increases the internal pressure and promotes quick removal of water molecules from the fruit piece (Azarpazhooh & Ramaswamy, 2009b). The moisture loss was more prominent with a gradual increase in temperature and concentration during MWODS process, as shown in Figures 3.2 (A) and (B).

The highest moisture loss was found with S:D-85:15 > S:MD-85:15; S:D-90:10 > S:MD-90:10 at 40% and 60% syrup concentration and at all temperature levels (40⁰C, 50⁰C and 60⁰C). The solute mixtures containing dextrose (S:D) showed better ML (moisture loss) than maltodextrin (S:MD) at different temperatures. The possible explanation for this outcome is that the addition of lower molecular weight dextrose exerts higher osmotic potential than sucrose alone and hence contributes to higher moisture loss. The similar outcomes were evident in previous study by Contreras and Smyrl (Contreras & Smyrl, 1981), where they argued that higher moisture loss by low molecular solutes is due to the ease of their penetration into the samples and the removal of the moisture out of the sample, whereas the high molecular weight solutes will have difficulty impregnating into the sample and removing moisture out of the sample. In other words, the lower water loss in the presence of MD than D at equal mass concentration was due to much higher melting point of high molecular weight MD. Therefore, the molar concentration of MD in solution remain lower and hence the lower driving force for diffusion. On the other hand, the heavy water loss of low molecular dextrose was mainly due to high osmotic potential. The high molecular maltodextrins resulted in lower ML at both solute concentrations and at all temperatures. The higher molecular weight maltodextrin could form a coating along the surface of the samples, thereby limiting the sugar intake into the sample. Thus this phenomenon helps to maintain the osmotic potential and ultimately makes way for further removal of additional moisture from the fruit (Azuara et al., 2002). Therefore, it was found that S: MD showed better ML than even sucrose (S-100:0) alone. A similar trend was also observed in a previous study by Azuara et al. (Azuara et al., 2002) demonstrating the use of maltodextrin is not only reducing the SG but also improving the ML when compared with the application of only sucrose as an osmotic agent. Hence, the ML of samples treated in sucrose only solution (S-

100:0) was the lowest among all test samples. Hawkes and Flinks (Hawkes & Flink, 1978) also found that osmotic dehydration in sucrose with the addition of other solutes improved the effectiveness of moisture removal in comparison to sucrose alone. When MD was used with cubed apricot for osmotic dehydration process, it was reported that the process gave 26% ML (İspir & Toğrul, 2009b). Similarly, the OD of beef using 60% MD and 15% NaCl gave 51% ML when treated for 3h at 15⁰C (Dimakopoulou-Papazoglou & Katsanidis, 2017). The monosaccharide glucose and polysaccharide high dextrose equivalent maltodextrin (HDEM) were applied for OD of tomatoes where they noticed 8.43 and 8.42 % ML with glucose and maltodextrin, respectively when treated for 1 h at 35⁰C conc. 56.5% (Dermesonlouoglou et al., 2016). On the other hand, MWODS process using sucrose and maltodextrin solute mixtures were giving better ML than sucrose that is 59.2% at 60% conc. and 60⁰C temp. in 30min, which is the fastest moisture removal process when compared with the other studies (Dermesonlouoglou et al., 2016; Dimakopoulou-Papazoglou & Katsanidis, 2017; İspir & Toğrul, 2009b).

Consistent with other osmotic dehydration studies (Karathanos et al., 1995; Lerici et al., 1985), temperature and concentration of osmotic solution showed a significant effect on ML performance of MWODS-mango samples. With respect to the temperature, the highest ML of 59.6% and 61.7% were observed with S:D-85:15 solute at 60⁰C and under 40% and 60% concentration levels, respectively. Also, with respect to the concentration levels, the highest ML of 59.6% and 61.7% were reported with S:D-85:15 solute both at 60⁰C temperature.

3.3.1.2 Solids Gain

The effects of solute type and different solute combinations with sucrose are shown in Figure 3.2 (C) and (D). The lower levels of SG were observed with S:MD-85:15 and S:MD-90:10 combinations at all temperature and syrup concentration levels. The overall trend for SG S:MD-85:15 < S:MD-90:10 < S (100:0) < S:D-90:10 < S:D- 85:15, was almost exactly as the hypothesis of the proposed research and the experimental design. More specifically, the lowest SG values of 5.0 % and 5.2 % were found with S:MD–85:15 at 40⁰C/40% and 40⁰C/60% process combinations, respectively.

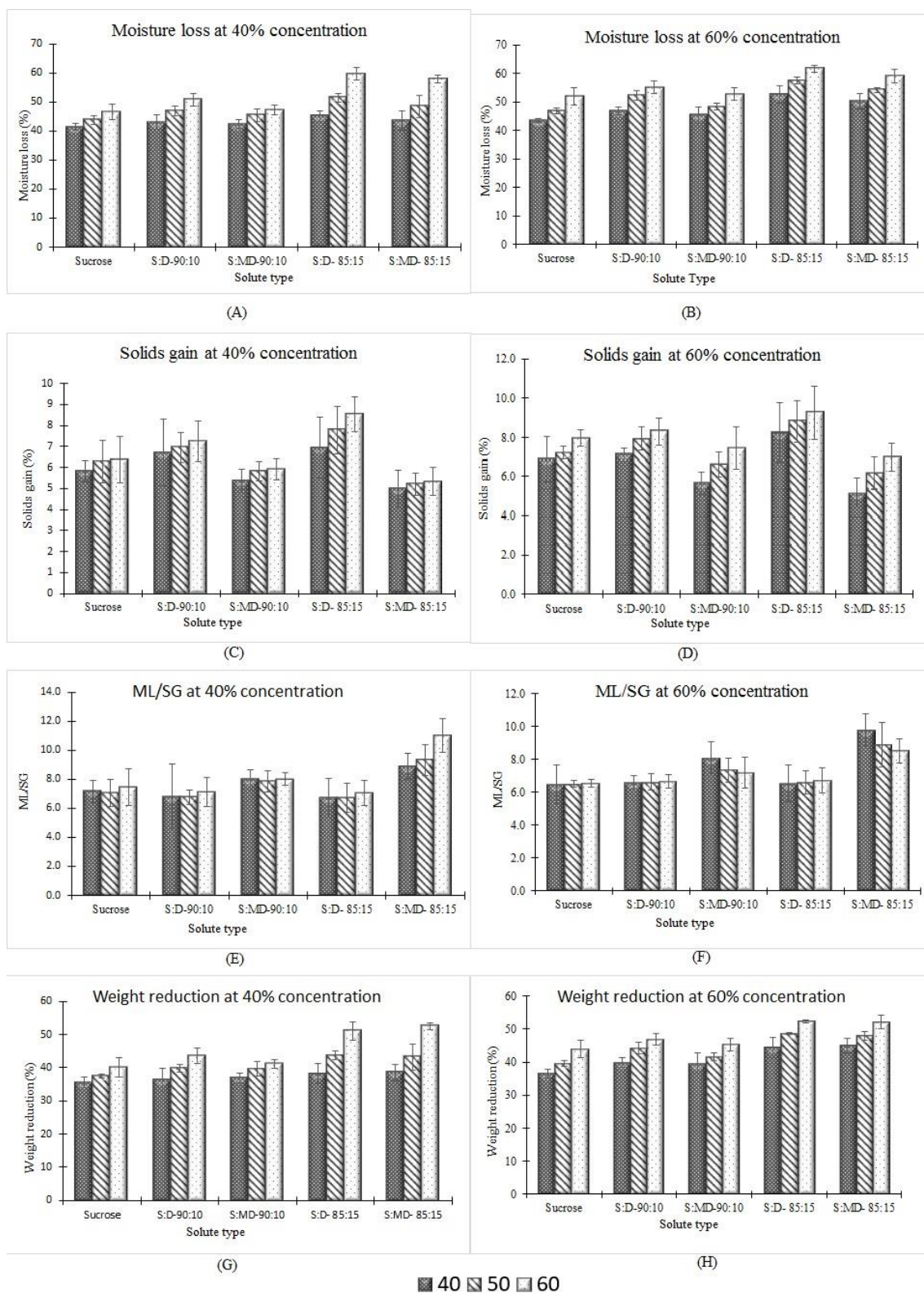


Figure 3.2: Moisture loss, solids gain, moisture loss to solids gain ratio and weight reduction for various solutes combinations and concentrations. (Average values with standard deviation shown)

This confirms the earlier discussion that, higher molecular weight solutes such as maltodextrins will form a coating on the surface of the fruit pieces and restrict the intake of osmotic solids into the mango samples. Hawkes and Flink (Hawkes & Flink, 1978) also reported that solids uptake is inversely correlated with the molecule size of the osmotic agent. Previous research recorded the same observation, where solids gain was limited in beef when treated with maltodextrin (Dimakopoulou-Papazoglou & Katsanidis, 2017). The lowest solids gain was also recorded when apricots were treated with maltodextrin solution (İspir & Toğrul, 2009b). In contrast, the highest SG of 8.53% and 9.29% were observed with S:D-85:15 at 40% and 60% concentration level, respectively. This confirms the role of added low molecular weight solute (dextrose) enhancing the SG potential of sucrose facilitated by the easier penetration possibility of lower molecular weight solutes from the osmotic solution into the fruit products (Raoult-Wack, 1994). It was also concluded in previous research studies that the low molecular weight solutes could be considered as best infusion agents (Shi & Le Maguer, 2002). The SG in the medium molecular weight sucrose solution, therefore, was moderated by added solutes according to their molecular weights relative to sucrose. The similar studies of conventional OD of blueberries with MD were showing 15% SG at 60 Brix 60°C temp. in 360 min. (Shi & Le Maguer, 2002); in other study when MD was used with cubed apricot the SG was measured around 42% at 70% and 45°C in 24h. (İspir & Toğrul, 2009b). Also, with beef OD, when MD was used around 60% with 5%NaCl, the SG was recorded around 19% at 15°C for 3h (Dimakopoulou-Papazoglou & Katsanidis, 2017). On the other hand, the MWODS process using S:MD (85:15) was giving 5.32% and 6.98% at 40% and 60% conc. at 60°C temp and 30min. This comparative observation clearly indicates that the quick and fast MWODS method using MD is prominently reducing SG.

In addition, the SG in all test runs also increased with an increase in the temperature and concentration of osmotic solutes, as has been found with other osmotic dehydration studies without the use of microwave heating (Azarpazhooh & Ramaswamy, 2009a; Wray & Ramaswamy, 2013). The highest SG was observed at 60°C and 60% concentration.

3.3.1.3 ML/SG ratio

The particular interest in osmotic dehydration process lies in the ML/SG ratio parameter. The OD process involving this scenario favors better loss of ML with limited SG. The ratio of

moisture loss/solids gain (ML/SG) or water loss/sugar uptake (WL/SU), is known as dehydration efficiency index (DEI) (Matuska et al., 2006). This has been highlighted as the strength of MWOD tests carried out in our laboratory over the past several years demonstrating a significant advantage of MWOD relative to conventional OD (COD). In a series of studies, MWOD applications were progressively showed to have better ML/SG potential with the continuous flow of osmotic medium over the samples osmotically treated inside a MW oven first under the immersion mode and then under spray mode (Azarpazhooh & Ramaswamy, 2009b; Li & Ramaswamy, 2006c; Wray & Ramaswamy, 2013). In the OD process, it is desirable to promote ML to have the enrichment of fruit solids in the product rather than gaining solutes from an osmotic solution which is represented by the SG factor. This will make the product a healthier fruit snack. Since this is also desirable from a quality point of view, the ML/SG ratio can be used as an indicator of osmotic drying influence on product quality.

In all the test runs, ML was more prominent than SG, and hence ML/SG ratios were predominantly high. As shown in Figure 3.2 (E) and (F), ML/SG ratio at S:MD-85:15 was the highest, followed by S:MD-90:10 at both concentration levels and at all temperature levels. This resulted from the higher potential of MD combinations which resulted in higher ML in combination with lower SG. On the other hand, the S:D combination had a higher potential for ML but also resulted in higher SG thereby restricting the gains with respect to the ML/SG ratio (Islam & Flink, 1982b). Similar results were found in previous studies with glucose, which is a low molecular weight solute, causing higher solids enrichment and lower the ML/SG ratio (Lenart, 1996). It has been investigated that low molecular weight solute favors impregnation rather than water loss (Nelson & Datta, 2001). It was reported that the OD process using MD solute when applied on mango chips at 40°C and 65% conc. was giving ML/SG around 6.5 in 60min (Yolanda & Rosana, 2009) and when treated with tomatoes it was giving 7.06 at 35°C, 56.5% conc. in 1h (Dermesonlouoglou et al., 2016). Whereas, MWODS treatment using S:MD mixture was giving ML/SG of 11 at 60°C/40% for 30min. This comparative observation with previous studies clearly indicates the effectiveness of MWODS process while using two solute mixtures. The effect of temperature and concentration on ML/SG, can also be observed from Figure 3.2 (E) and (F), where elevated temperatures and concentrations had some mixed effects on ML/SG ratio in according to their influence on ML and SG. In our previous studies with MWOD, similar observations were found with respect to the influence of osmotic variables on

ML/SG ratio (Azarpazhooh & Ramaswamy, 2009b; Li & Ramaswamy, 2006c; Wray & Ramaswamy, 2013).

3.3.1.4 Weight Reduction

Weight loss is another indicator of the efficiency of the dehydration process. The WR results are shown in Figure 3.2 (G) and (H) which is based on ML minus SG which reflects the net effect on the product weight (Wray & Ramaswamy, 2013). It is a resulting situation which moderates the ML factor with the added SG. The weight reduction would have been much higher if not the solids from osmotic solution entered the fruit tissue. This type of combination is observed only in OD situation while in all other types of drying WR is directly related to ML and is only contributed by ML. In this study, with all the solute combinations, the highest weight reduction was observed with S:MD with 52.6% with S:MD-85:15 at 60°C/40% (temperature/concentration). When compared with the conventional OD, 30% WR was reported with cubed Apricot at 70% concentration in 24h. Hence, this comparison gives an idea about the effectiveness of MWODS treatment using solute mixtures which was giving an effective amount of WR (52.6%) in only 30min of time frame (İspir & Toğrul, 2009b).

The highest WR with S:D solute type was 52.4% at 60°C/60% process nearly same as above and observed with S:D-85:15. These two extreme situations arose from a moderated influence of MD and D on ML and SG. Higher ML situation would favor a better WR while higher SG would favor the opposite. The MD and D supplemented situations had mixed influences on ML and SG and resulted in similar results with respect to WR. A unique situation of higher ML was also observed with added MD in some test conditions. The larger molecular size of maltodextrin forming a surface layer on the mango cubes contributes to limit the solids gain. This can normally expect to also act as a barrier to ML. However, the ML was not restricted as much because this provided a higher moisture gradient potential from the inside out and hence also contributed positively to ML from the product. The lowest WR results of 35.5 % and 36.6 % were found with S-100:0 at 40°C/40% and 40°C/60%, respectively, with no influence from D and MD. This also brings out the importance of adding osmosis influencing solutes like D and MD for promoting better osmotic dehydration rates.

Table 3.2: t-test results for significance of differences in moisture loss, solids gain, ML/SG and weight reduction between different solute mixtures

Difference ↓	Responses →	Moisture loss			Solids gain			Weight reduction			ML/SG		
		DF	t-value	Pr> t	DF	t-value	Pr > t	DF	t-value	Pr> t	DF	t-value	Pr> t
S:MD(85:15)–Sucrose(100:0)		23	8.67	< 0.01	23	-5.41	< 0.01	23	10.4	< 0.01	23	9.48	< 0.01
S:MD (85:15)–S:D(90:10)		23	9.39	< 0.01	23	-8.18	< 0.01	23	6.23	< 0.01	23	8.12	< 0.01
S:MD(85:15)–S:D(85:15)		23	-4.06	< 0.01	23	-10.8	< 0.01	23	0.31	> 0.05	23	9.21	< 0.01
S:MD(85:15)–S:MD(90:10)		23	6.15	< 0.01	23	-2.74	< 0.05	23	7.07	< 0.01	23	6.55	< 0.01
S:MD(90:10)–Sucrose(100:0)		23	2.24	< 0.05	23	-2.96	< 0.01	23	3.83	< 0.01	23	4.41	< 0.01
S:MD(90:10)–S:D(90:10)		23	-3.96	< 0.01	23	-6.6	< 0.01	23	-1.71	< 0.05	23	4.22	< 0.01
S:MD(90:10)–S:D(85:15)		23	-9.09	< 0.01	23	-9.06	< 0.01	23	-7.14	< 0.01	23	5.64	< 0.01
S:D(85:15)–Sucrose(100:0)		23	12.0	< 0.01	23	5.23	< 0.01	23	9.61	< 0.01	23	-0.53	> 0.05
S:D(85:15)–S:D(90:10)		23	9.38	< 0.01	23	4.37	< 0.01	23	7.87	< 0.01	23	-0.2	> 0.05
S:D(90:10)–Sucrose(100:0)		23	6.31	< 0.01	23	2.40	< 0.05	23	4.61	< .0001	23	-0.35	> 0.05

(Note: values in bold are not significant)

3.3.2 Statistical results

The analysis of variance results using paired t-test is summarized in Table 3.2, which can be used to validate the significance of the influence of the different solute types and combinations. All the solute types had significant influence ($P < 0.05$) ML, SG, WR, and ML/SG, except a few combinations. For example, the difference between S:MD- 85:15 and S:D-85:15 was not significant for WR. The moderating effects of D and MD to WR contribution was explained earlier because of their differing effects on ML and SG. Hence, even though the solute types may be significant for ML and SG responses, their influence on WR and ML/SG might be variable.

3.3.3 Quality analysis

3.3.3.1 Texture

The mechanical properties of the product such as texture are usually measured after the completion of the final drying. This is because osmotic drying can only accomplish partial drying by removing a portion of the water present, so the residual moisture after OD is still too high for the product to remain stable. Therefore, finished drying techniques are used to complete the drying process. However, during this study, the application of binary solute mixture was firstly introduced and hence the influence of the binary solutes during MWODS process on the product was a concern, before subjecting it to the finished drying process. Hence the product quality immediately after MWODS was evaluated. The fresh-thawed mango cubes with higher moisture content produce better structural integrity and represents the product with better textural characteristics. Whereas, the dehydrated products may have varying effects on the texture and color properties. Figure 3.3 shows the typical force deformation curve obtained with a freshly-thawed mango sample prior to the OD treatments. Various textural attributes were computed from the force deformation curves, while hardness and chewiness were used for comparing the effect of different treatments.

Post-MWODS samples with a noticeable loss of hardness in comparison to freshly thawed mango are summarized in Table 3.3, which is in line with the previous findings (Khin et al., 2007). A decreased hardness of the sample, due to the destruction of the cellular structure most likely through increased solids gain, resulted in softening of the product after MWODS treatment, which is considered desirable in some products requiring a chewy structure (Wray & Ramaswamy, 2015b).

Table 3.3: t-test results for significance of differences in L*, a*, b*, ΔE , hardness and chewiness between different solute mixtures

Responses → Difference ↓	DF	L*		a*		b*		Delta E		Hardness		Chewiness	
		Pr> t	t-value	Pr> t	t-value	Pr> t	t-value	Pr> t	t-value	Pr> t	t-value	Pr> t	t-value
S:MD(85:15)– Sucrose(100:0)	35	< 0.01	-2.92	< 0.05	-2.07	< 0.01	4.48	< 0.01	-5.06	< 0.01	9.77	< 0.01	11.3
S:MD (85:15)– S:D(90:10)	35	< 0.05	2.67	>0.05	-0.88	>0.05	1.99	< 0.05	-2.31	< 0.01	11.1	< 0.01	11.4
S:MD(85:15)– S:D(85:15)	35	>0.05	-0.64	< 0.01	4.86	< 0.01	7.12	< 0.01	-7.07	< 0.01	11.1	< 0.01	13.1
S:MD(85:15)– S:MD(90:10)	35	< 0.01	4.17	>0.05	0.09	< 0.01	2.74	< 0.05	-2.38	< 0.01	9.78	< 0.01	9.17
S:MD(90:10)– Sucrose(100:0)	35	< 0.01	-8.18	< 0.05	-2.71	< 0.01	3.29	< 0.01	-3.94	< 0.01	4.35	< 0.01	4.21
S:MD(90:10)– S:D(90:10)	35	>0.05	-6.65	>0.05	-1.64	>0.05	-0.65	>0.05	0.17	< 0.01	7.08	< 0.01	6.40
S:MD(90:10)– S:D(85:15)	35	< 0.01	-5.32	< 0.01	6.20	< 0.01	3.27	< 0.01	-3.39	< 0.01	7.86	< 0.01	8.96
S:D(85:15)– Sucrose(100:0)	35	< 0.05	-2.44	< 0.01	-7.87	>0.05	-0.42	>0.05	0.17	< 0.01	-5.07	< 0.01	-6.73
S:D(85:15)– S:D(90:10)	35	< 0.01	3.17	< 0.01	-6.69	< 0.01	-4.17	< 0.01	3.86	>0.05	-1.15	< 0.01	-4.44
S:D(90:10)– Sucrose(100:0)	35	< 0.05	-2.57	>0.05	-0.84	< 0.05	2.34	< 0.05	-2.38	< 0.01	-3.73	< 0.01	-2.83

(Note: values in bold are not significant)

The S:D-85:15 samples showed the highest decrease hardness at 40% and 60% concentrations, followed by S:D-90:10 at respective temperature levels. In contrast, the hardness of S:MD-85:15 had the highest amongst all solutes, followed by S:MD-90:10 and S:100:0, as shown in Figure 3.4 (A) and (B). Similar results were evident in previous findings when the mango chips pretreated by OD in the presence of maltodextrin solute showed more firm and better texture (Yolanda & Rosana, 2009). The highest hardness of 238 g and the lowest hardness of 76.6 g was observed with solute S:MD-85:15 at 60%/60°C and S:D-85:15 at 60%/60°C, respectively. The trend of increased hardness with increased solute temperature is in line with the findings of Matuska et al. (Matuska et al., 2006) , where it was reported that an increase in temperature leads to changes in the cell membrane structure resulting from a longer period of drying (lower moisture content). In contrast, the trend towards decreased hardness with increased temperature was associated with S:D-90:10 and S:D-85:15. The results favor the inclusion of MD for better texture retention in mango cubes.

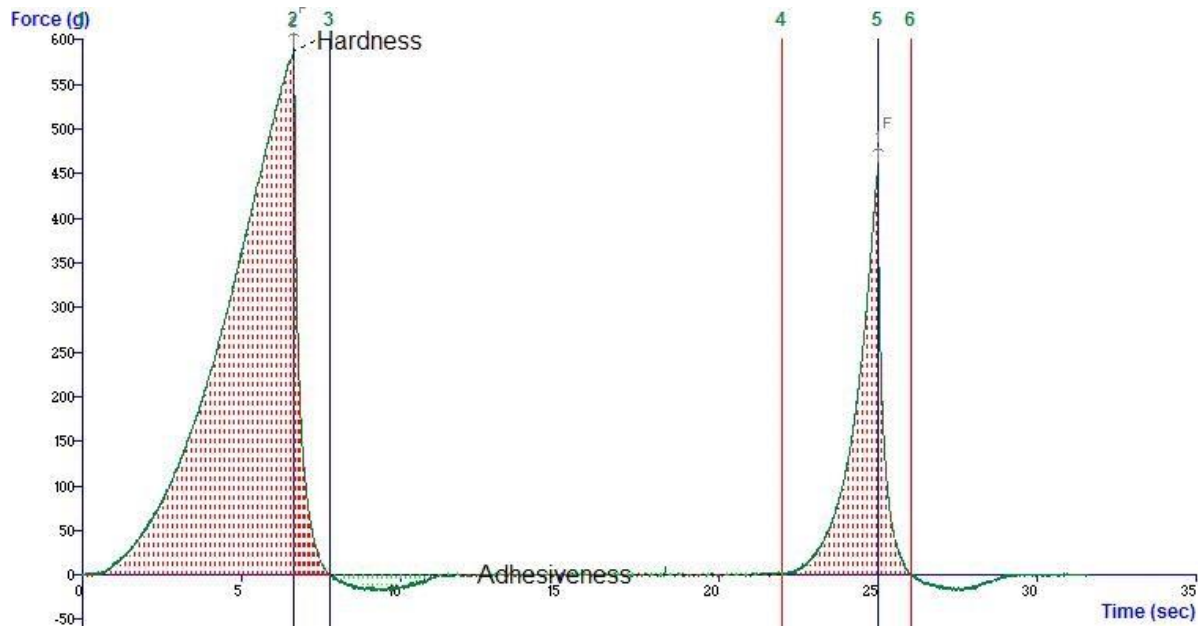


Figure 3.3: Typical force–time curve of fresh (thawed) mango cube from a TPA test

The chewiness of the experimental samples followed the same trend as hardness, as shown in Figure 3.4 (C) and (D). Since chewiness is the product of springiness and gumminess, a reduction in springiness will reduce the chewiness of the sample. A decrease in the chewiness was observed in the post-MWODS process in comparison with fresh (frozen thawed) mango samples as observed in other studies in which the sample texture became softer and more plastic due to reduced elasticity (Monsalve-Gonzalez, 1993). Decreased chewiness resulted from a reduction in springiness, which occurred due to loss of turgor—that is, reducing the cells' ability to regain their original form and/or size (Deng & Zhao, 2008). The sample of S:MD is showing higher chewiness than respective counter samples of S:D and S. The highest chewiness of 98.2 g mm was observed with S:MD-85:15 at 60°C/60% whereas, the lowest chewiness of 20.3 g mm was recorded with S:D-85:15 at 60°C/60%. These results also indicate that the presence of maltodextrin is reducing the loss of chewiness during MWODS process.

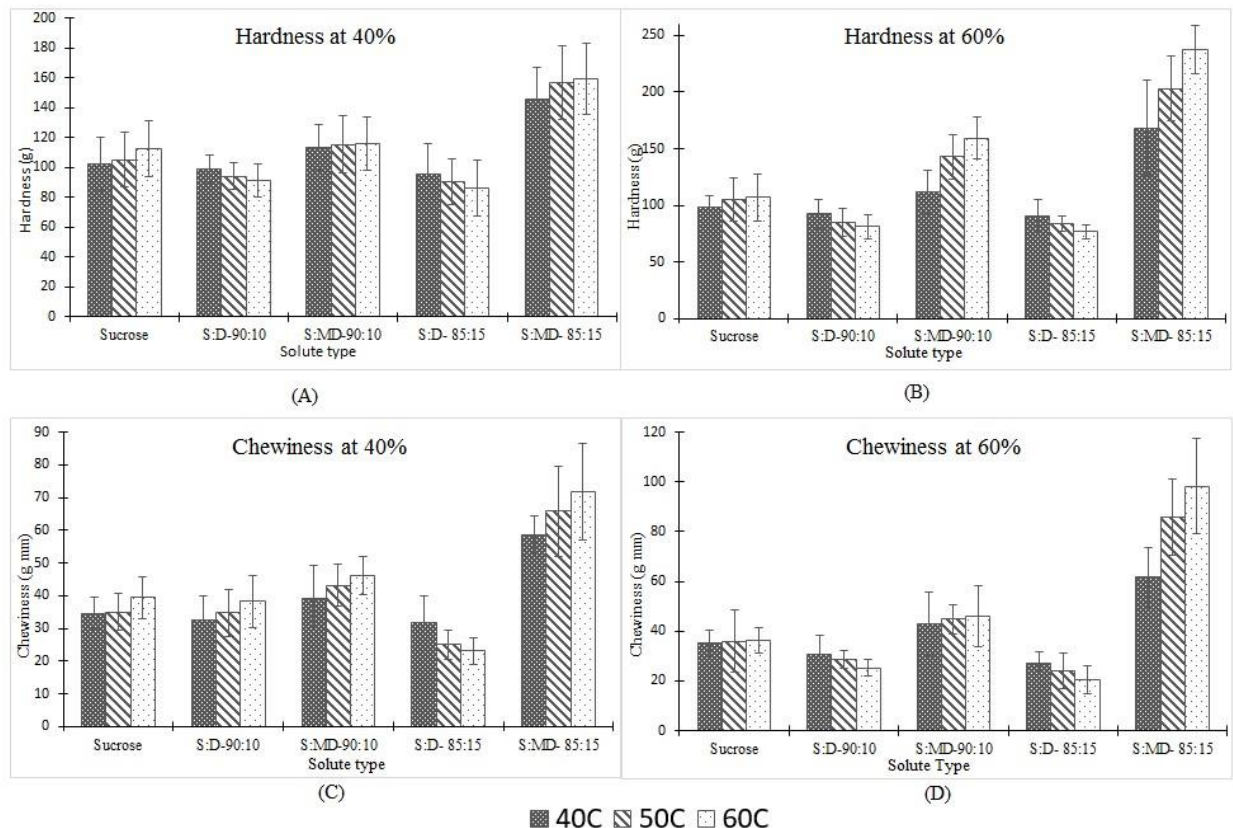


Figure 3.4: Graph for texture analysis (hardness and chewiness) of MWODS mango. Average values with standard deviation shown.

3.3.3.2 Color

The effect of MWODS process on the color (L^* , a^* , b^* , ΔE) of MWODS samples was not distinguished enough to differentiate it from fresh (frozen thawed) mango samples as shown in Figure 3.5. These results are in general agreement with the previous studies of MWODS (Wray & Ramaswamy, 2013). It is possible that the changes in the color of the post-MWODS samples were not noticeable due to minor destruction of the pigments. Since the MWODS process is faster than conventional OD and the color loss would be minimum, as a result there was not a noticeable difference in the color change from frozen thawed mango cubes to MWODS processed mangoes. Deng and Zhao (2008) reported that L^* value could increase due to increased solids gain and loss of pigments during the process (Deng & Zhao, 2008).

The value a^* represents the color change from green to red. Therefore, increasing a^* value has been used as an indicator of fruit browning, and a higher a^* value shows that the samples are more red. The a^* values of post-osmosed samples were slightly decreased as shown in Figure 3.5 (C) and (D), which indicates that there is no formation of browning due to the interaction between sugar, water, and heat under the microwave. The b^* value had a synergistic effect due to the sucrose concentration and temperature (Deng & Zhao, 2008). The post-osmosed samples showed reduced b^* value as compared to fresh (thawed) mango as shown in Figure 3.5 (E) and (F), which means the samples were losing their yellowness and becoming light yellow due to loss of color pigments during moisture transfer phenomenon.

The ΔE values will summarize the overall changes in the color of the samples in comparison with fresh thawed mango samples. S (100:0) showed the highest ΔE value of 22.9 units at 60%/60⁰C and lowest value of 9.06 unit at 40%/60⁰C in S:MD-85:15, suggesting that the binary osmotic solutes such as S:MD had a protective effect on the color of the sample. This is in line with the fact that the presence of maltodextrin leached out monomers, which is a reactive component for non-enzymatic browning present in plant tissues (Tabtiang et al., 2012). It was also concluded in previous studies that the dehydration of blueberries using maltodextrin enhanced palatability, good color and preferable texture (Chun et al., 2012). The research findings were also concluded that osmotically dehydrated tomatoes using maltodextrin and oligofructose solute mixtures were showing desirable sensory characteristics such as attractive appearance, bright color and good texture (Dermesonlouoglou et al., 2016).

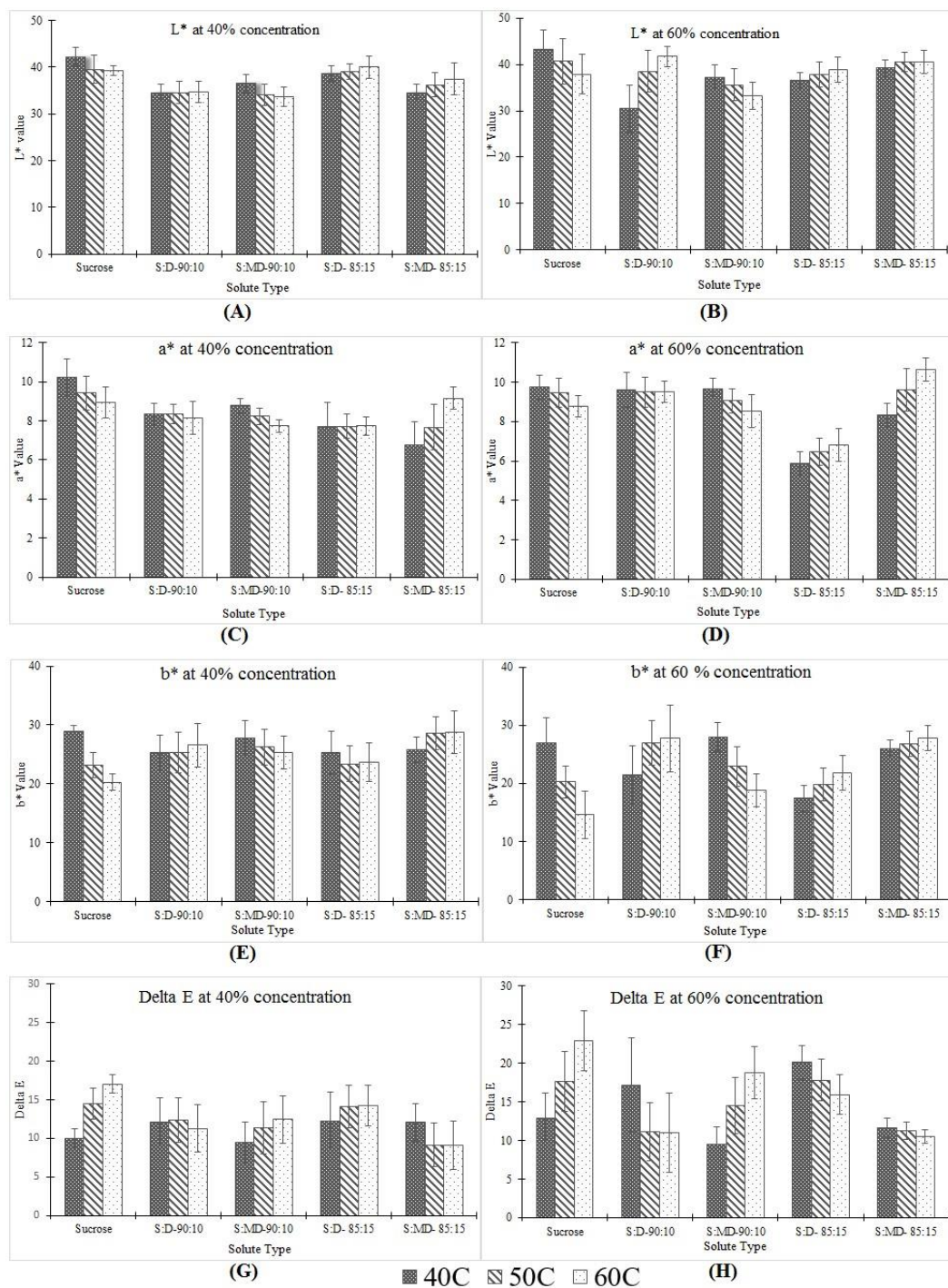


Figure 3.5: Graph for color analysis (L^* , a^* , b^* , ΔE) of MWODS mango. Average values with standard deviations)

3.4 Conclusions

A reduced solid gain in the presence of maltodextrin solute during osmotic dehydration was clearly observed in this study using the MWODS method for mango cubes. Overall, temperature and concentration had major effects on ML, SG, ML/SG and WR during MWODS process. Higher concentration and temperature yielded higher ML, which is considered the primary response to determining dehydration process. It has been demonstrated that the application of microwave energy along with the osmotic solutes such as S:D, S:MD, and S can achieve improved ML/SG ratio over the conventional OD. Additionally, maltodextrin content in osmotic agent improved the efficiency of the process by improving ML/SG ratio in comparison with other solutes mixtures. The qualitative analysis showed minor destruction of the pigments along with reduced hardness and chewiness, which can be considered as an advantage for the dehydrated product. The qualitative loss was minimal despite the presence of microwave energy.

Overall, it was found that MWODS process can be implemented successfully by using more than one osmotic solute. Nonetheless, a study on optimizing the MWODS process using two solutes should be conducted to develop a model for MWODS using binary solutes to explore the most efficient way to minimize the quality losses and maximize the benefits.

PREFACE TO CHAPTER 4

The comparative study of low, medium to low and high molecular weight osmotic solute mixtures were highlighted in Chapter 3 with the conclusion that the complex mixture of high molecular weight solute with commonly used sucrose solute was superior in performance. Therefore, further study was to understand the applicability of the wide range of high molecular weight solute mixed such as maltodextrin 10DE, 15DE and 18DE, each with sucrose on MWODS of mangoes and to focus on the modeling of mass transfer kinetics along with Azuara model evaluation. This objective was achieved to demonstrate if the Azuara model can be applied to two solute mixtures under MWODS treatment effectively as with MWODS using single sucrose solution.

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

Shinde, B. and Ramaswamy, H.S. 2019. Kinetic modeling of sucrose and maltodextrin (10-18 DE) moderated mass transfer rates in mango cubes during microwave osmotic dehydration under continuous medium spray conditions. Submitted to Drying Technology Journal. (*Paper in review*)

Shinde, B. and Ramaswamy, H.S., 2018. Microwave osmotic dehydration of mango cubes: Influence of osmotic solute mixtures. American society of agricultural and biological engineering, (ASABE) 2018. July 29 to August 01, 2018 Detroit, Michigan, USA. (Paper presentation).

CHAPTER 4

Kinetic modeling of sucrose and maltodextrin (10-18 DE) moderated mass transfer rates in mango cubes during microwave osmotic dehydration under continuous medium spray conditions

Abstract

Microwave osmotic dehydration of mango (*Mangifera indica*) was carried out under continuous spray mode with four osmotic solute mixtures (sucrose, sucrose in combination with maltodextrin 10DE, 15DE, and 18DE), at two temperatures (40⁰C, 60⁰C) and two concentrations (40%,60%) and four contact times (10, 20, 30, 40 min). The mass transfer kinetics were fitted to a first-order kinetic model, by way of an empirical parameter (k) representing an overall mass transfer coefficient. Azuara model was also evaluated to describe the mass transfer kinetics. The results showed that the overall mass transfer coefficients for moisture and solids contents were influenced by temperature, concentration and, more importantly, the solute composition. The highest mass transfer coefficient for moisture loss ($1.56E^{-02}$) and lowest for solids ($0.86E^{-02}$) observed with sucrose + maltodextrin 10DE (S+MD 10DE) combination at 60%/60C and 40%/40C, conditions, respectively. In addition, it was found that the Azuara model well fitted the experimental data for mass transfer kinetics ($R^2 > 0.92$). The highest moisture loss (ML) along with lower solids gain (SG) were associated with the solute sucrose + maltodextrin 10DE combination. The highest ML/SG ratio of 10 and weight reduction of 55.5% were observed with S+MD 10DE at 40⁰C/60%/40min and 60⁰C/60%/40min, respectively, with S+MD 10DE solute mixture. Overall, it was possible to successfully describe the mass transfer kinetics using empirical first order and Azuara models and MWODS with S+MD 10DE facilitated the maximum moisture loss and low solids uptake, both desirable from an osmotic dehydration point of view.

4.1 Introduction

Mango is the second most heavily produced tropical fruit which is less commercialized due to lack of processing resources in harvesting countries (Mitcham & McDonald, 1992). It is one of the most popular fruits in the world and it also contains a good source of nutrients such as ascorbic acid. Dehydration is considered as one of the oldest methods to preserve fruits and osmotic dehydration has shown to result in improved sensory quality, to reduce nutrient loss as well as conserve energy (Mandala et al., 2005; Riva et al., 2005). Osmotic dehydration (OD) is generally considered as a pre-treatment step before finish drying process because it cannot reduce the moisture content to the final desired level. During the OD process, two primary countercurrent mass transfer flows take place: water flow from the product to the solution and solute migration from the solution to the product (Dermesonlouoglou et al., 2007). The rate of diffusion of water from food during OD depends on many factors such as temperature and concentration of the osmotic solution, type of osmotic agents, the size and geometry of the food material, and the ratio of fruit to the solution (Azoubel & Murr, 2004; Torreggiani, 1993). Amongst these, the osmotic agent is one of the key factors and various osmotic agents such as sucrose, glucose, fructose, corn syrup, sodium chloride, sorbitol, etc., and their combinations have been evaluated for osmotic dehydration process (İspir & Toğrul, 2009b). Some researchers have used more than one osmotic agent and reported an improvement in water reduction along with limited solids uptake (Medina-Vivanco et al., 2002; Qi et al., 1998). Among such chemicals used for reducing solids uptake, maltodextrins appear to have been more widely used. Maltodextrins are polymers of dextrose, made from acid or enzyme hydrolysis of natural corn starch (Wang & Sastry, 2000). There are different grades of maltodextrins based on their dextrose equivalents (DE) values (Fitton, 1979). DE is a quantitative measure of the degree of starch polymer hydrolysis. The DE of maltodextrins vary from 3 to 20. The higher the DE, the greater the extent of starch hydrolysis.

Osmotic drying is an inherently slow process and traditionally involves a long time. Developing new techniques to improve OD has stimulated a lot of interest. Techniques that have been exploited for accelerating OD include: pulsed electrical field (Andrés et al., 2007), pulsed vacuum (Ito et al., 2007), ultrasound (Rodrigues & Fernandes, 2007), high pressure (Rastogi & Niranjan, 1998), and microwave (MW) (Krokida et al., 2000) with or without other

pretreatments. Li and Ramaswamy (Li & Ramaswamy, 2006b) investigated a novel process of microwave osmotic dehydration under immersion (MWODI) mode which was modified by Azarpazhooh and Ramaswamy (Azarpazhooh & Ramaswamy, 2012a) for operation under continuous medium flow in spray mode (MWODS). These two techniques involve carrying OD within a MW environment while all other MW OD works either using MW treatment before or after OD. These methods are aimed at enhancing the one-way moisture loss and restricting the solids gain. MW generates heat by exciting dipolar molecules (water molecules) and polarizing (ionic salts) components which ultimately leads to forcing out water from the food product (Sosa-Morales et al., 2010). Li and Ramaswamy (2006) investigated the mass transport coefficients under microwave osmotic dehydration (MWODI, immersion medium) and reported that osmotic dehydration under microwave heating has made it possible to obtain a higher diffusion rate of moisture transfer at lower solution temperatures (Akyol et al., 2006). Azarpazhooh and Ramaswamy studied diffusion model for MWODS (spray condition) and concluded that even higher moisture loss and lower solids gain could be obtained in MWODS method when compared with other osmotic dehydration methods (Azarpazhooh & Ramaswamy, 2009a). In a recent article (Shinde & Ramaswamy, 2019a), the mass transfer improvement and quality of microwave-osmotic dehydrated mango cubes under MWODS conditions in sucrose solution with added dextrose or maltodextrin (10DE) supplements was evaluated. This study demonstrated that maltodextrin supplement to sucrose syrup has the potential to further enhance moisture transfer potential and limit solids gain at the same time, much superior to the trends reported earlier with MWODS systems. In the above study, only a limited range of osmotic parameters and only one grade of maltodextrin (10DE) with one treatment time were used in order to provide a proof of concept.

Therefore, the focus of this study was to expand the scope of this previous research and evaluate and model the influence of MWODS on mass transfer kinetics in mango cubes involving a broader range of processing conditions: sucrose with three different grades of maltodextrins (10, 15 and 18 DE) supplements with two solute concentration (40% and 60%), two temperatures (40°C and 60°C) and four contact times (10-40 min). As with most other OD drying studies, the various output mass transfer parameters were ML, SG, WL and ML/SG ratio.

4.2 Equations and theory

A diffusion model based on Fick's second law is typically used to model mass transfer during osmotic dehydration process, which assumes that the external resistance to mass transfer is negligible as compared to the internal resistance (Crank, 1975). The unsteady state form of Fick's second law is based on a few considerations such as short processing time, constant solution concentration and negligible external resistance to mass transfer. Also, the application of Fick's second law during mass transfer modeling of osmotic dehydration process is suggested due to a decrease in water loss rate after short processing time. Crank (1975) solved Fick's equation, in a semi-infinite approach by concluding that the mass of diffusing substance varies linearly with the square root of time when the surface concentration is constant (Crank, 1975). No such studies have been carried out on evaluating the overall mass transfer coefficients in MWODS process using a combination of solute mixtures. Hawkes and Flink (1978) proposed a model to determine overall mass transfer (k) - an empirical parameter that can represent the water and solutes mass transfer coefficients:

$$NMC = X/X_0 \quad (4.1)$$

$$NSC = S/S_0 \quad (4.2)$$

These are then related to time as in Eqs. 4.3 and 4.4

$$NMC = 1 - k_w t^{0.5} \quad (4.3)$$

$$NSC = 1 + k_s t^{0.5} \quad (4.4)$$

where X, X_0 represents moisture content, S, S_0 solids content in the sample at time t and 0, respectively and k_w and k_s represent overall mass transfer coefficients for water and solute, respectively.

In addition, to predict the kinetics of moisture loss and solids gain, Azuara (1992) developed an empirical model based on the mass balance of water and sugar during osmotic dehydration process (Azuara et al., 1992). Ochoa-Martinez recommended that Azuara model should be favored to Page's, Magee's, and Crank's models to predict mass transfer in osmotic dehydration of fruits at atmospheric pressure since this model accurately predicts the mass transfer dynamics of osmotic dehydration and the dynamic period solids gain kinetics (Ochoa-Martinez et al., 2007b). The advantage of using Azuara's model is, it allows better calculation of

the equilibrium values of moisture loss and solids gain (ML_e and SG_e). The proposed model for moisture loss and solids gain is shown by Eq. 4.5:

$$ML_t = \frac{S_1 t (ML_e)}{1 + S_1 t} = \frac{t (ML_e)}{\frac{1}{S_1} + t} \quad (4.5)$$

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

$$SG_t = \frac{S_2 t (SG_e)}{1 + S_2 t} = \frac{t (SG_e)}{\frac{1}{S_2} + t} \quad (4.6)$$

where SG_t is the solids gain fraction at any time, t ; S_2 is a constant related to the rate of solids diffusion in the product; and SG_e is the solids gain fraction at equilibrium.

The weight reduction and ML/SG ratio were used to link the effectiveness of solute mixtures on dehydration of mango pieces. Therefore, the following equations were taken into consideration for ML, SG, ML/SG and WR:

$$\text{Moisture Loss (ML)\%} = 100 \frac{M_0 X_0 - M_t X_t}{M_0} \quad (4.7)$$

$$\text{Solids Gain (SG)\%} = 100 \frac{M_t S_t - M_0 S_0}{M_0} \quad (4.8)$$

$$\text{ML: SG ratio} = \frac{ML}{SG} \quad (4.9)$$

$$\text{Weight Reduction (WR)\%} = 100 \frac{M_0 - M_t}{M_0} \quad (4.10)$$

where M_0 and M_t are the total mass of the fruit sample at time 0 and time t , respectively; X_0 and X_t are the moisture fractions (kg/kg, wet basis) at time 0 and time t , respectively; S_0 and S_t are the solid fractions (kg/kg, wet basis) at time 0 and time t , respectively.

The above Eqs. (4.7-4.10) were used assuming a uniform mass transfer of solids into the product and considering that there is no significant loss of solids from the sample into the solution.

4.3 Materials and Methods

4.3.1 Raw material

Commercial grade sucrose (Lantic Sugar Ltd., Montreal, Qc, Canada), and three different grades of maltodextrins (10DE, 15DE and 18DE) (Univar Pvt. Ltd, Canada) were used in this

study for preparing the osmotic solutions, and the concentrations were maintained on a wet basis (wb). The numbers associated with MD refer to their dextrose equivalents, for example, the dextrose equivalent of MD 10DE is 10%. Lower numbers indicate that they are less hydrolyzed and have higher molecular weights. Frozen mango pieces were obtained from a local food freezing company (Nature's Touch, Canada) and kept frozen (-21°C to -27°C) until use. Prior to use, the mango pieces were thawed overnight (8-10 h) in a refrigerator (4°C - 7°C). The dimensions of the mango pieces were in the general shape of a cube with the side dimensions of approximately 15 ± 0.2 mm. The moisture content of the frozen-thawed (untreated) mango cube was measured using AOAC method, by keeping the cubes in an oven at 105°C for approximately 24h (until a constant weight was achieved). The average moisture content of mango cube was determined as 86.1% (wb).

4.3.2 Microwave Setup

The MWODS assembly consisted of a domestic microwave (Danby DMW1153BL 0.031 m³, Guelph, ON, Canada) with a nominal power output of 1100W and 2450MHz, which contained a spray head (Waterpik, Model RPB-173C, 12.5cm diameter, Waterpik Technology Inc., Markham, ON, Canada) attached to the custom-made glass sample chamber (12.5cm diameter). The schematic of MWODS assembly is shown in Figure 4.1. The test samples were placed in a Nylon mesh bag on the porous acrylic plate "stage" inside the glass sample chamber. This allowed to drain down the sprayed osmotic solution, while keeping the samples directly in contact with the microwave. The osmotic solution collected at the bottom of the sample chamber was recycled through a long coil heat exchanger immersed inside a heated water bath (Model TDB/4 Groen Division, Dover Corp, IL) and then pumped through the spray head, using a peristaltic pump (Model 75211-30 Digital gear pump, Barnant company IN). The temperature of the water bath was set to the inlet temperature of the osmotic solution, and the osmotic solution was then circulated through the assembly to equilibrate the set-up temperature before putting the samples into the system.

The temperature of the osmotic solution was monitored using a pair of in-line Type-T thermocouples connected to the digital thermometer (Omega DP-462, Omega technology, Laval, QC). The thermocouples were placed immediately before and after the microwave cavity to measure the temperature of the solution before going inside and immediately after coming out of

the microwave oven. The increase in the temperature of the osmotic solution after residing in microwave cavity was about 4-5⁰C, which was cooled as they passed through the long coils heat exchanger placed in the water bath to return to the original initial temperature before coming into the microwave. The length of the coils was long enough to allow the osmotic solution in the solution to sample ratio of 30:1 to pass through. The large amount of osmotic solution in a closed system also allowed to maintain a relatively constant solute concentration throughout the experiments for each solute type, which was measured using a handheld refractometer.

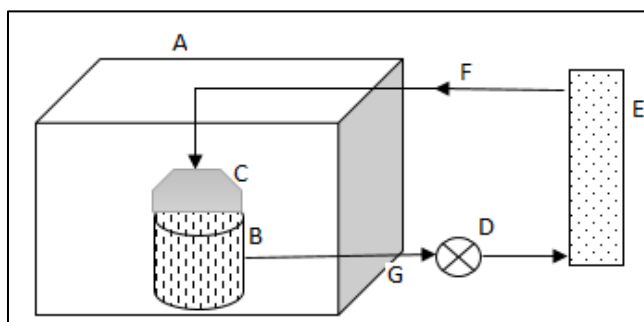


Figure 4.1: Schematic diagram of MWODS setup (A: microwave oven cavity, B: microwave transparent sample chamber, C: spray head, D: digital gear pump, E: water bath (containing heat exchanging coils, not pictured), and F and G are thermocouple measuring points immediately before and after the solution enters and leaves the microwave cavity, respectively.)

4.3.3 Osmotic dehydration procedure

The MWODS system was setup and solution were preheated according to the prescribed temperature of the run type. Individual samples of approximately 100g (12-15 cubes) were weighed and placed in a Nylon mesh bag to hold the samples. The bag was then placed on the acrylic stage in the sample chamber in a single layer. The pump was turned on and the solution allowed to flow, and then the microwave was turned on. The pump was then terminated after each selected contact time (10, 20, 30 and 40 min) according to the experimental run type. The excess osmotic solution from the surface of the product was removed by shaking the sample 3-4 time and wiping with a wet paper towel, and the mango cubes were then weighed and were either examined for quality parameters or dried to constant weight in an oven set at 105⁰C for approximately 24 h (AOAC, 1975).

4.3.4 Experimental design

Experimental design for MWODS involved using two different sucrose solution alone and those with sucrose and maltodextrin mixtures at 85 % sucrose and 15% maltodextrin of a specific grade. So there four different solutions: sucrose only, sucrose + maltodextrin 10 DE (S+MD10DE), sucrose + maltodextrin 15DE (S+MD15DE), sucrose + maltodextrin 18 DE (S+MD18DE). Each osmotic solution was used with the following process variables: solute concentration (40 and 60%), temperature (40 and 60°C) and contact time (10-40 min) while keeping flow rate (1050 mL/min) at a constant level.

4.3.5 Data analysis

Experiments were carried out in four replicates and mean values with standard deviations are presented in graphs. The analysis of variance was performed using JMP® v-13 (SAS Institute Inc., Cary, NC., U.S.A) to compare the results at 95% confidence level. The paired t-test was performed to understand the significant difference between the overall performance of each solute mixtures based on mass transfer and quality parameters.

4.4 Results and discussions

4.4.1 Osmotic dehydration kinetics

The effect of osmotic solute type, solution temperature, solute concentration and contact time on residual normalized moisture content and solids content are presented in Figure 4.2 (a to h). In general, it was found that contact time, temperature and solute concentration of the osmotic solution had a major effect on the MWODS kinetics of mango. These were all expected as they have also been observed in several earlier studies with other products (Lenart, 1984; Sereno et al., 2001). The primary focus of the study was on the influence of maltodextrins on the osmotic dehydration kinetics when it was carried out under MWODS conditions. It was clearly observed that solute mixtures containing maltodextrins had a big influence on changes in moisture loss and solids gain as indicated by the NMC and NSG curves. In general, with each sucrose - maltodextrin combination, there was a progressive decrease in the moisture content of samples along with solids gain with time at each solute concentration.

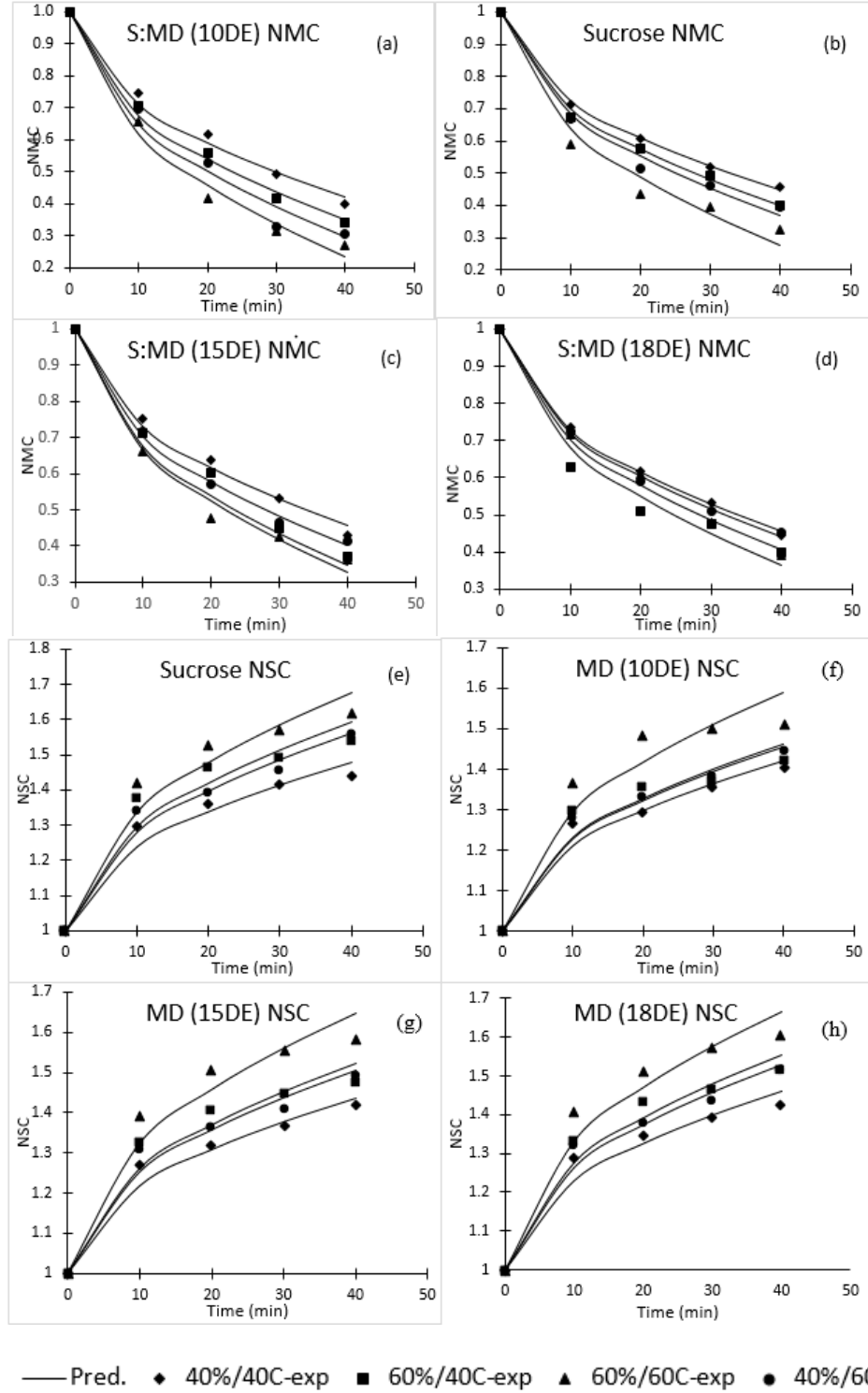


Figure 4.2: Osmotic dehydration kinetics: Performance of Empirical model (predicted vs. experimental) for normalized moisture content (NMC) and normalized solids content (NSC) at different temperature and concentrations with respect to contact time.

Further, when the different mixtures were compared, the moisture content in MWODS dried samples with S+MD 10DE were significantly lower as compared to S, S+MD 15DE and S+MD 18DE (Figure 4.2 (a-d)). Although desirable, this meant that S+MD 10DE favored a better ML than other solutes which was not entirely expected since lower molecular sucrose which provides the highest osmotic potential should have helped to increase the moisture loss. Perhaps this could be an effect that can be related to MW absorption and temperature rise in the solution and fruit pieces. In MD containing solutions, the non-dielectric composition could be expected to have some influence on the heating behavior since the moisture content is constant for each solute concentration. Further, since there is less solute infusion (as will be detailed in the next paragraph) into the samples, there would remain higher transient moisture content in the sample which would attract more MW energy due to better dielectric property. This would provide a mechanism for increased out-flux of moisture and hence compensate the osmotic benefit alone of sucrose solution. The lowest NMC of 0.271 was observed with 60%/60⁰C (concentration/temperature) processing condition. This phenomenon is in line with the findings of some previous research as well (Azuara et al., 2002). This trend with residual moisture content, while comparing the solute types, was more prominent at longer contact times (30 min and 40 min) and higher concentration: S+MD 18DE>S+MD 15DE>S>S+MD 10DE.

Figure 4.2 also shows the normalized solids content at different concentrations, temperatures and contact times of the different solute combination mixtures. The solids gain increased at elevated temperature and contact time as shown in Figure 4.2 (e-h). As expected, the highest NSC of 1.62 was found with sucrose at 60%/60⁰C and the value of 1.26 was found with maltodextrin S+MD 10DE (40%/40⁰C). As mentioned earlier, MD 10DE has a higher molecular weight and higher viscosity than other solutes (MD 15DE, MD18 DE, and sucrose) which when it forms a surface layer, will have a greater barrier effect for solute impregnation. This was the main hypothesis for the MD inclusion in the sucrose solution. The low molecular solutes tend to penetrate easily into the fruit samples and increase the solids intake, whereas high molecular solutes always face barriers to pass through the fruit tissue to impregnate the fruit. This phenomenon has been demonstrated in previous studies with other fruit types (Contreras & Smyrl, 1981) but without MW heating. The trend for solids uptake, while comparing the solute type was found as S+MD 10DE < S+MD 15DE < S+MD 18DE < sucrose.

4.4.2 Overall mass transfer coefficients

The typical solute mixture effect on the mass transfer coefficients is presented in Table 4.1 at different processing conditions. The mass transfer coefficients were obtained as the slopes of NMC vs. $t^{0.5}$ and NSC vs. $t^{0.5}$ plots, for each MWODS processing condition. It is arranged in the order of solute mixture type. Within each solute type, the maximum influence on both k_w and k_s were observed 60%/60°C followed by 60%/40°C 40%/60°C 40%/40°C indicating the concentration to be the dominant factor followed by temperature. This phenomenon can be related to the increase in membrane permeability at higher concentration and temperatures, favoring better osmotic potential, plasticization and swelling of the cell membrane which promotes mass transfer (Serenio et al., 2001; Tonon et al., 2007). In addition, increasing temperatures also cause a reduction in solution viscosity, reducing external resistance to mass transfer and making water and solutes transport easier (Tonon et al., 2007).

Again, as indicated earlier, the primary focus is a comparison of the different solute mixtures with MD incorporations. This can be compared analyzed by the values of k_w for different solutes at each level of concentration-temperature combinations in Table 4.1. Again, the highest overall water transfer coefficient was associated with S+MD 10DE which was highest with the 60%/60°C combination. The reason for such an influence has already been discussed in the previous section. However, the sucrose solution was found to have k_w lower than MD mixture. Since the sucrose solution will naturally increase the moisture transfer out of the fruit, but at the same time being a low molecular weight solute, sucrose can also impregnate into the fruit more and thus making it difficult for water to come out as compared with other solute mixtures (Azuara et al., 2002; Hawkes & Flink, 1978). Since other solute mixtures are the combination of low molecular sucrose along with high molecular weight maltodextrins (10DE, 15DE, and 18DE), which can form a dense layer on the surface of fruits to prevent solids gain and ultimately increase the osmotic potential and the water transfer coefficient (Azuara et al., 2002).

The type of solute mixture also affected the solute transport, as observed with the overall solute transfer coefficient, k_s . It was clear from Table 4.1 that the overall solids transfer coefficient of sucrose was highest in comparison with others and then decreased with a decrease in molecular size of osmotic solute. The maximum k_s value was found with sucrose that is

0.0138 at 60°C/60%. Once again, this is due to the low molecular weight solute mixture which makes it easier to incorporate into the fruit pieces and hence it will increase the k_s value. The solute mixture with S+MD 10DE gave lower k_s , among all processing conditions. This behavior can also be explained by the formation of a solid barrier at the fruit surface, which can make solids mass transfer more difficult (Dermesonlouoglou et al., 2016; Hawkes & Flink, 1978)

Table 4.1: Overall water (k_w) and solids (k_s) transfer coefficients during osmotic dehydration of mangoes at different conditions using various solute mixtures

Solute mixtures	Processing conditions	k_w	R^2	k_s	R^2
S:MD 10DE	40%/40°C	0.0118	0.9648	0.0086	0.9649
	60%/40°C	0.0133	0.9761	0.0093	0.9294
	60%/60°C	0.0156	0.9399	0.0120	0.9198
	40%/60°C	0.0144	0.9294	0.0094	0.9731
S:MD 15DE	40%/40°C	0.0111	0.9694	0.0089	0.9700
	60%/40°C	0.0125	0.9684	0.0107	0.9492
	60%/60°C	0.0137	0.9262	0.0132	0.9499
	40%/60°C	0.0122	0.9866	0.0103	0.9684
S:MD 18DE	40%/40°C	0.0111	0.9949	0.0094	0.9548
	60%/40°C	0.0121	0.9787	0.0113	0.9625
	60%/60°C	0.0130	0.7698	0.0136	0.9525
	40%/60°C	0.0114	0.9922	0.0108	0.9753
S	40%/40°C	0.0113	0.9938	0.0098	0.9564
	60%/40°C	0.0123	0.9743	0.0121	0.9412
	60%/60°C	0.0148	0.7748	0.0138	0.9490
	40%/60°C	0.0129	0.9354	0.0115	0.9753

The analysis of variance results using paired t-test are summarized in Table 4.2, which was used to determine the significant difference between solute mixtures with respect to k_w and k_s . Data in Table 4.2 confirms the detailed discussion on the influence of solute composition on MWODS. Further, the empirical model used for estimating the overall mass transfer coefficients

can also be used to relate the moisture and solids content to the process variables. In Figure 4.2, while the points represent the experimental values, the lines represent the predicted values from the empirical modified first order model. The fit appears to be excellent and the R^2 values were higher than 0.9 in all except two situations.

Table 4.2: t-test results for significance of differences in k_w and k_s , between different solute mixtures

Solute mixtures	k_w		k_s	
	Pr> t	t-value	Pr> t	t-value
S:MD 10DE - Sucrose	< 0.05	4.52	< 0.01	-5.94
Sucrose - S:MD 15DE	> 0.05	1.58	< 0.01	5.86
Sucrose - S:MD 18DE	> 0.05	2.18	< 0.05	3.81
S:MD 10DE - S:MD 15DE	< 0.05	3.68	< 0.05	-3.96
S:MD 10DE - S:MD 18DE	< 0.05	3.41	< 0.05	-5.8
S:MD 15DE - S:MD 18DE	> 0.05	2.64	< 0.01	-12.2

(Note: values in bold are not significant)

4.4.3 Equilibrium ML and SG for using Azuara model

The equilibrium moisture loss and solids gain were obtained by fitting the ML vs. t and SG vs. t data to the Azuara model. The equilibrium values were obtained as the reciprocal slopes of t/ML vs. t and t/SG vs. t plots as shown in Figure 4.3 (a to h), for each MWODS condition and the intercepts were used to compute the second parameter. The associated high R^2 value (> 0.92) indicated the acceptability of the model and the computed equilibrium values. Table 4.3 shows the values of parameters S_1 , ML_e , S_2 , and SG_e . Table 4.3 also compares the equilibrium values of moisture loss and solids gain under different types of osmotic solutes, osmotic solution concentrations and temperatures. Several previous studies have elaborated the usefulness in predicting the equilibrium values of moisture loss and solids gain and published an excellent result for the Azuara model (Azarpazhooh & Ramaswamy, 2009a; Ochoa-Martinez et al., 2007a; Waliszewski et al., 2002).

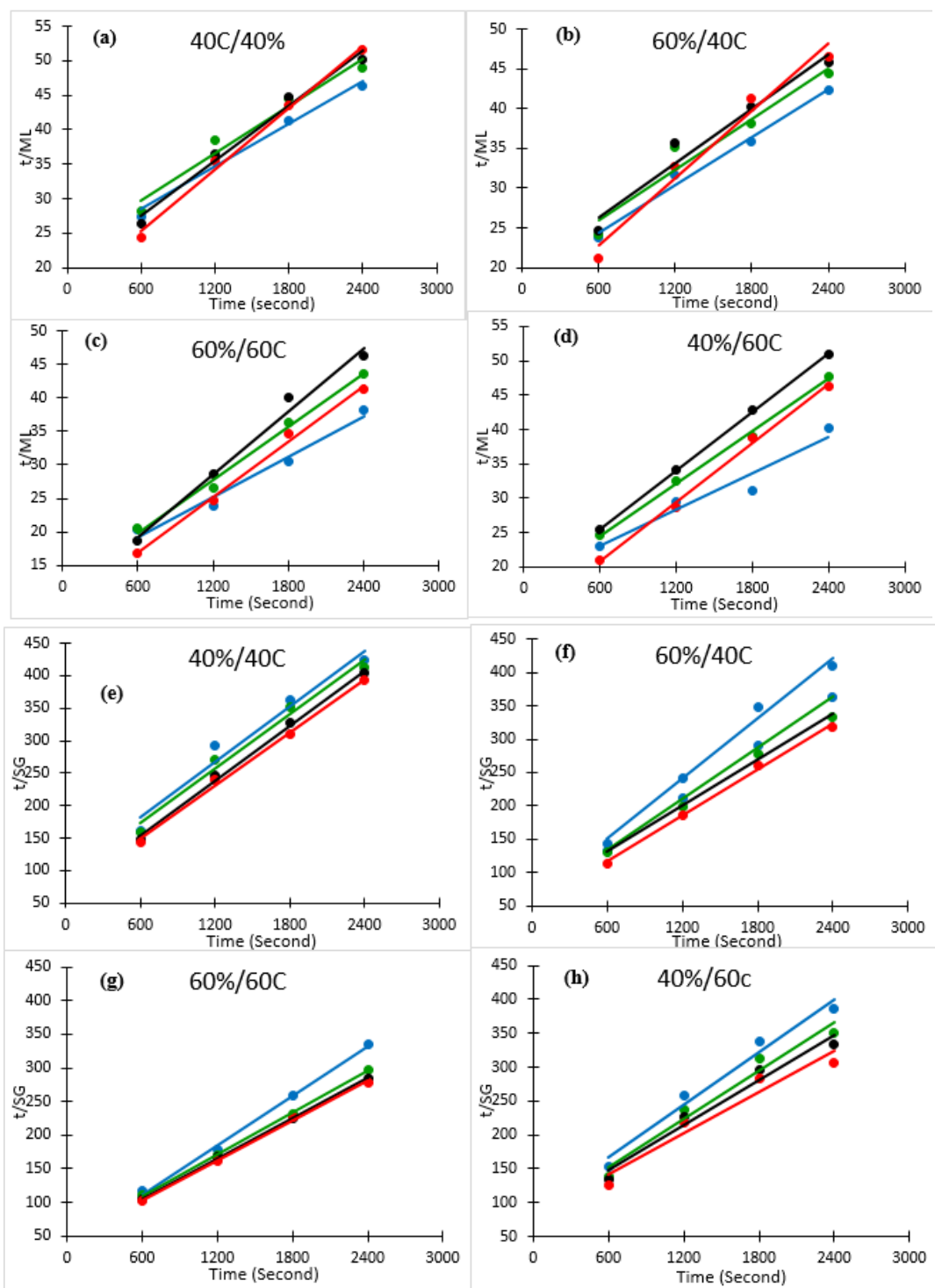


Figure 4.3: Linear plots of the Azuara model for determination of MLe and SGe for different solutes mixture at various temperatures and concentrations.

The equilibrium values were dependent on not only the osmotic solution temperature and concentration but also on the type of osmotic solute or solute mixtures applied in osmotic dehydration process because the solute type has varying potential for achieving the equilibration. The computed ML_e in some situations reached 100% in some situations which may be theoretical incorrect, but probably caused by experimental variability with the limited data points used in the study.

Table 4.3: Azuara model parameters and equilibrium values (ML_e and SG_e) during osmotic dehydration of mangoes at different conditions using various solute mixtures

Solute mixtures	Processing conditions	%ML_e	$S_1 (X10^{-4})$ min^{-1}	R^2	%SG_e	$S_2 (X10^{-3})$ min^{-1}	R^2
S+MD	40 ⁰ C/40%	97.1	4.61	0.9756	7.00	1.49	0.9637
10DE	40 ⁰ C/60%	100	5.42	0.9868	6.64	2.51	0.9895
	60 ⁰ C/60%	99.0	7.73	0.9748	8.22	3.08	0.9970
	60 ⁰ C/40%	112	5.04	0.9361	7.71	1.46	0.9729
S+MD	40 ⁰ C/40%	87.7	4.97	0.9619	7.15	1.57	0.9813
15DE	40 ⁰ C/60%	94.3	5.43	0.9451	7.84	2.22	0.9996
	60 ⁰ C/60%	75.8	11.1	0.9929	9.67	2.22	0.9996
	60 ⁰ C/40%	79.4	7.44	0.9979	8.38	1.48	0.9635
S+MD	40 ⁰ C/40%	75.8	6.71	0.9830	7.08	2.03	0.9967
18DE	40 ⁰ C/60%	88.5	5.76	0.9563	8.70	1.85	0.9965
	60 ⁰ C/60%	63.7	15.9	0.9882	10.1	2.05	0.9995
	60 ⁰ C/40%	70.4	8.36	0.9996	9.00	1.38	0.9668
S	40 ⁰ C/40%	67.1	9.11	0.9937	7.36	1.99	0.9959
	40 ⁰ C/60%	70.9	9.84	0.972	8.76	2.35	0.9965
	60 ⁰ C/60%	71.9	16.4	0.9943	10.2	2.20	0.9982
	60 ⁰ C/40%	69.9	11.6	0.9969	9.81	1.27	0.9423

4.4.4 Azuara Model for ML and SG

The trend curves of each solute mixtures, for predicted moisture loss and solids gain based on the Azuara model with superimposed experimental values are shown in Figure 4.4 (a to h) under given processing conditions. The two-parameter Azuara model (Azuara et al., 1992) was used for computing the equilibrium values and describing the mass transfer in MWODS of mango using different osmotic solute mixtures (Azuara et al., 1992).

Figure 4.4 (a-d) indicates that ML was favored by longer contact time, higher solution concentration and higher temperature. A similar effect has been observed with the osmotic dehydration of melon and pear (Ferrari & Hubinger, 2008; Park et al., 2002). Figure 4.4 (e to h) shows similar trends for solids gain which has also demonstrated previous studies that with increase in contact time there will be increase in the solids gain (Azarpazhooh & Ramaswamy, 2009a). It was found that the ML was distinctively higher when mangoes were subjected to S+MD 10DE solute mixture at most of the processing conditions. The highest ML (62.7%) was obtained at 60%/60C/40min with S+MD 10DE solute mixture, which demonstrates the efficiency of the solute type in MWODS process. On the other hand, the opposite trend was obtained with SG indicating S+MD 10DE to yield the least SG when compared to other solutes at all processing conditions. The high SG was obtained with sucrose at 60°C /60%/40min about 8.9%. This confirms the earlier discussion that, higher molecular weight solutes such as maltodextrins will form a coating on the surface of the fruit pieces and restrict the intake of osmotic solids into the mango samples. Hawkes and Flink (Hawkes & Flink, 1978) also reported that solids uptake is inversely correlated with the molecule size of the osmotic agent.

The performance of the empirical model as well as Azuara model is presented in Figure 4.5 (a to d). In Figure 4.5 (c) and (d), all points were scattered evenly and tightly around the diagonal line for ML and SG, indicating an excellent model performance and a good predictor of the ML ($R^2 = 0.9837$) and SG ($R^2 = 0.9691$) for all four solute mixtures and under the different processing conditions. Similarly, in Figure 4.5 (a) and (b), all of the points are scattered evenly around the diagonal line for NMC ($R^2 = 0.9385$) and NSC ($R^2 = 0.8003$).

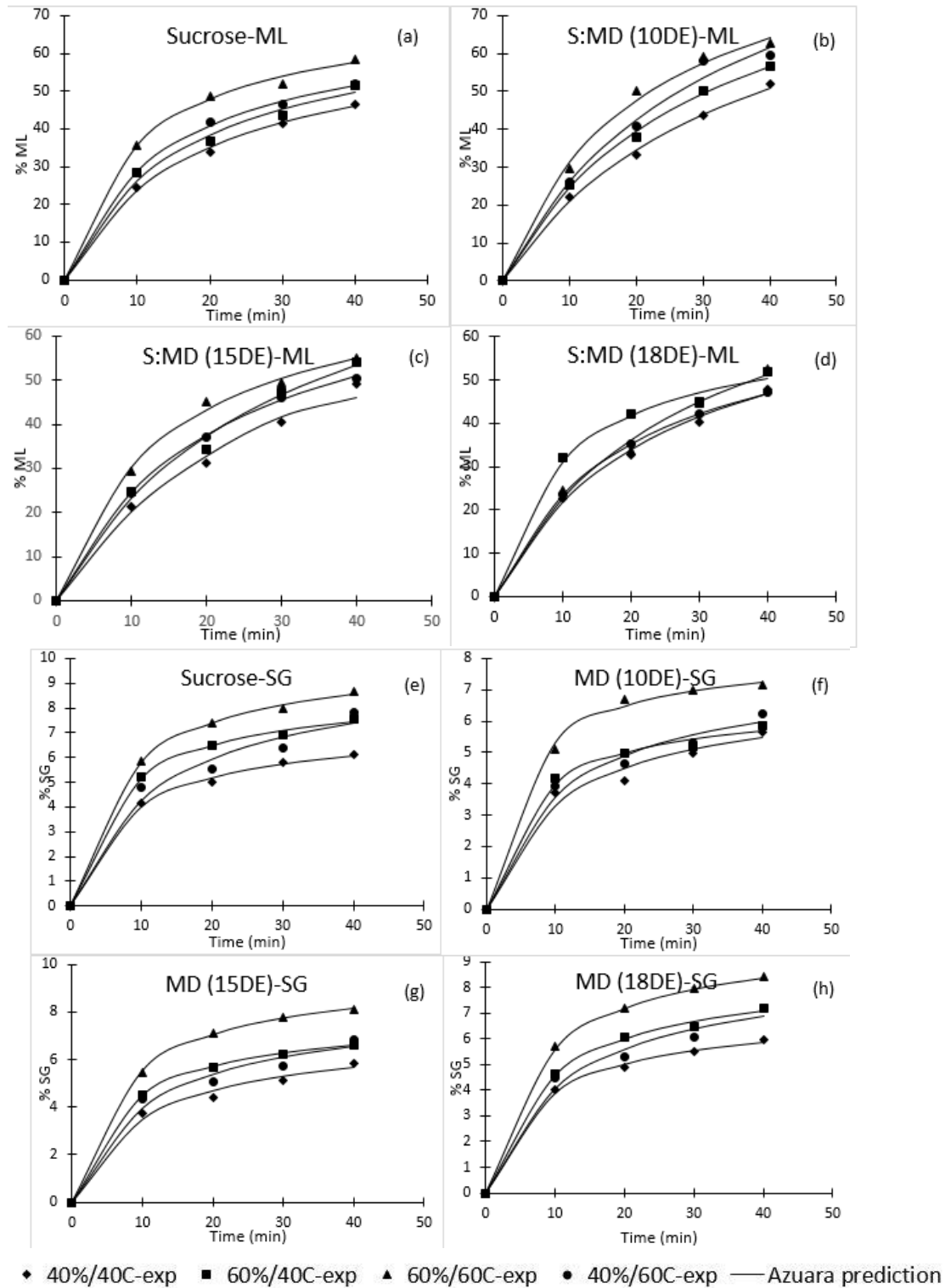


Figure 4.4: Performance of the Azuara model (predicted vs. experimental) for moisture loss (%ML) and solids gain (%) at different temperature and concentrations with different solute mixtures.

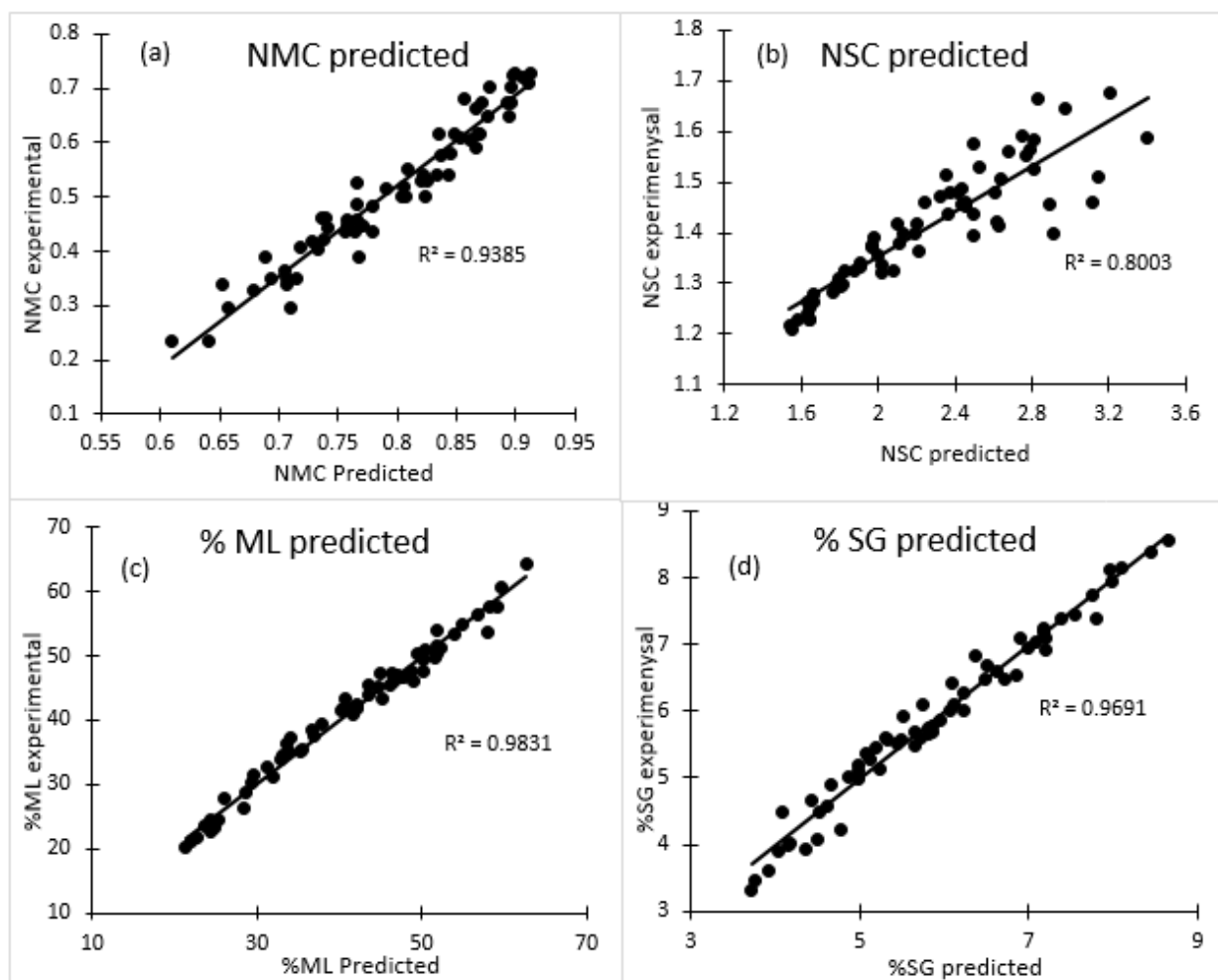


Figure 4.5: Performance of Empirical model (predicted vs. experimental) for (a) NMC and (b) NSC; and performance of Azuara model (predicted vs. experimental) for (c) moisture loss (%ML) and (d) solids gain (%SG)

4.4.5 ML/SG ratio

The ratio of moisture loss/solids gain (ML/SG) or water loss/sugar uptake (WL/SU), is known as the dehydration efficiency index (DEI) (Matuska et al., 2006). The particular interest in osmotic dehydration process lies in the ML/SG ratio parameter. In previous studies, the application of different solute mixtures was progressively showed to have better ML/SG potential when subjected to high molecular weight solute mixture such as sucrose and maltodextrin in MWODS treatment (Shinde & Ramaswamy, 2019a). Generally, it is desirable to promote ML in the OD process, to obtain enriched fruit solids in the product instead of gaining the solutes from an osmotic solution which is expressed by the SG parameter. Since ML was

more prominent than SG in all of the solute mixture application under MWDS condition, therefore the ratio of ML/SG was predominantly high in the experimental runs. Figure 6 (a to d) shows the ML/SG ratio of various solute mixtures at given processing conditions. The highest ML/SG of 11.0 was obtained with S+MD 10DE at 60⁰C /40%/30 min processing conditions. This result is also in line with the previous research study (Shinde & Ramaswamy, 2019a), since the high molecular weight MD restricts the impregnation of solids while medium-low molecular sucrose favors the ML. Therefore the combination of sucrose and maltodextrin resulted in higher ML/SG. The similar outcomes was also reported when maltodextrin was used with mango chips at 40⁰C and 65% concentration giving ML/SG around 6.5 in 60 min (Yolanda & Rosana, 2009) and also, when MD was used with tomatoes the ML/SG was 7.06 at 35⁰C, 56.5% concentration in 1h (Dermesonlouoglou et al., 2016).

Table 4.4: t-test results for significance of differences in WR and ML/SG, between different solute mixtures

Solute mixtures	WR		ML/SG	
	Pr> t	t-value	Pr> t	t-value
S:MD 10DE-Sucrose	< 0.01	5.17	< 0.01	7.5
Sucrose -S:MD 15DE	< 0.05	2.94	> 0.05	-1.58
Sucrose-S:MD 18DE	< 0.01	6.84	> 0.05	1.27
S:MD 10DE -S:MD 15DE	< 0.01	8.87	< 0.01	6.71
S:MD 10DE -S:MD 18DE	< 0.01	8.71	< 0.01	7.96
S:MD 18DE -S:MD 15DE	< 0.01	-4.52	< 0.05	-3.19

(Note: values in bold are not significant)

4.4.6 Weight reduction

The weight reduction parameter is based on ML minus SG which reflects the net effect on the product weight. The weight reduction would have been much higher if no solids from osmotic solution entered the fruit tissue. This type of combination is observed only in OD situation while in all other types of drying WR is directly related to ML and is only contributed by ML. The WR results of solute mixtures at different processing conditions are presented in Figure 4.5 (e to h). The highest weight reduction of 55.5% was found with the S+MD 10DE at 60C/60%/40min processing conditions.

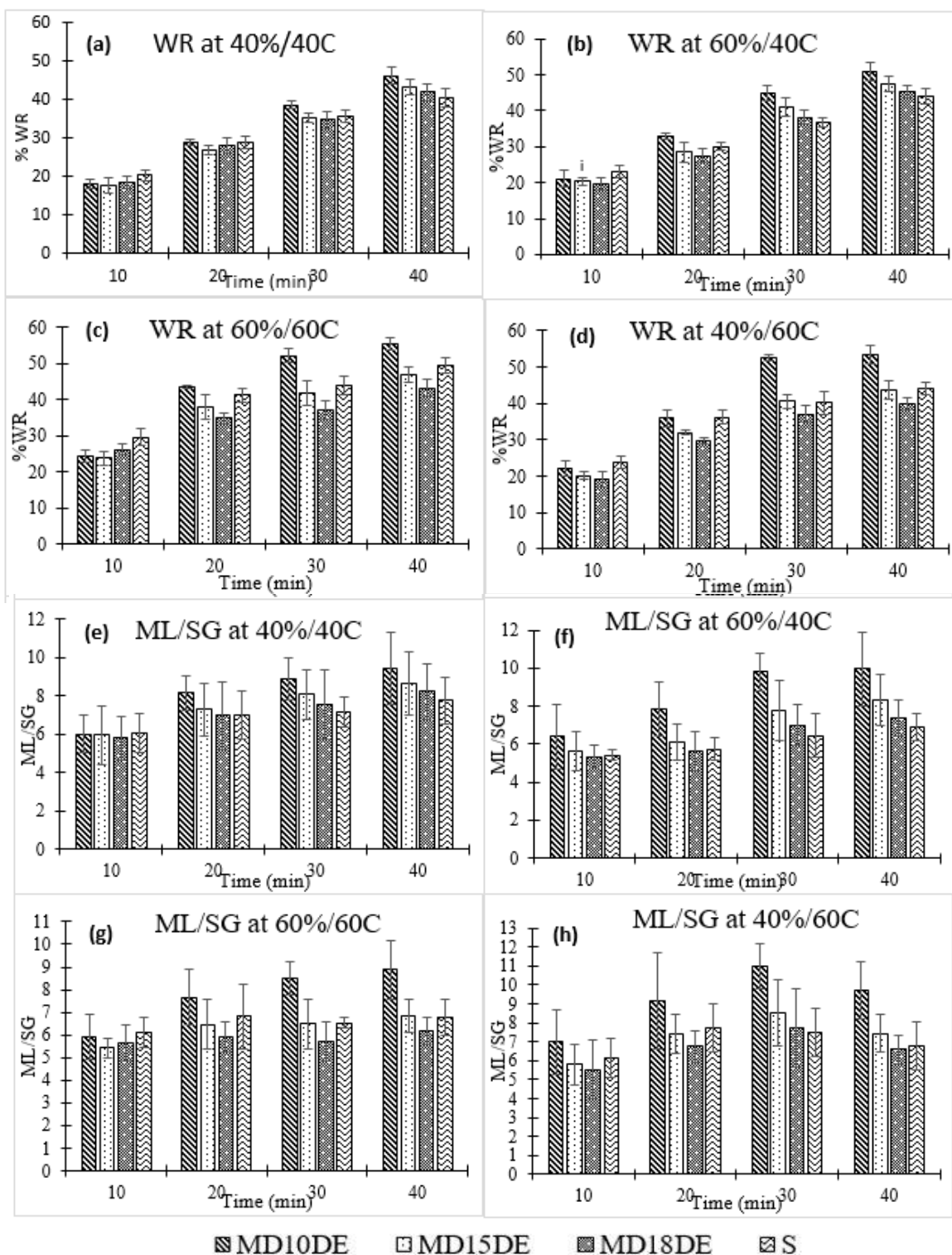


Figure 4.5: Weight reduction and moisture loss to solids gain ratio for various solutes at different temperatures, concentrations with respect to contact time. (*with standard deviation.*)

4.5 Conclusions

This study agrees with the previously accepted notion where it shows that increasing temperature and concentration leads to higher water and solids transfer coefficients. The solute mixture S+MD 10DE resulted in highest influence on k_w and k_s when compared with other solute mixtures. It was also observed that S:MD 10DE resulted in high NMC along with low NSC at any given temperature/concentration/time combinations in comparison with other solutes. Both modified first order empirical and Azuara models were effective in modeling the moisture loss and solids gain patterns. Overall, the results demonstrated that the use of maltodextrin 10DE along with sucrose improves mass transfer rates during MWODS process. S+MD 10DE gave the best performance leading to highest moisture loss and lowest solids gain in mango product, both conducive of leading to better drying efficiency and product quality.

PREFACE TO CHAPTER 5

The application of various grades of maltodextrin along with sucrose solute mixture on mass transfer kinetics was highlighted in previous chapters. Therefore, the recommended solute mixture of sucrose and maltodextrin (10DE) was further utilized in this chapter to understand the effect of osmotic process parameters on MWODS of mango while using sucrose and maltodextrin solute mixtures at a wider range of proportions (100:0 to 80:20). The study also determined the optimal processing conditions where it focused more on the usage of maltodextrin solute amount in sucrose solution during MWODS process. The mass transfer along with the quality of MWODS mangoes were studied and the model was developed to optimize the process based on quality characteristics and effective moisture loss with minimal solids uptake and high ML/SG ratio.

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

Shinde, B., and Ramaswamy, H.S. Optimization of maltodextrin (10DE) - sucrose moderated microwave osmotic dehydration of mango cubes under continuous flow spray mode (MWODS) conditions. Paper submitted to LWT journal. (*Paper in review*)

Shinde, B., and Ramaswamy, H.S., 2017. Optimization of microwave-osmotic dehydration under spray condition (MWODS) of mango (*Mangifera indica*) using combinations of sucrose and maltodextrin as the osmotic agents. Northeast Agricultural and Biological Engineering Conference (NABEC) 2017. Jul 30– Aug 2, Groton Connecticut, USA. (Poster presentation)

CHAPTER 5

Optimization of maltodextrin (10DE) - sucrose moderated microwave osmotic dehydration of mango cubes under continuous flow spray mode (MWODS) conditions

Abstract

The process of microwave osmotic dehydration of mango was optimized under continuous flow medium spray conditions (MWODS) with maltodextrin (10DE) moderated sucrose solutions. Optimization was carried out using a response surface methodology with a central composite rotatable (CCRD) design with three input variables at five levels (temperature, 33⁰C to 66.7⁰C; sucrose: maltodextrin ratio from 100:0 to 80:20; and solute concentration, 33 to 66.7%). The response parameters used for optimization were moisture loss (ML), solids gain (SG), weight reduction (WR), ML/SG ratio, color and texture values. For each response, RSM models ($P < 0.05$) were developed. As expected, all output variables were responsive to process variables and addition of maltodextrin to sucrose was found to have a significant effect on reducing the SG and increasing ML/SG, and solute concentration had significant effects on ML, SG and quality parameters. The process was optimized by desirability approach and MWODS at 56⁰C with solute 46% concentration and 84:16 sucrose:maltodextrin proportion had the highest desirability value.

5.1 Introduction

Osmotic dehydration has received many appraisals among consumers due to its ability to develop an intermediate moisture food product having lower water activity, which is imparted by solute gain and water loss (Ahmed et al., 2016). While the prospects of osmotic dehydration have attracted significant attention over the past decades as a pretreatment for conventional finish drying techniques. The techniques such as ultrasound, pulsed electric field, vacuum, centrifugal force or gamma-irradiation and microwave (MW) pretreatments have been combined with OD treatment to promote higher mass transfer (Rastogi et al., 2005) to improve the inherently slow osmotic dehydration process. Microwave environment has also been used in combination with OD making it a microwave osmotic dehydration (MWOD) method as opposed to MW pretreatment, and these have been proven to be effective to enhance water reduction by minimizing the processing time and ultimately limiting the solids uptake in comparison with conventional OD (Orsat et al., 2007; Ozkoc et al., 2014). The research has been discussed in detailed to explore the effect of microwave energy during OD using different process variables (Azarpazhooh & Ramaswamy, 2009b, 2011; Wray & Ramaswamy, 2013; Wray & Ramaswamy, 2015b) and compared with conventional OD to demonstrate the effectiveness of MWOD systems.

One of the additional effective factors influencing the OD is the selection of OD agents. This was recently evaluated for the MWOD process with medium flow under spray mode (MWODS) using solutes of various molecular weights such as dextrose, sucrose, and maltodextrin. It was found that amongst all solute combinations, the sucrose supplemented maltodextrin syrup facilitated the highest moisture loss and restricted solids gain along with minimum loss of color pigments (Shinde & Ramaswamy, 2019a). In the similar context, further work was based on evaluating the different grades of maltodextrins depending upon their dextrose equivalent values such as maltodextrin with 10DE, 15DE and 18DE which demonstrated that the solute mixture S+MD10DE (sucrose modified with maltodextrin 10DE) showed higher moisture transfer coefficient along with lowest solids transfer coefficient (Shinde & Ramaswamy, 2019b). However, in these studies the solute mixture proportion of sucrose and maltodextrins was restricted to 90:10 or 85:15. This current study is a logical extension of this maltodextrin moderated OD studies with a focus on optimization of process variables under

imposed set of constraints, such as maximizing ML, WR, ML/SG, hardness and chewiness while minimizing SG, ΔE .

In the last couple of decades, the response surface methodology (RSM) has been widely used for developing, improving and optimizing food processing. Furthermore, the desirability approach has been employed with RSM designs as a strategy for optimizing multiple responses. Although a large volume of literature is available on the use of different solutes and other variables, only a few have studied the application of complex solute mixtures using RSM which would enable osmotic dehydration with minimum impact on sensory attributes. Even those studied have been mostly related to conventional OD (Mourabet et al., 2017).

Therefore, in this context, the objective of the current study was to optimize the process of MWODS of mango cubes by employing a sucrose moderated maltodextrin solute mixture. Mango fruit was chosen due to its popular sensory characteristics (Jahurul et al., 2015) and limited process utilization (Sulistyawati et al., 2018). Further, the process impact on quality characteristics was evaluated immediately after the MWODS rather than the usual subsequent finish drying because this semi-dry product could also be further preserved by other techniques such as refrigeration and freezing or in intermediate moisture products without using the finish drying technique.

5.2 Materials and methods

5.2.1 Raw material, quality, and moisture content

Frozen mango cubes were obtained from a local food company (Nature's Touch, Canada) and kept frozen (-21°C to -27°C) until use. Prior to use, the mango pieces were thawed overnight (8-10 h) in a refrigerator (4°C - 7°C). The dimensions of the mango pieces were in the general shape of a cube with side dimensions of approximately 15 ± 2 mm. The quality of freshly thawed mango cubes was measured (methodology detailed later) and the initial values were for color: L^* 36.8 (± 3.4), a^* 9.9 (± 2.0), b^* 37.1 (± 3.1) and texture: hardness 600g (± 50), and chewiness 197g mm (± 19). Commercial grade sucrose (Lantic Sugar Ltd., Montreal, Qc, Canada) and maltodextrin 10DE (Univar Pvt. Ltd, Canada) were used for preparation of the osmotic solution. The selection of maltodextrin 10DE grade was based on the results of prior study (Shinde & Ramaswamy, 2019b). Where it was concluded that the sucrose moderated

maltodextrin 10DE (among different grades) had the highest potential for moisture removal and lowest solids gain.

The moisture content of the frozen-thawed (untreated) mango cube was measured using (AOAC, 1975) by keeping the cubes in an oven at 105⁰C for approximately 24h (until a constant weight was achieved). The average moisture content of mango cube was 86.1% (wb).

5.2.2 Microwave set up of MWODS

The setup of microwave osmotic dehydration under continuous flow medium spray condition (MWODS) is explained in detailed in Chapters 3 and 4 (Azarpazhooh & Ramaswamy, 2009b; Wray & Ramaswamy, 2013). Briefly, MWODS assembly is composed of a domestic microwave (Danby DMW1153BL 0.031 m³, Guelph, ON, Canada) with power output of 1100W and 2450MHz, which contained a spray head (Waterpik, Model RPB-173C, 12.5cm diameter, Waterpik Technology Inc., Markham, ON, Canada) attached to the custom-made glass sample chamber (12.5cm diameter). Test samples were placed in a Nylon mesh bag on the porous acrylic plate “stage” inside the glass sample chamber. This allowed to drain down the sprayed osmotic solution while keeping the samples direct in contact with the microwave. The osmotic solution collected at the bottom of the sample chamber was recycled through a long coil heat exchanger immersed inside a heated water bath (Model TDB/4 Groen Division, Dover Corp, IL) and then pumped through the spray head, using a peristaltic pump (Model 75211-30 Digital gear pump, Barnant company IN). The water bath temperature was set to the inlet temperature of the osmotic solution, and the osmotic solution was then circulated through the assembly to equilibrate the setup temperature before putting the samples into the system. A pair of in-line Type-T thermocouples were connected the digital thermometer (Omega DP-462, Omega technology, Laval, QC) to monitor the temperature of the osmotic solution. The thermocouples were placed immediately before and after the microwave cavity to measure the temperature of the solution before going inside the microwave and immediately after coming out of the microwave. The increase in the temperature of the osmotic solution after residing in microwave cavity was about 4-5⁰C, which was cooled as it passed through the long coil heat exchanger placed in the water bath to return to the original initial temperature before coming into the microwave. The length of the coil was long enough to allow the osmotic solution in the solution to sample ratio of 30:1 to pass through. A large amount of osmotic solution in a closed system

also allowed to maintain a relatively constant solute concentration throughout the experiments for each solute type, which was measured using a handheld refractometer.

5.2.3 Experimental procedure

5.2.3.1 MWODS experiments

Each batch consisted of 12-15 pieces of accurately weighed mango cubes (100 ± 1 g) placed in a single layer in two Nylon mesh bags (each containing 50g samples) and on the perforated acrylic stage in the sample chamber. The system was set up, and the osmotic solution was preheated according to the selected temperature of the run type. The osmotic solution at a fixed flow rate was allowed to flow through the system with the microwave turned fully on (100% power level). The pump was stopped after each test run of 30 min, and then samples were removed. The excess osmotic solution adhering to the product surface was shaken off and wiped with a wet paper towel, and the mango cubes were then weighed again. A portion of the sample was then used for moisture determination and the rest for quality evaluation. Moisture content was determined in a dry oven set at 105°C for approximately 24h (AOAC, 1975) (dried to constant weight).

5.2.4 Experimental design

Experiments were carried out based on a central composite rotatable design (CCRD) using three factors namely concentration, temperature, and sucrose to maltodextrin ratio, at five coded levels (-1.68, -1, 0, +1, +1.68) as shown in Table 5.1, and each run was carried in triplicate. The concentration ranged from 33.7-66.7 % and temperature ranged from 33.7 - 66.7°C , whereas the solute mixture of S: MD ranged from 100:0 to 80:20. The selection of the range for each variable was adopted from a previous research study (Shinde & Ramaswamy, 2019a). These covered ranges were broader than used in the previous study to be inclusive for the purpose of optimization. Flow rate (1050 mL/min) and osmotic dehydration time (30 min) were kept constant to limit the number of variables.

5.2.5 Dehydration responses and data analysis

Moisture loss (ML), solids gain (SG), moisture loss to solids gain ratio (ML/SG) and weight reduction (WR) were obtained using the following equations:

$$\text{Moisture Loss (ML)\%} = 100 \frac{M_0 X_0 - M_t X_t}{M_0} \quad (5.1)$$

$$\text{Solids Gain (SG)\%} = 100 \frac{M_0 S_0 - M_t S_t}{M_0} \quad (5.2)$$

$$\text{ML: SG ratio} = \frac{\%ML}{\%SG} \quad (5.3)$$

$$\text{Weight Reduction (WR)\%} = 100 \frac{M_0 - M_t}{M_0} \quad (5.4)$$

where M_0 and M_t are the total mass of the fruit sample at time 0 and t , respectively; X_0 and X_t are the moisture fractions (kg/kg, wet basis) at time 0 and t , respectively; S_0 and S_t are the solid fractions (kg/kg, wet basis) at time 0 and t , respectively.

5.2.6 Quality analysis

Separate sample runs were made for each run type, under the same conditions for quality analysis and the quality parameters were measured as elaborated below. As explained before, the OD is gaining importance due to its ability to produce an intermediate product which can be incorporated in different finished products. In general, despite the fact that the intermediate moisture product is not fully stable from a preservation point of view, the quality of MWOD mangoes would be useful in certain scenarios such as refrigeration and freezing, pie and cake preparations, inclusion in extrusion formulations, etc.

5.2.6.1 Texture evaluation

TA.XT Plus Texture Analyzer (Stable Microsystems, Surrey, UK) was used for texture profile analysis (TPA) of both the MWODS-treated samples and fresh (frozen-thawed) mango cubes. Two-cycle compression test was performed to obtain TPA, using a flat bottom probe of 25 mm diameter, with pretest speed of 5 mm/sec, the test speed of 5 mm s⁻¹ and post-test speed of 5 mm s⁻¹. The target compression was about a distance of 3 mm into the sample during two consecutive cycles to target a 25% deformation from the average height of the samples. These settings with minor modifications were used with guidance from Banjongsinsiri et al. (2004) (Banjongsinsiri et al., 2004), who used TPA on mango cubes. The analysis was performed with six replicates, and the average values (with standard deviation) were used. Hardness and chewiness were selected in this study as parameters to determine the texture influence of osmotically processed mango cubes. The peak force defined hardness during the first compression cycle and chewiness was obtained from the product of gumminess and springiness (Bourne, 2002). The chewiness was calculated using Equation 5.5.

$$\text{Chewiness} = \text{Gumminess} \times \text{Springiness} \quad (5.5)$$

Table 5.1: CCRD run numbers with results for MWODS dehydration and quality parameters

Experimental design				Experimental results									
Std. order #	Temp. (°C)	Conc. (%)	MD (%)	ML (%)	SG (%)	ML/SG	WR (%)	L*	a*	b*	Δ E	Hardness (g)	Chewiness (g mm)
1	40	40	4.0	42.3(1.9)	5.94(1.3)	7.33(1.3)	36.4(3.0)	40.1(2.0)	9.18(0.63)	19.1(2.12)	18.1 (2.1)	130 (16.4)	43.6 (7.48)
2	60	40	4.0	53.7(2.2)	6.45(1.1)	8.46(1.3)	47.2(3.2)	36.9(1.4)	9.53(0.75)	16.5(2.14)	20.9 (2.0)	122 (17.5)	59.5 (8.44)
3	40	60	4.0	47.4(1.2)	6.84(1.2)	7.03(0.9)	40.6(1.1)	41.7(1.5)	8.07(0.5)	20.5(1.86)	17.4 (1.7)	162 (14.8)	58.3 (8.33)
4	60	60	4.0	59.0(1.1)	8.87(1.0)	6.69(2.2)	50.1(0.2)	39.9(1.6)	7.80(0.71)	17.4(0.85)	20.0 (0.8)	189(18.4)	79.6 (9.89)
5	40	40	16	43.2(1.3)	4.61(1.0)	9.64(2.0)	38.6(2.4)	34.5(1.3)	9.34(1.26)	28.1(1.7)	9.2 (1.7)	146(15.1)	58.6 (10.1)
6	60	40	16	56.9(2.2)	4.98(1.1)	11.8(2.2)	52.0(3.3)	37.4(1.4)	9.86(0.88)	27.2(2.6)	10.1 (2.4)	158(13.8)	71.6 (9.27)
7	40	60	16	50.2(2.6)	5.06(1.2)	10.2(1.5)	45.1(1.4)	39.2(2.1)	9.49(0.3)	28.6(2.78)	8.8 (2.4)	168(12.4)	61.7 (6.52)
8	60	60	16	60.2(1.6)	6.82(1.1)	8.93(1.5)	53.4(0.6)	40.6(2.2)	10.6(0.61)	22.5(2.5)	15.1 (2.4)	235(19.8)	94.2 (8.68)
9	33	50	10	45.7(1.6)	5.76(1.3)	8.13(1.5)	40.0(0.3)	38.1(3.4)	8.40(0.9)	27.7(3.5)	10.4 (2.0)	114(11.9)	35.6 (10.6)
10	67	50	10	55.3(3.0)	7.11(1.1)	7.91(1.3)	48.2(4.1)	35.6(2.5)	10.1(0.37)	21.7(1.3)	15.5 (1.2)	158(17.0)	69.2 (10.5)
11	50	33	10	45.3(1.8)	6.03(1.0)	7.60(1.0)	39.3(0.8)	36.7(3.1)	9.58(0.7)	26.7(3.0)	10.8 (2.8)	110(15.3)	38.2 (9.22)
12	50	67	10	51.3(1.9)	7.43(1.0)	6.95(0.7)	43.9(1.0)	37.4(2.1)	9.34(0.27)	25.3(2.5)	12.0 (2.2)	169(18.0)	66.0 (9.62)
13	50	50	0	45.8(1.8)	9.14(1.1)	5.05(0.8)	36.6(2.9)	40.2(2.6)	8.42(1.3)	18.2(2.10)	19.4 (2.2)	140(16.6)	39.0 (7.79)
14	50	50	20	49.9(1.3)	5.05(1.3)	10.2(2.4)	44.9(0.1)	33.2(3.9)	9.98(0.63)	27.9(1.99)	10.6 (1.5)	210(14.7)	71.2 (9.62)
15	50	50	10	46.7(1.2)	6.94(0.9)	6.78(0.8)	39.7(0.2)	38.6(2.8)	9.70(0.94)	26.2(2.5)	11.3 (1.9)	192(16.7)	60.4 (10.1)
16	50	50	10	48.7(1.1)	7.11(1.0)	6.95(1.1)	41.6(0.2)	39.4(1.7)	9.40(1.94)	25.4(2.7)	12.2 (2.3)	186(18.1)	63.7 (10.7)
17	50	50	10	47.9(2.0)	6.65(0.9)	7.31(1.4)	41.2(3.0)	35.6(2.0)	8.36(0.39)	22.1(2.6)	15.2 (2.8)	198(10.8)	54.2 (9.27)
18	50	50	10	49.2(2.4)	6.73(1.5)	7.36(0.6)	42.5(1.5)	36.2(2.6)	9.8(2.03)	21.2(2.1)	16.0 (2.1)	206(13.4)	59.5 (8.48)
19	50	50	10	50.6(2.7)	5.54(1.2)	9.56(1.1)	45.0(4.2)	39.1(2.7)	9.36(2.03)	26.9(2.5)	10.7 (2.9)	195(18.5)	60.3 (10.1)
20	50	50	10	50.9(1.4)	6.50(1.6)	7.95(1.0)	44.4(0.2)	38.5(2.5)	8.97(1.5)	26.3(1.6)	11.2 (1.1)	201(17.5)	65.2 (10.1)
Frozen thawed								36.8(3.4)	9.90(2.0)	37.1(3.1)		600(50.0)	197 (19.0)

(Note: The Mean values of three replicates with (standard deviation shown) are given in the table)

5.2.6.2 Color

The color of post-MWODS samples was analyzed in the L^* , a^* , b^* system using a tristimulus Minolta Chroma Meter (Minolta Corp., Ramsey, NJ, USA). The Chroma Meter was warmed up 20 min prior to use and the color was calibrated against a white standard. Six measurements were made with each sample, and the values were averaged to obtain the L^* (lightness), a^* (green (−) to red (+)), and b^* (blue (−) to yellow (+)) values of the individual trials. The ΔE (total color change) was also determined as per Equation 6 (Maftoonazad & Ramaswamy, 2008).

$$\Delta E = \sqrt{(L_0 - L)^2(a_0 - a)^2(b_0 - b)^2} \quad (6)$$

5.2.7 Statistical analysis

A central composite rotatable design of three factors (process temperature, solute concentration and maltodextrin content in the solute mixture) with five levels, 6 central points, and 6 axial points to 14 full factorial design was used. All statistical analysis was carried out using Design-Expert version 6.10 software (Stat-Ease Inc, Minneapolis, Minnesota, USA). The significant terms ($p < 0.05$) in the model were found by analysis of variance (ANOVA) for each response.

5.3 Result and discussion

5.3.1 Response surface methodology

The response surface methodology was used due to its multipurpose application in research work, where a representative minimum experimental runs give optimal trends using analysis of variance (ANOVA). One of the essential parts of RSM is to obtain a statistically significant model for each response as a function of process variables ANOVA. The output response results from the CCRD design of experiments are summarized in Table 5.1 (standard deviation included in parenthesis for each output to represent experimental variability). Table 5.2 details the selected RSM model for each response based on their statistical significance as well as lack of fit values. The linear model ($p < 0.0001$) was selected for the responses ML, SG, ML/SG, WR, L^* , b^* , ΔE , and chewiness, the quadratic model was selected for hardness ($p < 0.05$), whereas a^* was best represented by an interaction 2FI model ($p < 0.0001$). The lack of fit values for each selected model was not significant ($p > 0.05$), which makes the models be

significant. A non-significant lack of fit explains that the selected model can predict the responses using the predicted equations (Table 5.2) for each response (ML, SG, etc.), as a function of input variables with >95% confidence.

Table 5.2: Predicting equations and compiled ANOVA results for CCRD responses

Responses	Model	Predicting equations in terms of actual variables	Lack of fit	R ²
ML	Linear	$= +14.14 + \mathbf{0.46}^*T + 0.22^*C + 0.18^*M$	0.1374(NS)	0.8032
SG	Linear	$= +2.73 + 0.05^*T + 0.06^*C - \mathbf{0.17}^*M$	0.4950(NS)	0.8101
ML/SG	Linear	$= +7.19 + 9.48E^{-003}^*T - 0.04^*C + \mathbf{0.24}^*M$	0.5675(NS)	0.6585
WR	Linear	$= +11.4 + \mathbf{0.41}^*T + 0.17^*C + 0.35^*M$	0.2014(NS)	0.7361
L*	Linear	$= +36.3 - 0.04^*T + 0.11^*C - 0.19^*M$	0.3514(NS)	0.5001
a*	Interaction	$= +10.8 + 0.08^*T - 5.87E^{-004}^*C - 1.04^*M - 2.43E^{-003}^*T^*C + 9.92E^{-003}^*T^*M + 0.01^*C^*M$	0.7441(NS)	0.8240
b*	Linear	$= 27.51 - 0.17T - 0.03C + \mathbf{0.60}M$	0.6457(NS)	0.7162
ΔE	Linear	$= +17.8 + 0.06^*T - 5.22E^{-003}^*C - \mathbf{0.66}^*M$	0.4742(NS)	0.6974
Hardness	Quadratic	$= -522 + 11.9^*T + \mathbf{13.1}^*C - 1.15^*M - 0.18^*T^2 - 0.17^*C^2 - 0.12^*M^2 + 0.11^*T^*C + 0.13^*T^*M + 1.38E^{-15}^*C^*M$	0.0519(NS)	0.9363
Chewiness	Linear	$= -41.4 + \mathbf{1.39}^*T + 0.78^*C + 1.21^*M$	0.0518(NS)	0.7848

Where T is Temperature (°C), C is Concentration (%), M is maltodextrin content (%) in the solute. Note that highly significant ($p < 0.0001$) models and variables are in bold, significant ($p < 0.05$) are normal type, while non-significant ($p > 0.05$) factors are italicized.

5.3.2 Effect of MWODS processing variables

The MW oven was kept fully on and therefore, the power level was not a variable. Therefore, the performance power constraints should be respected and the main variables which are directly related to power are the flow rate and the sample size which were kept constant in this study. Hence with any scale-up considerations involving higher flow rates or higher sample sizes, the power factor should be taken into consideration otherwise the desired temperature set points and the observed MW effect may not be fully correlated. The response models are presented in Table 5.2. The various quality parameters and osmotic parameters are important factors while optimizing the process, as they have direct impact on quality and the dehydration

process. Therefore, using the analysis of variance (ANOVA) for each response, the significant and non-significant model terms were defined in this study.

The effect of process variables on each of the osmotic dehydration performance indicators ML, SG, ML/SG, and WR are shown in Figure 5.1. They have a bearing on the drying characteristics (ML, SG, WR; higher values indicate generally better performance) and selective desirability (ML/SG). While the ML is desirable, SG which infuses solution solutes into the fruits matrix is often desired to be minimized. Hence a higher ML/SG ratio is indicative of desirable performance. Figure 5.2 shows the influence of processing variables on color and textural parameters. The mechanical properties of the product such as texture and color are usually measured after the completion of the final drying. However, during this study, these are evaluated at the intermediate level (prior to the second stage finish drying) to assess their use in other product/process applications. The effect of variables on the color of post-MWODS samples was measured with L^* , a^* , b^* parameters, whereas the parameter ΔE was used to explain the overall color difference as influenced by the MWODS and also between fresh (frozen-thawed) mango and MWODS processed mangoes. During this study, each of the factors was also examined separately to understand the effect of each processing variables (temperature, concentration and MD content). These response plots are presented in Figure 5.2 (e to l) to show a better understanding of the effects of each processing factor with color parameters.

5.3.3 Effect of Temperature

The temperature was found to be a significant ($p < 0.05$) factor for the mass transfer parameters ML, SG, WR as shown in Table 5.2 and Figure 5.1. Amongst all parameters, ML and weight reduction were found to be affected significantly ($p < 0.001$) by temperature with a linear relationship. The temperature had a positive effect which was indicative of the fact that an increase in temperature would result in higher ML, SG, ML/SG, and WR. One of the reasons behind massive moisture loss was the rapid heating of water molecule in presence of microwave, increasing the internal pressure and promoting quick removal of water from the fruit piece (Azarpazhooh & Ramaswamy, 2009b; Wray & Ramaswamy, 2015a). The previous research work explained the prominent effect of temperature on ML in some key points (Azarpazhooh & Ramaswamy, 2009b; Wray & Ramaswamy, 2015a).

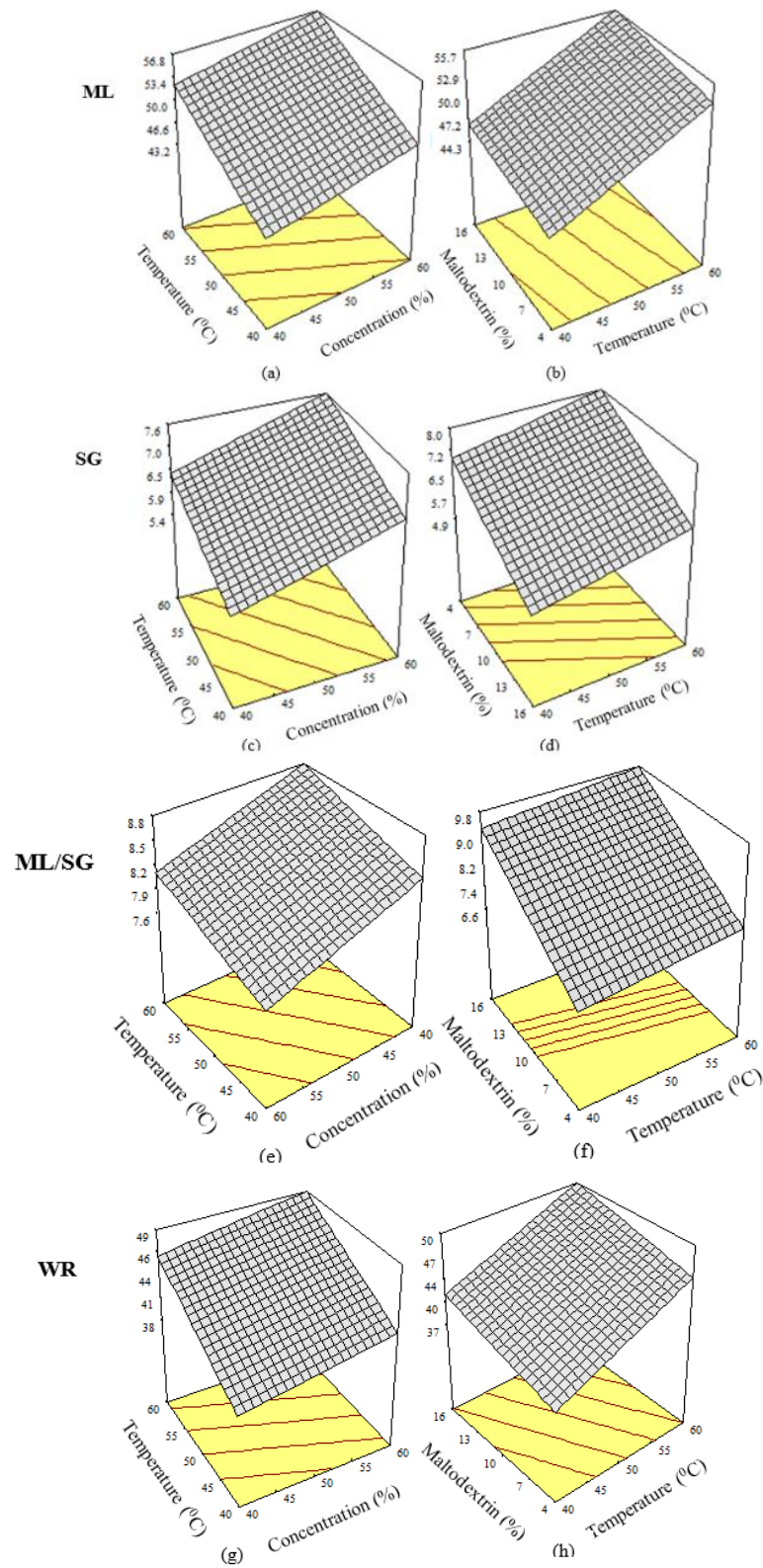


Figure 5.1: Response surface plots for mass transfer for ML, SG, WR, ML/SG. (When not a variable, inputs were fixed at their center point: concentration 50%, maltodextrin 10%)

The higher temperature reduces the viscosity thereby improving the mobility of water, allowing better contact between OD solution and mango cubes and ultimately improves the extraction ability of the OD solution. This fact was most useful under spray conditions where high viscosity could be an issue while using high concentration OD solutions (Azarpazhooh & Ramaswamy, 2009b). Another point is that thermal energy can cause swelling of an individual cell thereby increasing the permeability of cell wall which ultimately improves the water movement in the samples. Finally, the elevated temperatures generally promote moisture diffusion, and therefore water can easily diffuse out of the sample into the OD solution.

Similarly, for solids gain (SG) the temperature factor had a positive coefficient which resulted in an increase in SG. This is in agreement with previous studies (Azarpazhooh & Ramaswamy, 2011; Azarpazhooh & Ramaswamy, 2012a; Wray & Ramaswamy, 2013). In addition, weight loss is a combined indicator of the efficiency of osmotic dehydration process. The weight reduction (WR) is directly related to the moisture loss but gets moderated by the solids gain. Typically, the ML is larger than SG by an order of the magnitude. Therefore, it is not surprising to see the response WR follows a similar trend of ML (Wray & Ramaswamy, 2013). These results of mass transfer responses are in line with the general conclusion of other osmotic studies as well as previous studies of MWOD under immersion as well as spray conditions (Azarpazhooh & Ramaswamy, 2012a; Li & Ramaswamy, 2006b; Van Nieuwenhuijzen et al., 2001; Wray & Ramaswamy, 2015a). However, for ML/SG response, the temperature was found to be a not significant factor, likely because of a similar influence on ML and SG.

For hardness, the temperature was found to be one of the significant factor ($p < 0.05$) and a quadratic model provided a better fit whereas, for chewiness, the linear model performed better ($p < 0.0001$). Higher temperatures resulted in better hardness and chewiness retention of the sample. The possible explanation of this outcome is, that the temperature rise along with elevated concentration led to increases in the penetration of solutes into the fruit tissue, producing a firmer structure, low porosity and loss of elasticity in fruit tissues (Tabtiang et al., 2012). The temperature had positive influences with a^* and ΔE and negative with L^* and b^* . This is indicative of higher temperatures leading to an increase in a^* (redness) and ΔE (an overall change in the color of the dried product), while decreasing the b^* value or the yellowness of the sample. Also, some interactions were significant confirming some previous trends (Chun et al., 2012; Tabtiang et al., 2012).

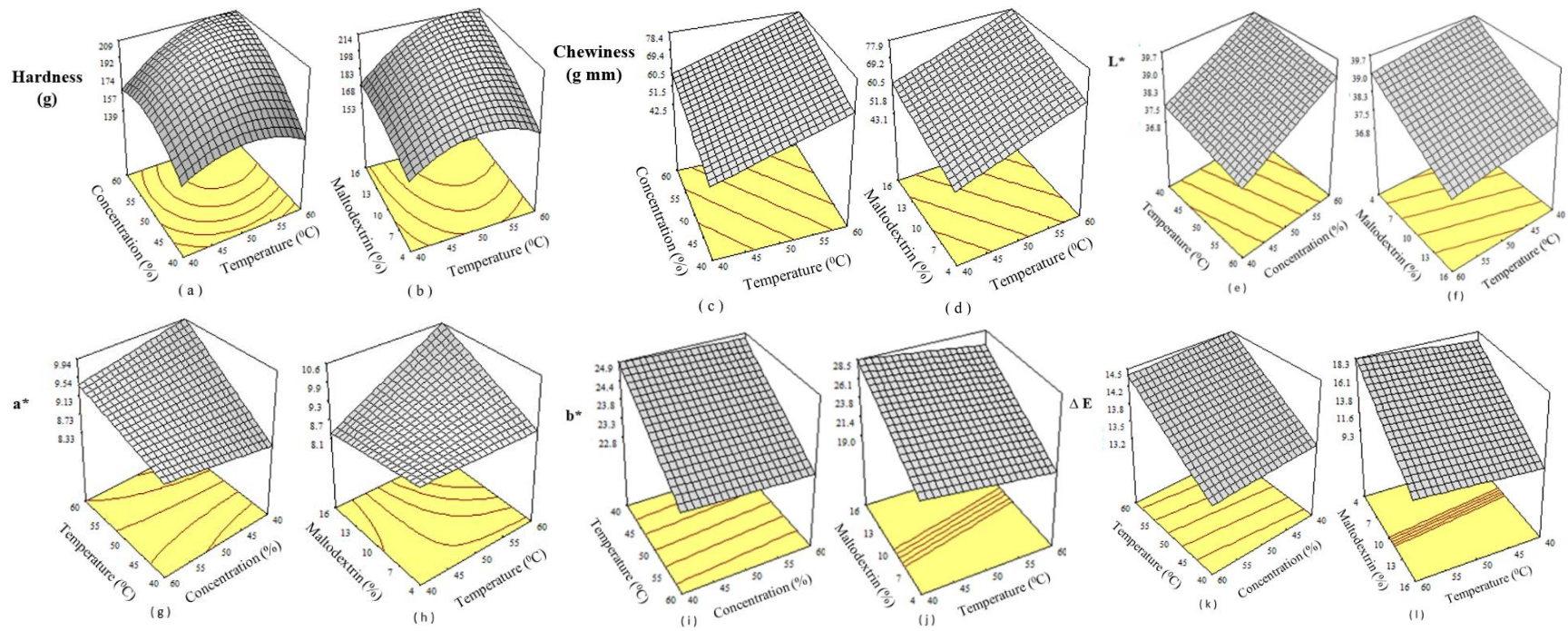


Figure 5.2: Response surface plots for hardness, chewiness, L*, a*, b*, ΔE . (When not a variable, inputs were fixed at their center point: concentration 50%, maltodextrin 10%)

5.3.4 Concentration

The solute concentration is considered as one of the crucial factors while studying OD process. The concentration was found to be significant ($p < 0.05$) for ML, SG, and WR at linear model. Based on the sum of the squares, it was found that concentration had a higher effect than temperature, and MD content on SG response during MWODS as observed in earlier studies (Azarpazhooh & Ramaswamy, 2012a; Van Nieuwenhuijzen et al., 2001). On the contrary there was a negative effect of concentration on ML/SG, indicating an undesirable element for the use of extreme concentrations which has also been observed in earlier studies (Wray & Ramaswamy, 2013). These results can be visualized in the response surface plots in Figure 5.2.

The concentration effect on hardness was found to be significant at the quadratic model, as demonstrated by the response surface plots presented in Figure 5.2 (a to b). The positive effect of concentration was also explained earlier while discussing the temperature effect. The influence of concentration on chewiness was linear. Whereas, the concentration was found to be not significant for the color parameters a^* , b^* , and ΔE .

5.3.5 Maltodextrin incorporation

Maltodextrin content was found to be one of the significant factors for most mass transfer responses as well as quality characteristics. The maltodextrin influence on MWODS ML was significant ($p < 0.05$) with a linear model as shown in Table 5.2. The positive coefficient of MD content indicates that there was an increase in ML with increased MD content in osmotic solution. This fact explains that the higher molecular MD creates a barrier on the surface of the mango cubes and tends to cause an increase in osmotic pressure which ultimately targets on moisture removal of fruit samples (Azuara et al., 2002). Hawkes and Flinks (1978) also observed that osmotic dehydration in the presence of sucrose with the addition of other solutes improved the effectiveness of moisture removal in comparison to sucrose alone (Hawkes & Flink, 1978). Further, the negative coefficient of MD on SG is a major significant influence that favors the use of maltodextrin inclusion. SG is suppressed at higher MD content. This confirms the role of the added high molecular weight maltodextrin in restricting the solids uptake which is enabled by a probable dense coating on the surface of the fruit pieces. Earlier research (Hawkes & Flink, 1978; İspir & Toğrul, 2009a) also confirms that solids uptake is inversely correlated with the

molecule size of the osmotic agent. Therefore, SG can be moderated by adding other high molecular solutes to the sucrose syrup.

Other studies with conventional OD of apricot cubes have reported 42% ML and 26% SG under 70% concentration and 45°C temperature in 24h (İspir & Toğrul, 2009a); Also, with beef, 51% of ML, 19% SG at 15°C temp., 60% MD for 3h (Dimakopoulou-Papazoglou & Katsanidis, 2017). With the current study using MWODS, the maximum ML was about 60.2% at 60°C, 60% concentration and 16% MD fraction in 30 min, whereas 6.82% of SG was observed under similar condition. On the other hand, MWODS using only sucrose solution under similar concentration resulted in 52% ML and 7.96% of SG (Shinde & Ramaswamy, 2019a). Therefore, these observations attributed to the fact that current study of MWODS with modified MD solute mixture produced higher ML and lower solids uptake in 30mins.

A particular interest in OD studies is its influence on ML/SG ratio which as discussed earlier is representative of a more desirable osmotic dehydration which limits the penetration of the osmotic solution into the fruit and favors concentration of internal fruit sugars by moisture removal. Response surface plots ML/SG are shown in Figure 5.2 (e) and (f) showing the influence of MD content in osmotic solution. It was observed that MD contents were significant ($p < 0.05$) for ML/SG; there was a consistent increase in ML/SG with MD content. The positive coefficient of MD and concentration in most of the models explained the same fact that with elevated concentration and MD content, a higher ML/SG ratio can be achieved. This supports the results obtained in previous studies where mango chips when treated with MD solute at 40°C and 65% concentration gave ML/SG around 6.5 in 60 min (Yolanda & Rosana, 2009) and with tomatoes it was 7.06 at 35°C, 56.5% concentration in 1h (Dermesonlouoglou et al., 2007). Higher value of 11.8 for ML/SG was found at 60°C, 40% concentration and 16% MD content when treated for 30 min in this study. Similarly, when comparing these results with the previous MWODS with sucrose only solution (Shinde & Ramaswamy, 2019a) the ML/SG reported was 7.46 at 60°C/40%/30min.

With respect to WR, again the positive coefficient of MD term indicates that with an increase in MD content there was also an increase in WR, which can be seen in Figure 5.2 (h). Since higher ML along with lower SG would favor a better WR. The application of MD in OD solutes restricts SG due to the difficulties in impregnating into the fruit pieces because of the higher molecular size. Also, MD favours ML by increasing the moisture gradient from inside to

the solution and ultimately contributes positively to ML response. Hence, the addition of MD to the regular sucrose solute improves the MWODS process by improving WR factor.

A quadratic model was found while relating the maltodextrin influence on hardness with the negative coefficient which explains that it has an inverse relationship. This result is in agreement with previous studies where OD of blueberries showed decreased hardness with increased MD agent concentration (Chun et al., 2012). This is because ML increases with increased MD content and as a result, the fruit tissues may have reduced turgor pressure and so as to reduced hardness. Therefore, an increased concentration and temperature, along with elevated MD content will balance the conditions for better textural qualities of osmotically dried mangoes. For the chewiness, the MD factor was found to be significant with positive coefficient and a linear model, indicating the fact that increased MD would produce the samples with better chewiness, a desirable feature when these products are incorporated into fruit cakes or cereals. This outcome was supported by the fact that the solute mixtures containing polysaccharides such as maltodextrin protect the cell wall and exhibit less damage to the middle lamella and therefore less severe shrinkage could occur in cell content. A similar effect was reported when studying OD with strawberries where increased chewiness and firmness were recorded in the presence of maltodextrin content (Dermesonlouoglou et al., 2016). The response surface plots for chewiness shown in Figure 5.2 (k) and (l) indicate the chewiness association with process variables.

MD was found to have a significant effect ($p < 0.05$) on all color parameters, as presented in Figure 5.2 (e to l). It had a positive coefficient with b^* and negative with L^* , a^* and ΔE . Thus, an increase in MD content will retain the yellowness of the samples and minimize the lightness, redness as well as a color change of post-MWODS samples. In short, MD has a protective effect on the leaching of the color pigment. It was also supported by other studies with blueberries (Chun et al., 2012) and tomatoes (Dermesonlouoglou et al., 2007) using maltodextrin solute, enhanced palatability, better color, and preferable texture.

5.4 Optimization and model validation

Optimization step was performed by setting the input parameters of concentration, temperature and percent maltodextrin in osmotic solution (OS) to be “in range” while ML, ML/SG ratio, WR, hardness, and chewiness value were to be "maximized". In addition, the quality parameters hardness and chewiness were set at a "maximum range". The color

parameters L^* and a^* were set on the targeted values of the fresh mangoes such as 36.5 and 9.90 respectively, whereas the b^* value was set to "maximum" due to the fact that fresh mangoes had highest b^* value compared to post-MWODS samples. In contrast, the SG and ΔE total color change were set to be 'minimized.' The program was run for the optimum conditions and the best solutions obtained are presented in Table 5.3. The table summarized the optimum conditions of independent variables within the specified constraints and also shows the predicted values of the responses under the suggested conditions. The resulting Solution 1 had the highest desirability of 0.774. Although the highest desirability would be 1.0, with conflicting influences of different constraints, only a compromise is possible and the best one had the highest desirability value of 0.774. This condition was selected for the validation process. The selected optimized conditions were found at 56°C temperature, 46% concentration and 16% maltodextrin content in osmotic solution and these conditions were used for model validation.

The MWODS experiments were performed using the derived optimum drying conditions and mass transfer parameters as well as quality attributes of the resulting products were determined. The predictive model validation along with confidence intervals and the experimental values (mean of 4 measurements) are presented in Table 5.3. At this point, ML, SG, ML/SG, and weight reduction were calculated as 51.3%, 5.88%, 8.95 and 45.4%, respectively and compared reasonably well with the predicted values. While comparing the result, 43% ML and 8.1% SG, and 5.30 ML/SG were found during optimization of potato at 47°C, 50% sugar and 10% salt concentration in 6h time (Ravindra & Chattopadhyay, 2000). A principal reason for the difference in values, especially ML/SG is the use of MWODS technique in the current study, which has the capability to provide higher ML and lower SG and hence higher ML/SG ratio as supported by many previous studies from our laboratory (Azarpazhooh & Ramaswamy, 2009b, 2012a; Wray & Ramaswamy, 2013). A major factor for process economics is the fact that the MWODS process only takes 30 min as compared to more than 6h for conventional OD. A significant outcome of this present study is the synergistic effect of the appropriate use of MD combination to further optimize these outcomes.

Table 5.3: Solutions for optimum conditions and predictive model validation

Constraints/ Responses	Optimization solutions						Model validation			
	1	2	3	4	5	6	Predicted value	CI Low	CI High	Observed value
Temperature	56	55	54	58	52	58	56	-	-	-
Concentration	46	47	49	44	51	41	46	-	-	-
MD content	16	16	16	14	16	16	16	-	-	-
ML	53.0	53.0	53.0	52.9	52.1	52.8	52.9	50.9	55.0	51.3 (2.00)
SG	5.57	5.61	5.68	5.81	5.62	5.38	5.56	5.07	6.05	5.88 (1.09)
ML/SG	9.74	9.69	9.60	9.48	9.53	9.94	9.72	8.79	10.8	8.95 (1.55)
WR	47.4	47.4	47.4	47.2	46.5	47.5	47.5	45.2	49.8	45.4 (1.54)
L*	36.5	36.6	36.9	36.5	37.1	35.9	36.5	35.2	37.8	38.1 (2.28)*
a*	9.90	9.90	9.90	9.90	9.68	9.90	9.89	9.41	10.4	10.9 (2.06)*
b*	27.0	27.1	27.1	25.8	27.4	26.9	27.0	25.1	28.9	29.2 (1.95)*
ΔE	10.3	10.2	10.2	11.5	10.0	10.4	10.3	8.21	12.3	8.62 (1.57)
Hardness	201	205	212	184	213	173	199	185	213	160.5 (19.9)
Chewiness	70.9	71.2	71.9	68.7	70.2	69.0	70.7	64.6	76.8	63.99 (9.17)
Desirability	0.774	0.770	0.757	0.725	0.722	0.707	-	-	-	-

Mean values of four replicates with (standard deviation shown). CI: Confidence Interval (95%),

‘*’ Denotes observed value falls outside of predicted value confidence interval

As mentioned earlier, the optimization achieved in this case had too many constraints. If these were relaxed, much higher desirability values could be reached for optimization at the relaxed levels. Some of these are listed in Table 5.4. This table lists the desirability function to range from 0.77 to 1.0, clearly much better than the ones presented in Table 5.3. However, the constraints are very limited. For example, if one is only interested in maximizing the ML, then it would only be possible to generate optimum conditions with desirability values between 0.8 to 0.85, but if one looks at only keeping the SG minimum, it is possible to get the perfect desirability in terms of achieving the result. However, the conditions involved would represent very mild conditions and will result in only 42.6 to 47.5% ML. Simultaneous high ML and low SG is the primary focus, hence ML/SG needed to be maximized in this study, and because of conflicting influence of process parameters on ML/SG, the desirability levels are lower. Therefore, it should be noted that optimization results depend on imposed restrictions.

Table 5.4: Optimization conditions at limited constraints

Set	Constraint	Temp. (°C)	Conc. (%)	MD (%)	ML (%)	SG (%)	ML/SG	WR (%)	ΔE	Hardness (g)	Chewiness (g mm)	Desirability
1	Maximize	60	59	15	57.4	6.71	8.98	50.7	11.3	223	83.2	0.845
	ML	60	59	15	57.3	6.72	8.96	50.6	11.3	223	82.9	0.840
		60	58	14	57.2	6.74	8.91	50.5	11.5	222	82.3	0.833
2	Minimize	39	36	14	42.6	4.53	9.40	38.1	11.0	120	42.9	1.000
	SG	50	32	16	46.9	4.42	10.3	42.5	10.0	112	53.2	1.000
		51	32	16	47.5	4.56	10.2	42.9	10.4	110	54.0	1.000
3	Maximize	53	32	16	48.6	4.61	10.3	44.0	10.2	114	57.0	0.776
	ML/SG	53	32	16	48.7	4.62	10.3	44.1	10.2	114	57.3	0.775
		54	32	16	48.9	4.65	10.3	44.3	10.2	115	57.8	0.775
4	Maximize	60	59	15	57.4	6.71	8.98	50.7	11.3	223	83.2	0.842
	WR	60	59	15	57.4	6.70	8.99	50.7	11.2	223	83.1	0.840
		60	58	14	57.2	6.74	8.91	50.5	11.5	222	82.3	0.829
5	Minimize	45	56	16	50.1	5.58	9.25	44.6	9.6	197	67.3	0.969
	ΔE	45	56	16	50.1	5.58	9.24	44.5	9.6	196	67.2	0.968
		45	56	16	50.3	5.61	9.25	44.7	9.6	198	67.8	0.967
6	Maximize	57	58	16	56.1	6.31	9.28	49.8	10.3	226	81.1	0.930
	Hardness	57	58	16	56.1	6.32	9.27	49.8	10.3	226	81.2	0.930
		57	58	16	56.1	6.30	9.30	49.8	10.3	226	81.0	0.930
7	Maximize	60	59	15	57.4	6.71	8.98	50.7	11.3	223	83.2	0.813
	Chewiness	60	59	15	57.4	6.69	8.99	50.7	11.2	223	83.1	0.811
		60	59	15	57.3	6.68	9.00	50.6	11.2	224	83.0	0.810

5.5 Conclusions

A CCRD model with the RSM experimental design was used to evaluate and optimize the effect of individual parameters such as temperature, concentration, and MD content in the osmotic solution on each responses such as mass transfer and quality parameters. As with most osmotic dehydration processes, both temperature and concentration of osmotic solution were found to be significant in influencing all outcome responses. Additionally, the major factor, the driving force for this research, the MD supplement to sugar in the osmotic solution was found to be a highly significant positive factor for the mass transfer parameters ML, WR, and ML/SG. Most importantly, MD incorporation had a negative coefficient or reciprocal influence on SG, which was the significant finding of this study. It was clearly demonstrated that MD incorporation has the ability to restrict solids uptake as well as enhance ML, which in combination helped further enhance the ML/SG ratio, a desirable feature for osmotic dehydration. In addition, the inclusion of MD supplements resulted in better quality products retaining both color and textural attributes. To sum up, when optimizing the process, it was found that the best scenario for carrying out MWODS for mango cubes is to carry it out at 56⁰C, 46% solute concentration and 16% of maltodextrin in sucrose.

The quality analysis was carried out on the intermediate mango product which resulted from the MWODS process and this can be used in different ways for further processing: held refrigerated or frozen for incorporation in baking and cooking applications or finished dried to be used as a dehydrated product.

PREFACE TO CHAPTER 6

In the previous chapters, the application of microwave osmotic dehydration under continuous flow medium spray condition of mango using sucrose and maltodextrin was studied, optimized and evaluated. However, the osmotically pretreated sample needs to be finish-dried to achieve desired final level of moisture content for achieving low enough water activity for shelf-stability. There are several methods utilized for finish-drying process, but hot air drying method is one of the oldest and most widely used technique. The various pretreatments such as osmotic dehydration would have a significant effect on the quality of the finished dried product. These are considered in this chapter. MWODS pretreated samples with sucrose and maltodextrins were subjected to finish-air drying process to evaluate the influence of two solute mixtures along with concentration and temperature of osmotic solution on dried product. A central composite design was used to optimize the process to achieve a high quality product with optimal conditions.

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

Shinde, B. and Ramaswamy, H.S., 2018. Effect of microwave osmotic pre-treatment with sucrose and maltodextrin solute mixture on finished air drying of mangoes by using CCRD. Northeast Agricultural and Biological Engineering Conference (NABEC) 2018. July 15-18, 2018 in Morgantown, WV, USA. (Poster presentation)

CHAPTER 6

Evaluation and Optimization of Finish - Air Drying Quality of MWODS treated mango cubes using maltodextrin moderated sucrose solution

Abstract

The microwave osmotic dehydration of frozen-thawed mango cubes under a continuous flow of maltodextrin moderated sucrose solution spray condition (MWODS) was evaluated based on quality of finish air dried product. Experiments were designed according to a central composite rotatable design to evaluate the effect of maltodextrin moderated sucrose solution (sucrose+maltodextrin (10DE) in 85:15 proportion), on the finish air dried product. The process variables were temperature (30 to 70°C), concentration (30 to 70%), contact time (10 to 50 min) and flow rate (0.8 to 3.8L/min). The optimum processing conditions were determined based on several process and product related quality parameters such as moisture loss (ML), solids gain (SG), weight gain, and ML/SG, color, texture, rehydration capacity (RHC), bulk density and drying time. The MWODS contact time was found to be the most significant contributor in all response parameters followed by temperature. The optimum values found were: temperature, 51.7 °C; solute concentration, 58.5 %; contact time, 30.6 min and solution flow rate of 1.8 L/min. Finally, this optimized processing condition was further used to compare three different solute mixtures [sucrose alone, and sucrose+dextrose, sucrose+maltodextrin (10DE) at 85:15% ratio] to understand the final effect of various solutes on the dried product. Based on the color and textural parameters along with rehydration characteristics of the finished product, the sucrose+maltodextrin mixture was shown to result in the most desirable quality while the air dried product without MWODS pretreatment (control) showed the least. Overall, the results suggested that the sucrose-maltodextrin combination offers quality advantage for MWODS-air drying of mango cubes.

6.1 Introduction

Drying is one of the oldest and most widespread processing operations in the food industry today, which can account for up to 15% of all industry energy usage (Fernandes et al., 2006). It also allows the transformation of high perishable fresh foods into a shelf-stable commodity, lowers storage and transportation costs by reducing weight and eliminating the need for refrigerated storage, and offers the possibility of adding value to processed foods (Orsat et al., 2007). The most frequently used air drying process is considered as the simple process for the preservation of fruits and vegetables, which is widely used. However, the particular concern with air drying is the high quality losses due to degradation of color and flavor (Vega-Gálvez et al., 2009) as well as loss of nutritional profile, effects that are primarily attributed to exposure to high temperatures and long drying times, and shrunken products with tough texture, severe browning and low nutritive value (Deng & Zhao, 2008). Therefore, the key to improve the quality of dried products is to limit changes to the above mentioned quality characteristics during the drying process by employing a pretreatments such as osmotic dehydration.

Several studies have been carried out focusing on reducing the long operational time during conventional OD by giving pre-treatment such as skin removal or puncture, coating methods, pulsed electric field, and high hydrostatic pressure applications (Rodriguez et al., 2016). In addition, application of microwave environment during OD treatment has shown successful results that did not need any pre-treatments before OD (Wray & Ramaswamy, 2013). This novel method was applied originally on apple cubes under immersion mode which was then improved to a spray based system (Azarpazhooh & Ramaswamy, 2009b). The technique, microwave-OD under continuous flow medium spray conditions (MWODS) has been explored in several studies, as well as MWODS combined finished drying (Azarpazhooh & Ramaswamy, 2011; Azarpazhooh & Ramaswamy, 2009b, 2011; Wray & Ramaswamy, 2015b). In all these studies, only sucrose was used as the osmotic solute. The current series of studies are an extension of the above based on further improving the performance of MWODS using solute mixtures as osmotic agents.

The type and mixtures of solute agents have also been considered important during OD, which can directly affect mass transfer as well as finished drying quality characteristics. Therefore, the application of osmotic solutions made from sugar, water, salt, etc., has received

considerable attention (Derossi et al., 2008). On a similar note, the recent studies involving various solute mixtures such as sucrose, dextrose, and maltodextrin have also demonstrated that the application of maltodextrin moderated sucrose solution on MWODS of mangoes promoted the moisture loss and limited the solids uptake when compared with other solutes (Shinde & Ramaswamy, 2019a). Continued research further compared the different grades of maltodextrin, based on their dextrose equivalent values and found out that MD with 10DE had best mass transfer rates among the three grades studied (Shinde & Ramaswamy, 2019b) and finally these were optimized for MWODS (Shinde & Ramaswamy, 2019c). In all these MWODS studies, the quality parameters were assessed immediately after the MWODS treatment which only produced an intermediate product with relatively high residual moisture content with high water activity to be considered as a stable finished product. They can be refrigerated and used as ingredients in bakery products or preserved by freezing or further air dried to make them shelf-stable.

The major focus of this current study was to evaluate the product quality of MWODS mango cubes following finished air drying and to optimize the various parameters using central composite rotatable design. Hence, during this study, the solute mixtures of sucrose and maltodextrin were examined using four-variable constraints such as temperature, concentration, contact time as well as flow rate. The optimized condition was further used to evaluate the effect of three different solute mixture such as sucrose+maltodextrin, sucrose+dextrose and sucrose alone on the quality difference in terms of color, texture, bulk density, rehydration capacity.

6.2 Materials and methods

6.2.1 Raw material

Commercial grade sucrose (Lantic Sugar Ltd., Montreal, Qc, Canada), and maltodextrin (10DE) (Univar Pvt. Ltd, Canada) were used with tap water for preparing the osmotic solution, and the concentrations were maintained on the wet basis (wb). Maltodextrin 10DE was selected because of its better performance as compared to MD15DE and MD18DE (Shinde & Ramaswamy, 2019a, 2019b). Frozen mango pieces were obtained from a local food freezing company (Nature's Touch, Canada) and kept frozen (-21°C) until use. Prior to use, the mango pieces were thawed overnight (8-10 h) in a refrigerator (4°C - 7°C). The dimensions of the mango pieces were in the general shape of a cube with side dimensions of approximately 15 ± 2 mm. The moisture content of the frozen-thawed (untreated) mango cube was measured using AOAC

method (AOAC, 1975), by keeping the cubes in an oven at 105⁰C for approximately 24h (until a constant weight was achieved). The moisture content of mango cube was determined as 86.1% (wb) on average.

6.2.2 Microwave Setup

The MWODS assembly is as explained in Chapter 3.

6.2.3 Osmotic dehydration procedure

The MWODS system was set up and the solution was preheated according to the setpoint temperature (30⁰C-70⁰C) of the run type, as explained in the Table 6.1. Individual samples of 100 ±2 g (12-15 cubes) were weighed and placed in a Nylon mesh bag to hold the samples. The sample was then placed on the acrylic stage in the sample chamber in a single layer. The pump was turned on, and the solution allowed to flow, and then the microwave was turned on. The pump was then terminated after each prescribed contact time (10, 20, 30, 40, 50min) according to the experimental run type. The excess osmotic solution from the surface of the product was removed by shaking the sample 3-4 times and wiping, and the mango cubes were then weighed and were either examined for quality parameters or dried to constant weight in an oven set at 105⁰C for approximately 24 h (AOAC, 1975).

6.2.4 Experimental design

Experimental CCRD design for MWODS was used to estimate the experimental runs under the given variable constraints, as shown in Table 6.1. The solute combination of sucrose+maltodextrin (10DE) was used in sucrose:maltodextrin :: 85:15 proportion. For preparing this, first the appropriate concentration of solute was selected and then the solute portion was adjusted to 85 proportion of sucrose and 15 proportion of maltodextrin. This proportion was adopted from the previous study which showed better mass transfer properties along with high-quality attributes (Shinde & Ramaswamy, 2019a). The CCRD design was used with four variables, namely solute concentration, temperature, contact time and flow rate, at five coded levels (-1.68, -1, 0, +1, +1.68) and each run with three replicates was demonstrated. The design program does not require any duplicates of each run type, as there are six repeated run types (center points) to determine the variability of the process and the lack of fit of the model.

However, the triplicates of each run was performed due to the intrinsic variability of the microwave drying process. Thus, the mean value of triplicates was entered into Design Expert for further analysis.

The concentration (%) and temperature ($^{\circ}\text{C}$) were numerically ranged from 33-70. The concentration range 40-60% and temperature range 40-60 $^{\circ}\text{C}$, have been commonly employed in most osmotic dehydration experiments. Lower limits of concentration are normally set based on limited mass transfer performance rather than higher ones due to high viscosity limiting the flow rates. With temperature again the lower limits were set to 30 $^{\circ}\text{C}$ as any temperature lower than this will limit the mass transfer and higher limits were set to 70 $^{\circ}\text{C}$ as any temperature higher than this might lead to the browning effects. The CCRD design covered this range for the design with 33 and 70 levels as the extreme cases for statistically-based projection purposes. In addition, the overall contact time and flow rate ranges were set between 10-50 min and 0.8 to 3.8 L/min, respectively. The selection of the range of the contact time was adopted from previous studies (Wray & Ramaswamy, 2013). whereas the range of flow rate was chosen depending on the capacity that the digital pump could achieve.

Similarly, the effect of different OD solute mixtures on finished air drying was finally studied using sucrose, sucrose+dextrose (85:15), sucrose+maltodextrin (85:15) and frozen thawed - air dried mangoes (without MWODS) and the results were compared.

6.2.5 Finished air drying

The post-MWODS mangoes were subjected to finish-air drying process to obtain final moisture content of 20% (db). A domestic drying oven (Equi-Flow Food Dehydrator, Marysville, Wash., U.S.A.) was used with a thermostat to maintain the temperature conditions of $60 \pm 1^{\circ}\text{C}$, 0.64 ± 0.02 m/s and relative humidity of approximately 15%. The post-MWODS mangoes were arranged in a single layer on a metal mesh which was suspended from a balance inside the drying chamber. The mangoes were subjected to a constant horizontal airflow inside the hot air-drying chamber. The door of the hot air chamber was quickly opened 4 times during the process to rotate the sample mesh bed about 90 $^{\circ}$ to alternate the side of the sample exposed to the oncoming hot air. The mass of the sample was recorded every 10 min without opening the door.

6.2.6 Dehydration responses

The dehydration parameters moisture loss (ML), solids gain (SG), moisture loss to solids gain ratio (ML/SG), and weight reduction (WR) are well explained in Chapter 3.

Table 6.1: CCRD experimental design for MWODS in real and coded values

Std Order no.	Temp.(⁰ C)	Conc. (%)	Contact time(min)	Flow rate (L/min)
1	40 (-1)	40(-1)	20(-1)	1.5(-1)
2	60 (+1)	40(-1)	20(-1)	1.5(-1)
3	40(-1)	60(+1)	20(-1)	1.5(-1)
4	60(+1)	60(+1)	20(-1)	1.5(-1)
5	40(-1)	40(-1)	40(+1)	1.5(-1)
6	60(+1)	40(-1)	40(+1)	1.5(-1)
7	40(-1)	60(+1)	40(+1)	1.5 (-1)
8	60(+1)	60(+1)	40(+1)	1.5(-1)
9	40(-1)	40(-1)	20(-1)	3.0(+1)
10	60(+1)	40(-1)	20(-1)	3.0(+1)
11	40(-1)	60(+1)	20(-1)	3.0(+1)
12	60(+1)	60(+1)	20(-1)	3.0(+1)
13	40(-1)	40(-1)	40(+1)	3.0(+1)
14	60(+1)	40(-1)	40(+1)	3.0(+1)
15	40(-1)	60(+1)	40(+1)	3.0(+1)
16	60(+1)	60(+1)	40(+1)	3.0(+1)
17	30(-1.68)	50(0)	30(0)	2.3(0)
18	70(+1.68)	50(0)	30(0)	2.3(0)
19	50(0)	30(-1.68)	30(0)	2.3(0)
20	50(0)	70(+1.68)	30(0)	2.3(0)
21	50(0)	50(0)	10(-1.68)	2.3(0)
22	50(0)	50(0)	50(+1.68)	2.3(0)
23	50(0)	50(0)	30(0)	0.8(-1.68)
24	50(0)	50(0)	30(0)	3.8(+1.68)
25	50(0)	50(0)	30(0)	2.3(0)
26	50(0)	50(0)	30(0)	2.3(0)
27	50(0)	50(0)	30(0)	2.3(0)
28	50(0)	50(0)	30(0)	2.3(0)
29	50(0)	50(0)	30(0)	2.3(0)
30	50(0)	50(0)	30(0)	2.3(0)

6.2.7 Rehydration capacity

The dried samples were accurately weighed and soaked in excess distilled water for 14 h at room temperature (23⁰C). The ratio of sample to distilled water was maintained around 1:25. Then they were taken out and placed on a filter paper further placed under a slight suction for 1 min to remove the surface moisture before weighing the samples. Rehydration capacity (RHC) was determined in triplicate using the procedure described by Azarpazhooh and Ramaswamy (2011) (Azarpazhooh & Ramaswamy, 2011). The rehydration capacity was then determined according to equation 5:

$$\text{Rehydration capacity} = \frac{W_r - W_d}{W_d} \quad (5)$$

Where, W_r and W_d are the masses of the rehydrated and dry material (g), respectively.

6.2.8 Bulk density

Bulk density is an important parameter from the perspective of easy transportation and space-saving properties which can be defined as, the ability of the dried product to be stored in minimum space area with maximum mass balance. This attributed that the bulk density should be maximum for finished dried products. (Rahman, 2005). The bulk density was determined in triplicates by weighing the dried mangoes and their volume and expressed in kg/m³.

6.2.9 Quality analysis

Separate sample runs were made for each experimental run type (temp./conc./flow rate/time combinations), under the same conditions for quality analysis and measured the quality parameters as elaborated below. The quality of each experimental run type was examined after the finished drying process.

6.2.9.1 Texture evaluation

TA.XT Plus Texture Analyzer (Stable Microsystems, Surrey, UK) was used for texture profile analysis (TPA) of finished air dried samples after MWODS treatments. Two-cycle compression test was performed to obtain TPA, using a flat bottom probe of 25 mm diameter, with a pretest speed of 5 mm/sec, the test speed of 5 mm s⁻¹ and post-test speed of 5 mm s⁻¹. The target compression was about a distance of 3 mm into the sample during two consecutive cycles

to target a 25% deformation from the average height of samples. These settings with minor modifications were used with guidance from Banjongsinsiri et al. (2004), who used TPA on mango cubes (Banjongsinsiri, 2004). The analysis was performed with six replicates, and the average values (with standard deviation) were used. A wide range of responses such as hardness, chewiness, adhesiveness, cohesiveness, factorability, gumminess, and springiness can be obtained from TPA analysis (Bourne, 2002). Hardness and chewiness were selected in this study as parameters to determine the texture influence of osmotically processed mango cubes. The peak force defined hardness during the first compression cycle, and chewiness was obtained from the product of gumminess and springiness (Bourne, 2002). The chewiness was calculated using Equation 6.1.

$$\text{Chewiness} = \text{Gumminess} \times \text{Springiness} \quad (6.1)$$

6.2.9.2 Color

The color of MWODS-air dried samples was analyzed in the L^* , a^* , b^* system using a tristimulus Minolta Chroma Meter (Minolta Corp., Ramsey, NJ, USA). The Chroma Meter was warmed up 20 min prior to use, and the color was calibrated against a white standard. Six measurements were made with each sample, and the values were averaged to obtain the L^* (lightness), a^* (green (−) to red (+)), and b^* (blue (−) to yellow (+)) values of the individual trials. The ΔE (total color change) was also measured where the total color change of processed samples was determined in comparison with the color of freeze-dried mangoes (without MWODS pretreatment). Since many studies have concluded that freeze-dried products can produce highly acceptable quality of the dried product (Wray & Ramaswamy, 2015d). Hence for this study the freeze-dried mangoes were chosen as a control sample to measure the color change of dried mangoes. It can be measured using the following equation 6 (Maftoonazad & Ramaswamy, 2008).

$$\Delta E = \sqrt{(L_0 - L)^2(a_0 - a)^2(b_0 - b)^2} \quad (6.2)$$

6.2.10 Effect of solute mixtures

The optimized conditions based on the desirability approach were employed for further study of comparison of different solute mixtures. In this approach, the mangoes pre-treated with MWODS using the solute mixture such as sucrose+maltodextrin and sucrose+dextrose in 85:15

proportion, as well as sucrose (100%) were subjected for finished air-drying process and the results of quality characteristics along with rehydration capacity (RHC) and bulk density were compared. All of the MWODS pretreated samples with different solute mixtures were compared with fresh air-dried mangoes without pretreatment.

6.2.11 Statistical analysis

The statistical analysis using JMP[®] v-13 (SAS Institute Inc., Cary, NC., U.S.A) was carried out to understand the difference between each solute mixture on the quality of dried mangoes. All the experiments were performed in four replicates and the mean values were compared to a 95% confidence interval using the analysis of variance function with Tukey groupings to understand if the compared values were significantly different.

6.3 Results and discussions

6.3.1 Response surface methodology

The response surface methodology was applied using composite rotatable design to obtain minimum experimental runs, which can give an optimal result using analysis of variance (ANOVA). The responses gathered from CCRD can be used for optimization using statistical models and parametric performances can be compared using ANOVA. The results obtained from the design are presented in Table 6.2. Table 6.3 summarizes the selection of a model for each response based on their statistical significance as well as the lack of fit values. The quadratic model ($p < 0.05$) was selected for ML, SG, WR, ML/SG, hardness, L^* , b^* , E, RHC, and bulk density responses. Whereas a^* , chewiness and drying time were best represented by a linear model ($p < 0.05$). The lack of fit values for each selected model was not significant ($p > 0.05$), as shown in Table 6.3. An insignificant lack of fit explains that the selected model can predict the responses using the predicted equations, as a function of input variables.

Table 6.2: CCRD run numbers with results for mass transfer, texture and color parameters, RHC and bulk density

Std Orde r no.	ML (%)	SG (%)	ML/SG	WR (%)	Hardness (N)	Chewiness (N mm)	L*	a*	b*	Δ E	RHC (%)	BD (Kg/m ³)	DT (min)
1	32.0(±3.8)	4.65(±1.1)	7.18(±2.5)	27.4(±4.9)	91(±5.7)	44.0(±2.4)	21.1(±1.4)	11.6(±1.3)	21.8(±1.4)	24.6(±1.4)	89.3(±4.2)	387(±21)	960(±28)
2	39.3(±1.4)	6.04(±0.5)	6.54(±0.7)	33.3(±1.9)	98(±5.2)	36.7(±2.1)	19.1(±1.5)	10.7(±1.0)	19.8(±1.9)	23.9(±2.4)	72.2(±2.9)	380(±24)	870(±21)
3	34.1(±1.1)	5.70(±0.3)	5.99(±0.5)	28.4(±1.3)	90(±4.9)	52.8(±2.2)	20.1(±1.6)	11.1(±1.0)	29.1(±2.1)	21.8(±1.5)	84.4(±3.6)	385(±26)	950(±20)
4	41.0(±1.6)	7.11(±0.8)	5.78(±0.4)	33.8(±0.8)	120(±7.1)	47.3(±2.0)	18.3(±1.5)	9.50(±1.5)	21.6(±1.6)	18.6(±2.0)	88.2(±3.9)	379(±22)	720(±17)
5	44.2(±2.2)	6.33(±0.4)	7.01(±0.8)	37.9(±2.6)	125(±7.7)	47.3(±3.4)	19.2(±1.9)	10.9(±1.2)	27.4(±2.0)	26.1(±2.6)	80.1(±3.5)	365(±18)	800(±22)
6	56.5(±2.2)	7.98(±0.4)	7.10(±0.7)	48.5(±2.7)	128(±7.9)	38.1(±1.9)	17.3(±1.8)	10.7(±1.3)	20.1(±1.9)	29.7(±1.2)	75.4(±3.2)	349(±15)	650(±10)
7	51.5(±1.6)	7.42(±1.5)	7.06(±1.2)	44.1(±0.1)	130(±9.1)	55.2(±3.5)	18.1(±1.9)	10.0(±1.0)	24.4(±1.8)	20.2(±1.7)	90.1(±4.6)	357(±17)	690(±12)
8	58.8(±0.9)	9.73(±0.4)	6.04(±0.2)	49.0(±0.5)	137(±10)	49.6(±3.2)	16.1(±1.7)	8.60(±1.0)	15.4(±1.8)	22.5(±1.3)	81.5(±3.3)	341(±16)	438(±8.0)
9	28.9(±0.9)	6.12(±0.4)	4.72(±0.1)	22.7(±0.7)	112(±6.8)	49.9(±2.2)	22.0(±1.9)	9.80(±1.5)	21.1(±1.8)	21.5(±2.5)	79.1(±3.5)	385(±24)	858(±19)
10	32.8(±0.8)	6.99(±1.2)	4.77(±0.9)	25.8(±1.9)	119(±7.4)	44.9(±2.9)	20.5(±1.9)	9.10(±1.2)	22.4(±1.8)	21.4(±1.8)	91.3(±4.5)	380(±21)	720(±16)
11	30.8(±1.7)	6.47(±1.2)	4.86(±1.1)	24.3(±2.8)	125(±7.8)	58.9(±3.1)	21.0(±1.9)	9.40(±1.1)	22.1(±1.8)	21.6(±0.8)	100(±6.9)	381(±19)	810(±16)
12	40.7(±1.7)	7.40(±1.5)	5.64(±1.4)	33.3(±3.2)	133(±8.9)	52.7(±2.1)	20.3(±1.6)	9.70(±1.3)	23.3(±1.8)	21.6(±2.2)	105(±6.6)	376(±17)	710(±13)
13	38.4(±2.5)	6.98(±1.1)	5.60(±1.2)	31.5(±3.6)	141(±13)	52.4(±2.5)	21.1(±1.9)	9.90(±0.9)	26.9(±2.2)	23.5(±2.8)	77.4(±3.3)	379(±19)	660(±10)
14	41.8(±1.9)	8.10(±0.9)	5.18(±0.3)	33.7(±1.0)	144(±11)	47.5(±1.9)	19.4(±1.8)	7.70(±1.4)	21.6(±1.9)	26.4(±2.4)	86.1(±3.5)	377(±20)	528(±10)
15	40.9(±0.7)	7.89(±0.8)	5.21(±0.4)	33.0(±0.1)	149(±14)	61.2(±3.2)	19.9(±1.7)	8.50(±1.2)	20.4(±1.4)	18.7(±1.7)	99.1(±4.2)	376(±18)	540(±8.0)
16	50.0(±1.9)	9.86(±0.5)	5.08(±0.5)	40.1(±2.4)	151(±16)	56.3(±2.1)	18.1(±1.9)	7.20(±1.3)	18.2(±1.7)	23.2(±0.8)	106(±6.1)	362(±15)	432(±7.0)
17	36.2(±0.8)	6.95(±0.3)	5.21(±0.4)	29.2(±1.1)	103(±6.1)	51.8(±2.5)	22.4(±1.7)	10.2(±0.8)	22.2(±1.7)	20.5(±1.5)	101(±5.3)	373(±12)	800(±20)
18	58.8(±2.5)	10.8(±0.9)	5.46(±0.7)	48.0(±3.5)	133(±8.9)	45.7(±2.8)	18.0(±1.8)	9.20(±1.7)	19.3(±1.9)	25.6(±2.5)	101(±5.5)	350(±13)	490(±10)
19	35.4(±2.2)	5.36(±0.7)	6.62(±0.3)	30.0(±2.0)	99(±6.9)	41.7(±2.1)	20.0(±1.7)	10.1(±1.4)	19.0(±1.9)	24.4(±2.5)	100(±5.2)	380(±24)	850(±22)
20	52.1(±2.1)	8.06(±0.1)	6.47(±0.3)	44.0(±2.2)	135(±9.6)	55.8(±3.2)	16.4(±1.7)	9.70(±1.7)	18.5(±1.9)	18.2(±0.6)	110(±6.5)	358(±17)	622(±11)
21	22.6(±2.1)	4.51(±0.5)	5.01(±0.1)	18.1(±1.6)	80.2(±5.2)	43.9(±1.9)	24.2(±1.6)	11.4(±1.5)	28.3(±1.5)	14.5(±1.9)	81.4(±3.4)	380(±24)	980(±24)
22	48.3(±0.7)	9.13(±1.4)	5.35(±0.8)	39.2(±0.7)	117(±8.0)	50.1(±2.5)	15.4(±1.7)	9.30(±1.4)	22.9(±1.9)	23.1(±2.3)	71.2(±3.2)	375(±21)	360(±9.0)
23	54.6(±1.6)	6.77(±0.8)	8.10(±0.7)	47.9(±0.9)	80.4(±4.5)	40.8(±1.7)	19.4(±1.9)	10.3(±1.2)	21.1(±1.9)	23.5(±1.8)	53.5(±2.1)	352(±14)	750(±15)
24	39.7(±1.0)	8.29(±0.9)	4.81(±0.4)	31.4(±0.2)	118(±5.4)	50.2(±2.8)	20.0(±1.7)	9.00(±1.3)	18.4(±2.0)	24.8(±2.0)	61.2(±2.9)	377(±19)	542(±8.0)
25	38.8(±2.1)	7.45(±0.2)	5.21(±0.4)	31.3(±2.3)	71.1(±3.9)	41.4(±2.1)	17.5(±1.9)	9.10(±1.4)	19.3(±2.0)	26.0(±3.0)	91.4(±4.0)	378(±17)	674(±12)
26	37.5(±1.2)	7.91(±0.2)	4.75(±0.3)	29.6(±1.5)	65.5(±3.5)	43.8(±1.9)	16.8(±1.8)	9.90(±1.2)	19.9(±1.7)	26.1(±2.4)	100(±5.6)	383(±20)	645(±10)
27	36.7(±1.8)	8.10(±0.7)	4.55(±0.6)	28.6(±2.5)	75.2(±3.7)	44.5(±2.1)	17.9(±1.8)	8.91(±1.4)	20.2(±1.6)	25.1(±1.8)	97.3(±4.7)	387(±19)	690(±14)
28	36.9(±2.7)	7.11(±0.3)	5.18(±0.2)	29.8(±2.4)	70.4(±4.0)	48.9(±2.0)	18.0(±1.6)	10.1(±1.7)	18.4(±1.8)	26.3(±1.4)	108(±6.8)	382(±19)	660(±12)
29	39.5(±1.8)	7.04(±0.5)	5.59(±0.7)	32.4(±2.4)	61.4(±3.4)	42.8(±2.4)	16.5(±1.9)	8.90(±1.2)	18.6(±1.9)	27.2(±1.5)	105(±5.8)	378(±21)	690(±15)
30	40.0(±1.9)	6.92(±0.3)	5.79(±0.5)	33.1(±2.2)	55.0(±3.1)	44.4(±1.9)	17.7(±1.7)	9.50(±1.5)	20.6(±1.6)	25.0(±1.3)	103(±5.1)	375(±16)	630(±12)
FF							36.8(±3.4)	9.91(±2.0)	37.1(±3.1)				

FF: Frozen thawed and freeze dried

Table 6.3: Predicting equations and compiled ANOVA results for CCRD responses

Responses	Model	Predicting equations in terms of actual variables	Lack of fit	R ²
ML	Quadratic	ML= +38.2+ 4.39 *T+ 2.80 *C+ 6.41 *t- 3.45 *F+1.97*T ² +1.03*C ² - 1.05*t ² +1.88*F ² +0.39*T*C+0.26*T*t-0.47*T*F+0.42*C*t+0.44*C*F-1.67*t*F	0.0506 (NS)	0.9568
SG	Quadratic	SG = +7.42+ 0.81 *T+ 0.57 *C+ 0.96 *t+0.33*F+0.31*T ² -0.23*C ² -0.20*t ² - 0.03*F ² +0.10*T*C+0.15*T*t-0.12*T*F+0.16*C*t-0.10*C*F-0.13*t*F	0.7147 (NS)	0.9518
ML/SG	Quadratic	ML/SG =+6.68+0.02*T-0.07*C+0.36*t- 0.61 *F-0.27*T ² -0.03*C ² -0.22*t ² - 0.21*F ² +0.03*T*C-0.09*T*t+0.14*T*F+0.11*C*t+0.36*C*F+0.10*t*F	0.6945 (NS)	0.8717
WR	Quadratic	WR =+30.8+ 3.57 *T+2.21*C+ 5.47 *t- 3.79 *F+1.65*T ² +1.26*C ² - 0.85*t ² +1.91*F ² +0.29*T*C+0.09*T*t-0.34*T*F+0.25*C*t+0.55*C*F-1.52*t*F	0.1497 (NS)	0.9483
L*	Quadratic	L*=+17.4-0.93*T-0.63*C-1.28*t+ 0.59 *F+0.70*T ² +0.20*C ² +0.60*t ² +0.58*F ² +0.05*T*C- 0.09*T*t+0.13*T*F-0.11*C*t+0.03*C*F+0.16*t*F	0.0802 (NS)	0.8681
a*	Linear	a*=+9.67-0.41*T-0.30*C-0.49*t-0.60*F	0.4035 (NS)	0.7070
b*	Quadratic	b*=+19.5-1.53*T-0.32*C-0.73*t-0.38*F+0.48*T ² -0.01*C ² + 1.69 *t ² +0.24*F ² -0.27*T*C- 1.06*T*t+1.31*T*F-1.78*C*t-0.59*C*F+0.19*t*F	0.0714 (NS)	0.8819
ΔE	Quadratic	ΔE = +26.0+0.81*T-1.72*C+ 1.35 *t-0.29*F-0.56*T ² -0.99*C ² - 1.62 *t ² -0.28*F ² - 0.13*T*C+1.08*T*t+0.33*T*F-0.83*C*t+0.84*C*F-0.24*t*F	0.0866 (NS)	0.9078
Hardness	Quadratic	Hardness=+66.8+5.29*T+6.21*C+12.1*t+9.63*F+ 15.5 *T ² + 15.3 *C ² +10.7*t ² +10.8*F ² +1. 69*T*C-2.31*T*t-1.69*T*F-1.19*C*t+0.44*C*F-1.56*t*F	0.0541 (NS)	0.9146
Chewiness	Linear	Chewiness =+48.0-2.52*T+ 4.24 *C+1.35*t+ 3.00 *F	0.3938 (NS)	0.8028
RHC	Quadratic	RHC =+100+0.25*T+ 5.14 *C-1.38*t+ 4.09 *F+0.51*T ² +1.55*C ² - 5.73 *t ² - 10.4 *F ² +0.56*T*C-0.14*T*t+3.71*T*F+0.56*C*t+3.14*C*F-0.04*t*F	0.6120 (NS)	0.9127
Bulk density	Quadratic	BD =+380-4.88*T-3.71*C-6.54*t+5.12*F-4.03*T ² -2.16*C ² -0.03*t ² -3.28*F ² -0.69*T*C- 1.56*T*t+1.19*T*F-1.44*C*t-0.44*C*F+5.69*t*F	0.1275 (NS)	0.8608
Drying time	Linear	Drying time =+690- 75.8 *T- 50.5 *C- 129 *t- 51.5 *F	0.0537 (NS)	0.9229

Where T is Temperature (°C), C is Concentration (%), t is contact time (min) and F is flow rate (L/min). Note that highly significant (p < 0.0001) models and variables are in bold, significant (p < 0.05) are normal type, while non-significant (p > 0.05) factors are italicized.

6.3.2 Temperature

Temperature was found to be a significant ($p < 0.05$) affecting mass transfer parameters such as ML, SG ($p < 0.0001$) and WR as indicated by the coefficients represented in the coded model (Table 6.3). The positive coefficients of temperature indicated that the temperature was positively correlated with the ML, SG, and WR, which means an increase in the temperature would increase these parameters, as shown in Figure 6.1. The contribution of temperature in water reduction and so as in weight reduction has been demonstrated in earlier reports and reasoned that the high temperature increases the viscosity thereby improving the mobility of water, allowing better contact between OD solution and mango samples and ultimately improves the extraction ability of the OD solution (Azarpazhooh & Ramaswamy, 2009b; Li & Ramaswamy, 2006a; Wray & Ramaswamy, 2015b). The trend also showed that even though the contact time was the highest significant factor in ML, the temperature would also increase the ML to their maximum values as per the results, shown in Table 6.3. It also has been proven that increased temperature attributed elevated SG (Azarpazhooh & Ramaswamy, 2011; Wray & Ramaswamy, 2015b). In contrast, the temperature was found to be a non-significant factor for ML/SG, which is somewhat different from previous works. The reason behind non-significance is unclear. However, an increased number of experiments might produce noise during model analysis. Hence reduced experimental runs would be adequate to understand the individual effect of temperature on ML/SG response. The similar observations were made with rehydration capacity (RHC) where the temperature was found to be a non-significant ($p > 0.05$), as the RHC is largely associated with finished drying method and its parameters. The effect of each processing variable on RHC is presented in Figure 6.2. Since the traditional air drying method mostly alters the structural properties of the product which leads to changes in the rehydration capacity of the dried product.

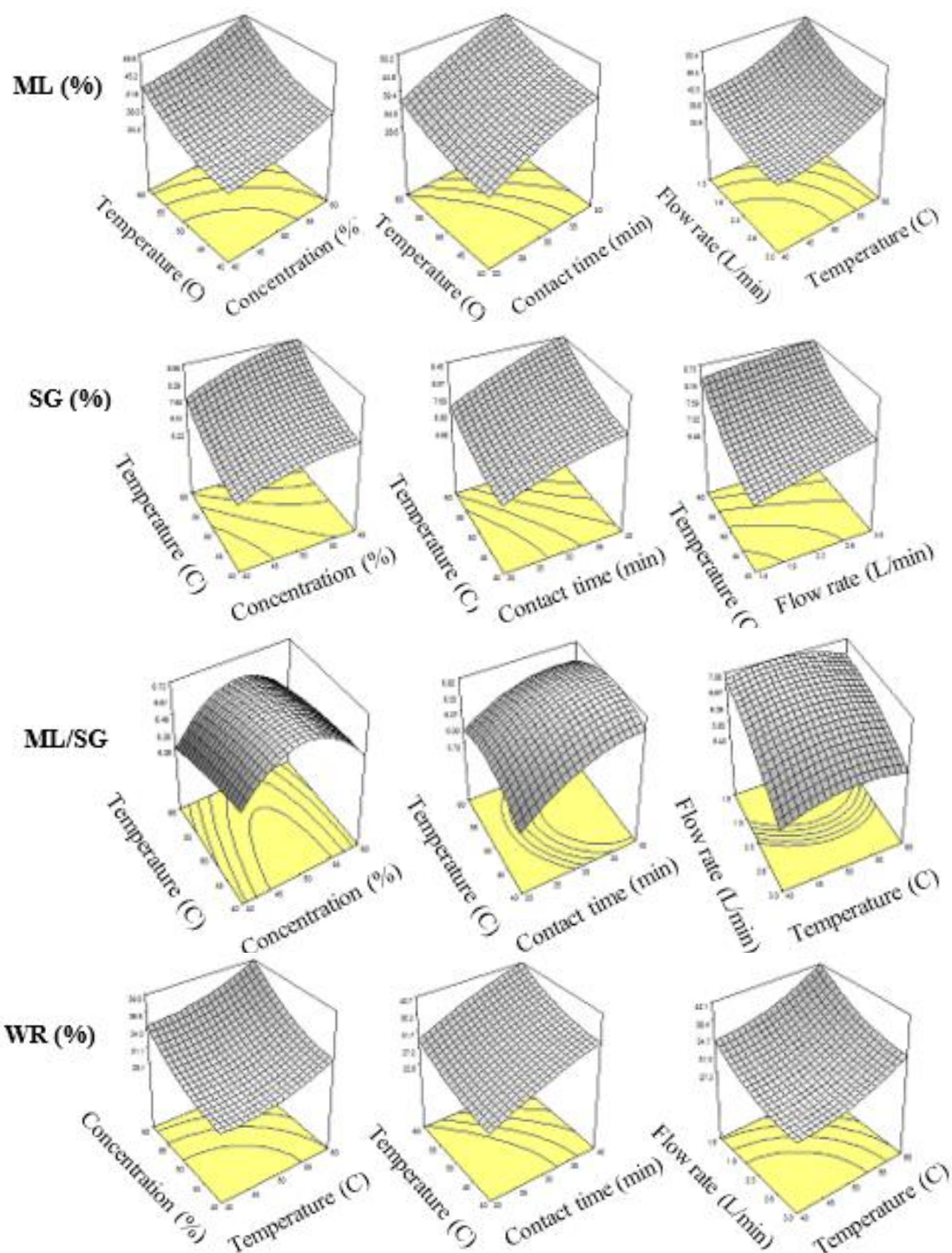


Figure 6.1: Response surface plots for mass transfer for ML, SG, ML/SG, WR. (When not a variable, inputs were fixed at their center point: Conc. 50%, contact time 30min, flow rate 2.3 L/min)

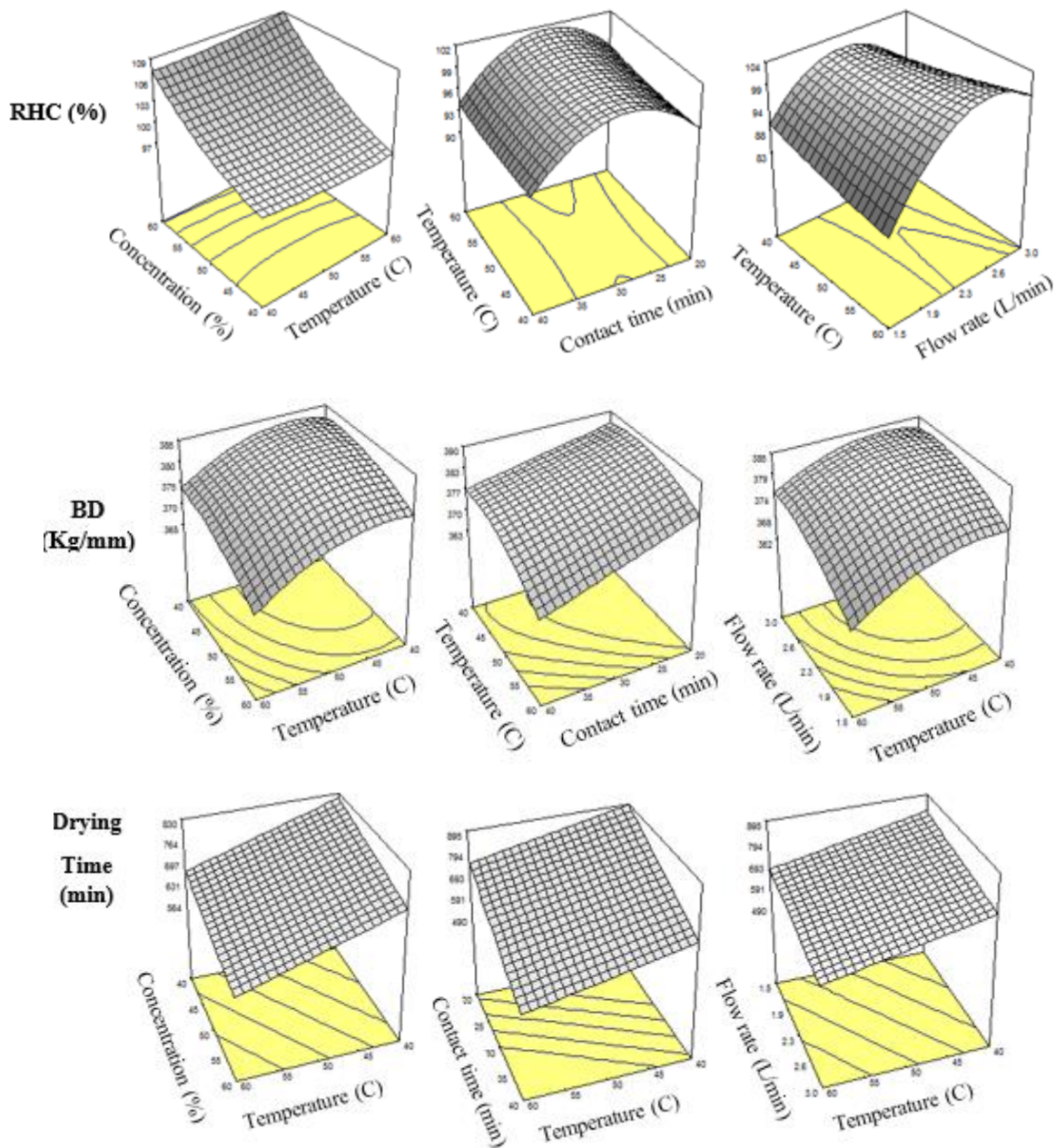


Figure 6.2: Response surface plots for texture analysis for hardness, chewiness, rehydration capacity, bulk density and water activity. (When not a variable, inputs were fixed at their center point: Conc. 50%, contact time 30min, flow rate 2.3 L/min)

The bulk density of dried mangoes was found to be affected negatively by temperature, as shown in Figure 6.2. This might be due to the presence of microwave radiations during the osmotic process which can cause the puffiness in the product. Since the puffiness of the samples was due to open cellular structure, which is likely to be attributed by the high temperature for

long run (Wray & Ramaswamy, 2015b) and hence an increase in temperature have shown reduced bulk density. Similarly, the negative coefficient of the factor temperature indicated that the finished drying time was negatively correlated with the temperature. This indicates that an increase in temperature will reduce the finished drying time. As proven earlier, the higher temperature contributed to higher moisture loss during OD treatment, resulting in lower moisture content products for air drying and ultimately requiring lower drying time for second stage air drying process (Azarpahzoooh & Ramaswamy, 2011).

The quality parameters were also found to be affected by temperature as shown in Figures 6.3 and 6.4. Regardless of the fact that MWODS samples were generally softer than the fresh samples, the air dried finished product was found to be harder than fresh samples, which was also observed in prior studies (Wray & Ramaswamy, 2015b). The hardness was positively correlated with temperature which indicates that increased temperature will increase the hardness of the product. The response surface plots indicating the effect of process variables on hardness are presented in Figure 6.3. Whereas, the negative coefficient of temperature indicated that it had a negative effect on chewiness. Since the higher temperature is responsible for destruction of cell bonds and ultimately changes the mechanical behavior. Also, water reduction at high temperatures during OD can result in the detachment of middle lamella of plant cells in the loss of cell turgor which in turn affects the cell wall and puncture strength (Chiralt & Talens, 2005; Wray & Ramaswamy, 2013) and reduces chewiness of the product.

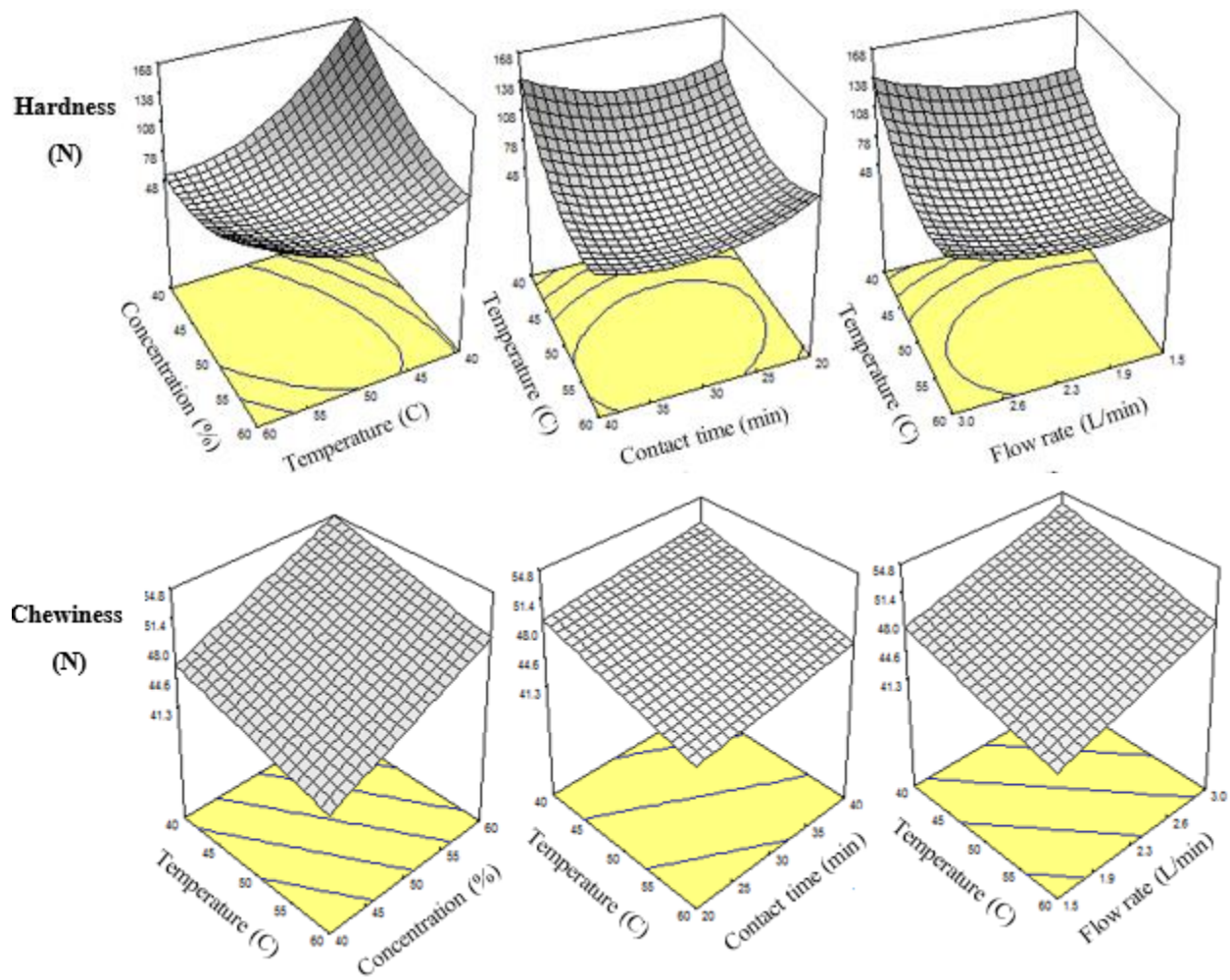


Figure 6.3: Response surface plots for texture analysis for hardness and chewiness (when not a variable, inputs were fixed at their center point: Conc. 50%, contact time 30min, flow rate 2.3 L/min)

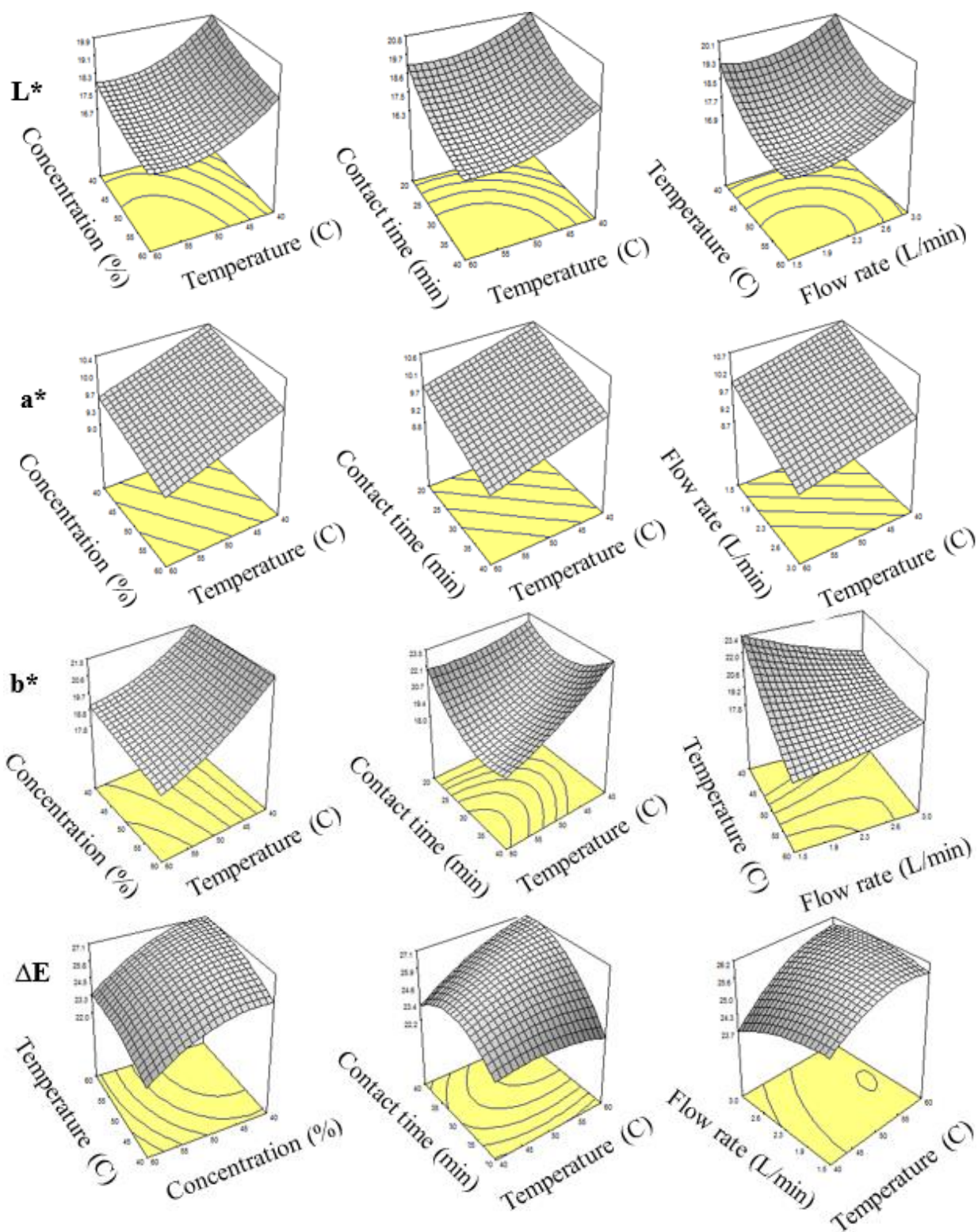


Figure 6.4: Response surface plots for texture analysis for color measurements such as L*, a*, b*, ΔE (when not a variable, inputs were fixed at their center point: Conc. 50%, contact time 30min, flow rate 2.3 L/min)

For color parameters, the temperature had a negative effect on L^* value which can be observed from the coefficients of each term presented in Table 6.3. This is largely attributed to an increase in darkness (low L^* value) of the samples with increased temperature. Moreover, the substantial darkening is likely due to the browning phenomenon. A similar effect was confirmed by Wray and Ramaswamy (2015), where they notified that temperature, contact time and flow rate had negative effect on lightness (L^*) of the samples (Wray & Ramaswamy, 2015b). Similarly, based on ANOVA analysis the significant ($p < 0.05$) factors such as temperature and contact time had negative effect on b^* values which can be observed in Table 6.3. The b^* values are likely related to the loss of the brighter yellow color (positive b^* value) to the darker blue (negative b^* value), which indicates that the temperature and contact time have deleterious effect on yellow color pigments in the samples.

6.3.3 Concentration

The solute concentration was also found to be a highly significant ($p < 0.0001$) factor for ML and SG and significant for WR, with a positive coefficient, which attributed that any increase in concentration determines higher ML and SG. This fact was also proven in previous studies (Shinde & Ramaswamy, 2019c) that an increase in solute mixture S:MD gives better moisture loss (Shinde & Ramaswamy, 2019a), given that the higher molecular MD has difficulties in impregnating into the samples and hence it creates a barrier on the surface of the mangoes and increases the osmotic potential which ultimately targets on moisture removal of fruit samples, and ultimately increases the WR (Azuara et al., 2002). Since the overall weight reduction is the combined effect of the moisture loss and solids gain (Azarpazhooh & Ramaswamy, 2009b; Li & Ramaswamy, 2006b; Van Nieuwenhuijzen et al., 2001), therefore both ML and WR follows a similar trend. Whereas, an increased concentration promotes higher SG which was proven in many studies (Azarpazhooh & Ramaswamy, 2011; Wray & Ramaswamy, 2015b).

However, upon comparing the results, it was found that the highest SG of 9.6 was observed in this study at 60°C-60%-40min-3L/min processing condition which was lower than the prior work of MWODS with cranberries using sucrose only solution, and it was 14.6% of SG at 60°C-60%-45min-3.7L/min (Wray & Ramaswamy, 2015b). The major difference between these studies was the selection of fruit and the solute mixture of sucrose and maltodextrin

combination. As stated in prior work (Shinde & Ramaswamy, 2019a) and proven in previous research studies that the solute MD restricts the entry of solids inside the fruit sample and hence gave minimum solids gain value (Dimakopoulou-Papazoglou & Katsanidis, 2017; İspir & Toğrul, 2009b; Shi et al., 2009). This confirms the earlier discussion that, higher molecular weight solutes such as maltodextrins will form a coating on the surface of the fruit pieces and restrict the intake of osmotic solids into the mango samples. It was also reported by Hawkes and Flink (1978) that the solids uptake is inversely correlated with the molecule size of the osmotic agent (Hawkes & Flink, 1978). However, concentration was found to be non significant factor in ML/SG, which is unclear. The reduction on the experimental runs might be effective to understand the effect of each variable on ML/SG.

The concentration was found to be one of the significant factor with positive coefficient for RHC. The higher concentration of MWODS exhibited higher RHC, which is in disagreement with the prior findings with cranberries (Wray & Ramaswamy, 2015b). As discussed before, the higher molecular MD content in solute mixtures restricts the solids uptake and while enhancing the water molecules to diffuse. Hence it maintained the void space in the plant tissue to some extent and therefore the rehydration capacity increased with increase in the concentration level. From the ANOVA shown in Table 6.3, it was observed that the concentration was negatively correlated with BD and drying time. As discussed, and proven earlier, an increased concentration during MWOD increases the ML and hence reduces the finished drying time (Azarpahzoooh & Ramaswamy, 2011).

For quality parameters, the concentration was found to have a positive coefficient with hardness and chewiness, and also it was found to be the largest contributor to chewiness based on sum of squares (not shown) determined through ANOVA as shown in Table 6.3. It has been proven in previous studies that an elevated concentration along with contact time tended to increase the solids uptake which ultimately results in a chewier product (Monsalve-Gonzalez, 1993; Wray & Ramaswamy, 2013). From the ANOVA analysis (Table 6.3), it was observed that the concentration was the largest contributor to ΔE value. A similar factor was found to be most effective in total color change while studying MWODS with cranberries and apple cylinders (Azarpahzoooh & Ramaswamy, 2012b; Wray & Ramaswamy, 2015b). The total color change was

mostly due to the color degradation during finished air-drying process and color change due to the browning effect of the MWODS process.

6.3.4 Contact time

The contact time was found to be the most significant ($p < 0.0001$) variable affecting ML and SG depending upon their respective coefficients, as shown in Table 6.3. The major cause of massive moisture reduction at higher contact time is the rapid heating of water molecules in the presence of microwaves which increases the internal pressure and promotes quick removal of a water molecule from the sample (Azarpazhooh & Ramaswamy, 2009b). Whereas, increased SG with contact time is a commonly accepted notion and earlier proven in MWODS studies (Azarpazhooh & Ramaswamy, 2011; Wray & Ramaswamy, 2015b). In addition, the contact time also had a substantial influence on WR, which is positively correlated. As discussed before, it follows a similar trend to that of ML which was also proven earlier work (Azarpazhooh & Ramaswamy, 2009b; Li & Ramaswamy, 2006b; Van Nieuwenhuijzen et al., 2001). While discussing ML/SG response, it was observed that the contact time had significant effect on ML/SG with positive coefficient. This indicated that at increased contact time there will be increased ML/SG ratio. Since the MWODS process produces higher ML than SG and hence the ratio of ML/SG was found to be higher at elevated contact time (Shinde & Ramaswamy, 2019a).

The non-significant term contact time would result in low impact on RHC, which is mostly associated with the finished dried product characteristics. However, the contact time was found to be a significant factor for BD with a negative coefficient. As explained earlier the longest exposure to microwave radiations resulted in puffiness of the product and reduces the BD (Wray & Ramaswamy, 2015b). For drying time, the contact time was found to be a significant ($p < 0.05$) model term with negative coefficient, which indicates that an increase in contact time will reduce the finished drying time. The highest coefficient of contact time as shown in Table 6.3 indicated that the contact time was the largest contributor to drying time, which minimizes drying time upon an increase in MWODS contact time, as proven and discussed earlier (Azarpazhooh & Ramaswamy, 2011).

Based on the results of ANOVA (Table 6.3), it was found that contact time was the most significant factor and the largest contributor to hardness. A similar outcome was also recorded in

prior studies (Azarpazhooh & Ramaswamy, 2012b; Wray & Ramaswamy, 2015b). Similarly, the contact time had positive coefficient, which was due to the increased solids gain at higher contact times, which promotes the chewer product (Monsalve-Gonzalez, 1993; Wray & Ramaswamy, 2013). Looking at an individual color parameter, contact time had a negative effect on L^* , a^* and b^* value which was largely attributed to increased darkness (low L^* value) and loss of the brighter yellow color (positive b^* value) at elevated contact time. The increase in the darkness was most likely due to the browning phenomenon, reduced carotenoid and charring during finish drying process. A similar effect was confirmed by Wray and Ramaswamy (2015b). For total color change in finished dried products, the factor contact time was found to a highly significant factor. The color change was due to the color degradation at elevated contact time. A similar factor was found to be most effective in total color change while studying MWODS with cranberries and apple cylinders (Azarpazhooh & Ramaswamy, 2012b; Wray & Ramaswamy, 2015b).

6.3.5 Flow rate

The flow rate had a negative coefficient for ML, WR and ML/SG. The possible explanation of this cause is, the flow rate absorbs a part of microwave radiation, which further reduces the ML and ultimately, affects the WR and ML/SG ratio. Hence the overall performance and the trend of these variables agreed to the prior reports (Li & Ramaswamy, 2006a; Wray & Ramaswamy, 2015b). Whereas the flow rate was found as a significant ($p < 0.05$) variable with positive coefficient, affecting SG. A similar trend was observed in earlier research work (Azarpazhooh & Ramaswamy, 2011; Wray & Ramaswamy, 2015b) with, solids uptake increased with an increase in temperature, concentration, contact time, and flow rate. The flow rate was found to be significant term affecting RHC of finish dried products. whereas, flow rate had a positive coefficient with RHC based on ANOVA as shown in Table 6.3. On the other hand, an increased flow rate might give a slight cooling effect to the samples inside the microwave cavity along with protective maltodextrin solute helps to reduce the cell destruction caused due to high temperature and ultimately produce better RHC product. Therefore, the term flow rate was found to be significant term unlike previous studies (Azarpazhooh & Ramaswamy, 2012a; Wray & Ramaswamy, 2015b).

The flow rate had a positive coefficient with the selected model of bulk density, which contributes to an increased bulk density with an improved flow rate, as shown in Figure 6.2. Since the fact has been proven earlier that the flow rate had a negative effect with moisture loss (Wray & Ramaswamy, 2015b), which means the dried product conserves the weight when given the MWODS treatment at a higher flow rate. For drying time, the flow rate had negative effect, which makes sense with earlier discussion with ML that the flow rate reduces the ML during MWODS process and hence it had negative effect on finished drying time.

For quality attributes, the flow rate was positively correlated with hardness and chewiness, as presented in Figure 6.3. This indicated that an increase in flow rate ultimately increases the solids uptake and hence increases the hardness and chewiness of the finished dried samples. On the other hand, the flow rate was found to have a non-significant factor for b^* and total color change which is in agreement with the prior work (Wray & Ramaswamy, 2015b). However, the flow rate was found to have significant effect on L^* , a^* value which is unusual than previous results. One of the reasons might be due to the maltodextrin which acts as a protecting layer and reduces the leaching of color pigments (Dermesonlouoglou et al., 2016). Hence, the presented flow rate was found to have negative coefficient with a^* , b^* and ΔE value, which indicates that upon increasing flow rate, there will be increase in contact between fruit and solute mixture and hence reduces the total color change. This fact implies that the MD content in solute leached out monomers, which is a reactive component for non-enzymatic browning present in plant tissues and reduces the effect of browning (Tabtiang et al., 2012). This fact was also supported by Chun et. al, (2012) (Chun et al., 2012), where they found reduced discoloration upon incorporation of maltodextrin.

6.3.6 Optimization and process validation

The optimization function of design expert software was used to obtain the optimized condition for MWODS-air dried samples. The optimum conditions were achieved to obtain maximum ML, WR, ML/SG, rehydration capacity and bulk density while keeping SG, ΔE , hardness, chewiness at minimum level, while the process variables such as temperature (40-60°C), concentration (40-60%), contact time (10-30 min) and flow rate (1.5-3.0 L/min) in the experimental range. Amongst various optimization conditions, the solution which obtained higher desirability (0.591) was chosen to validate the process, as indicated in Table 6.4. The

optimized condition: temp. 51.7⁰C, Conc. 58.5%, contact time 30.6 min and flow rate 1.8 L/min were found at the given constraints. The optimized variables were validated for each response where, the optimum conditions were rounded to 50⁰C, 60%, 30 min and 2 L/min, to make it easier to perform the experiment. The variable constraints such as temperature, concentration, contact time and flow rate, were kept “in the range” to optimize the process. The responses such as ML, WR, ML/SG, RHC, and bulk density were targeted for “maximum “values. Whereas, SG, ΔE, hardness, chewiness and drying time were kept at “minimum” level.

Table 6.4: Predictive model validation

Responses	Predicted value	CI Low	CI High	Observed value
ML	45.6	42.2	46.9	46.1(±1.78)
SG	7.6	7.23	8.02	7.57(±1.00)
ML/SG	6.65	6.27	7.03	6.17(±0.85)*
WR	36.9	34.6	39.2	38.6(±2.12)
L*	16.8	15.9	17.7	18.1(±1.48)*
a*	9.73	9.37	10.1	9.99(±1.19)
b*	19.8	18.5	21.2	21.2(±1.59)
ΔE	22.8	21.6	24.0	24.4(±1.02)*
Hardness	86.1	75.8	96.5	92.8(±1.91)
Chewiness	50.5	48.2	51.7	49.3(±1.62)
RHC	99.2	93.9	104	98.2(±6.45)
Bulk density	371	365	376	369.3(±11.4)
Drying time	671	641	700	674(±40min)

Mean values with (standard deviation shown). CI: Confidence Interval (95%), ‘*’ Denotes observed value falls outside of predicted value confidence interval

6.3.7 Effect of solute mixtures at optimum MWODS performance level

The effect of three different solute mixture was compared to based on the quality of finished air dried product and the results are presented in Table 6.5. The preceding optimization study was based on a single type of sucrose-maltodextrin combination found effective in previous studies (Shinde & Ramaswamy, 2019a). This final phase of the study was to compare

the effect of the three solute combinations employed in the earlier study (Shinde & Ramaswamy, 2019a) on the finished air dried fruit quality. The responses such as L^* , a^* , b^* , ΔE , hardness, chewiness, RHC and bulk density have been chosen to compare. As shown in Table 6.5, it was found that the color parameters such as a^* , b^* and ΔE of S:MD were significantly different ($p < 0.05$) from other samples. While discussing an individual parameter, the L^* value of S:MD was the highest and closest to freeze-dried mangoes. This attributed to the fact that, indicated that the maltodextrin content in the solute mixture acts as a protecting layer and reduces the darkness caused due to browning at elevated processing conditions such as high concentration, temperature and contact time (Wray & Ramaswamy, 2015b). On the contrary, the lowest L^* value (darker samples) was recorded with air dried samples, which implies the importance of MWOD pretreatment during the dehydration process. In addition, the minimum a^* and maximum b^* value was observed when samples were treated with MD and which is closer to the a^* and b^* values of control samples (freeze-dried). As explained earlier, the MD content restricts the leakage of color pigments and creates the barrier layer on the fruit samples and hence reduces the destruction of color pigments (Dermesonlouoglou et al., 2016). As shown in Table 6.5, the overall color change (ΔE) was found to be the lowest with MD whereas maximum color change (ΔE) was reported with untreated (without MWODS) air-dried samples. This proves the commonly accepted notion that pretreatment such as osmotic dehydration would reduce the quality destruction of processed products (Wray & Ramaswamy, 2015d).

Table 6.5: Comparison of dried mangoes using different osmotic solutes

	Sucrose	S:D	S:MD	Fresh-AD
L^*	15.3(1.18) ^{ab}	14.0(1.86) ^{bc}	16.8(1.80) ^a	12.2(1.55) ^c
a^*	15.0(1.25) ^a	13.0(1.86) ^a	9.94(1.17) ^b	14.1(1.76) ^a
b^*	14.8(1.29) ^b	15.7(1.94) ^b	19.8(1.60) ^a	11.0(2.08) ^c
ΔE	31.3(1.39) ^b	31.3(2.50) ^b	26.2(1.97) ^c	36.0(2.07) ^a
Hardness	68.1(1.81) ^{ab}	67.7(1.89) ^{ab}	65.6(1.35) ^b	69.2(2.32) ^a
Chewiness	52.6(1.90) ^{ab}	52.1(1.85) ^{bc}	49.3(1.62) ^c	55.2(1.82) ^a
Bulk density	397.5(8.52) ^{ab}	389.5(8.71) ^b	379.3(9.70) ^b	411(9.22) ^a
RHC	79.5(7.48) ^{ab}	89.0(4.09) ^{ab}	98.2(6.45) ^a	78.8(7.84) ^b

Mean values with standard deviation shown—values that do not share a letter (a,b,c) are significantly different (Determined by Tukey method on 95% confidence interval)

Secondly, the textural changes were recorded to understand the behavior of each solute mixture on finished dried mango fruit. The lowest hardness and chewiness were observed when samples were treated with MD solute mixture. The similar results were also reported when MD was used to treat mango chips (Yolanda & Rosana, 2009). Similarly, the highest RHC was found with the samples pretreated with MD solute mixture. In contrast the MD samples exhibited lower bulk density compared with others due to high molecular MD content which restricts the entry of solids inside the fruit and ultimately reduces the bulk density by reducing the finished dried weight of mangoes (Azuara et al., 2002; İspir & Toğrul, 2009b; Shi et al., 2009). Overall, it was found that the solute mixture S:MD could produce better quality products when compared with S:D and sucrose as well as untreated (without MWODS) samples.

6.4 Conclusions

Overall it was found that temperature, concentration and contact time contributed to an increase the ML, ML/SG, WR; whereas, SG showed a similar trend with lower intensity. The study revealed that the intense processing conditions may compromise the quality of the product where any increase in temperature, concentration, contact could adversely affect the quality parameters of the dried mangoes. On the other hand, the optimal processing conditions could produce better quality air dried product under the given constraints. The predictive models were obtained using ANOVA, whereas the optimized parameters were achieved and verified at optimized processing constraints. However, by its very own nature the quality of dried products deviates from the fresh samples due to the process applications, whereas the MWODS minimizes the quality destruction by reducing the excess amount of dehydration time. Different solute mixtures were also compared at optimized conditions and it was recognized that the mangoes treated with S:MD under MWODS followed by air drying produced better results than S:D and sucrose solutes. In conclusion, the dehydrated product with minimum color change, comparatively firm texture along with high rehydration capacity was obtained by MWODS-air dried samples treated with S:MD solute mixture.

PREFACE TO CHAPTER 7

Healthy, long lasting and good quality food products have become the first choice of consumers. Therefore, keeping this in mind the research in the following chapter focuses on the application of microwave vacuum drying process on MWODS pretreated mangoes. The MWODS process using sucrose and maltodextrin mixture with more common finish-air drying process is detailed in the previous chapter. The current chapter is focused on microwave vacuum drying process and its application on post-MWODS mangoes. The microwave vacuum process was employed for cranberry in a previous research to understand the effect of MWODS on MWV product. However, the application of various solute mixture during MWODS and its effect on MWV product was never explored. Hence, in this chapter the influence of solutes such as sucrose and maltodextrin mixture is studied and the process is optimized using CCRD to understand the use of various power level settings in MWV while working with various parameters of MWODS process. Also, the structural properties are studied to identify the effect of solute mixture (sucrose and maltodextrin) on MWODS- MWV process.

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

Shinde, B. and Ramaswamy, H.S., 2018. Effect of osmotic solute mixture (sucrose: maltodextrin) on microwave osmotic dehydration under continuous flow medium spray-microwave vacuum process. Canadian institute of food science and technology (CIFST) 2018. May 27 to 29, 2018 in Niagara-on-the-lake, ON, Canada. (poster presentation)

CHAPTER 7

Effect of microwave vacuum drying on mango cubes pretreated with microwave osmotic dehydration under continuous flow maltodextrin moderated spray conditions

Abstract

A new combination drying technique of microwave osmotic dehydration under continuous flow medium spray condition (MWODS) followed by microwave vacuum drying (MVD) was used to evaluate the effect of processing factors such as temperature and concentration during MWODS and power level during MVD. The MVD as a finished drying technique was used in two settings with two different power levels, where initial power level was kept as one of the processing variables, used to reduce the moisture content in post-MWODS mangoes by 50%. Whereas, the final power level was maintained at 10% to achieve 20% moisture content in the dried product. The central composite rotatable design (CCRD) was used to select the different combination levels for the experimental design with the MWODS-MVD process. Finally, the optimized conditions were achieved by selecting to maximize ML, ML/SG, WR, bulk density, rehydration capacity and aiming to minimize ΔE , hardness, chewiness and drying times. Optimum levels were found at 58.5°C temperature, 50.0% concentration and 23.3% initial power level and applied for further study of comparison of sucrose solute and maltodextrin modified sucrose solute, with different finished drying methods such as MVD, freeze drying, vacuum and air drying. Overall, the MWODS treated mangoes using the maltodextrin mixture solute and MVD for finished drying resulted in better quality characteristics than other samples. It was also observed that the quality of MWODS-MVD samples treated with maltodextrin was closer to the standard set by freeze dried samples.

7.1 Introduction

The application of microwave heating has gained a lot of importance in the past couple of decades, in which researchers have used microwave drying in combination with other methods as in microwave freeze drying (Jiang et al., 2013), microwave fluidized bed drying (Momenzadeh et al., 2011), microwave-assisted pulsed spouted bed dehydration (Lu et al., 2014) and microwave vacuum drying (Drouzas & Schubert, 1996). Although the application of microwave energy in drying process is one of the fastest drying method which results in substantial reduction in drying time, the microwave energy is rarely used alone for drying purpose due to the uneven drying, scorching and off-color effects. However, the new techniques such as microwave vacuum drying has overcome the limitations of microwave drying where vacuum reduces moisture from the food sample at lower temperature and alleviates the physical damage from the microwave and accelerates the drying process with electromagnetic radiations (Gunasekaran, 1990; Raghavan & Orsat, 2007). Moreover, the absence of air during MVD process makes it more beneficial, where oxidation process can be eliminated and ultimately color damage can be reduced (Gunasekaran, 1990). Hence despite of the high installation and operating cost, the MVD process makes it more advantageous over conventional drying processes as it allows better color, texture and flavor of dried product (Yongsawatdigul & Gunasekaran, 1996a). In addition, the pretreatments such as osmotic dehydration is beneficial for retention of quality characteristics of dried product, which can be applied before finished drying process.

Osmotic dehydration has been widely known for quality retention and better energy conservation properties during the drying process. However, the conventional OD is very time consuming and hence many techniques such as pulsed electrical field, pulsed vacuum (Ito et al., 2007), ultrasound (Rodrigues & Fernandes, 2007), high pressure (Rastogi & Niranjana, 1998), and microwave drying have been studied to accelerate the process of dehydration. Moreover, the application of microwave energy during osmotic dehydration has been demonstrated to offer significant benefits over the conventional osmotic dehydration (Li & Ramaswamy, 2006c), which accelerate the moisture reduction, reduces solids gain as well as minimizes the quality losses in a short period (Azarpazhooh & Ramaswamy, 2012a). The microwave osmotic dehydration under continuous flow medium spray condition (MWODS) has also been further proven be even more beneficial for accelerating mass transfer in cranberries, which otherwise

requires skin pretreatment before OD due to the moisture barrier skin properties (Wray & Ramaswamy, 2013). It was also shown in prior studies (Chapter 3 and Chapter 5) that the application of solute mixtures is favorable in MWODS where maltodextrin moderated sucrose solution was found to be the effective to further limit the solids uptake and maximize the moisture reduction while reducing the quality losses of mangoes during MWODS pretreatment (Shinde & Ramaswamy, 2019a; Shinde & Ramaswamy, 2019c). In the previous chapter (Chapter 6) it has also been shown that the maltodextrin supplemented sucrose solution during MWODS had better impact on finished air dried samples, especially when compared with the mangoes pretreated with sucrose and dextrose supplemented sucrose solutions.

Therefore, the approach for current study was focused on the combined effect of MWODS-MVD process where the application of maltodextrin moderated sucrose solution was evaluated through different processing variables such as temperature and concentration of MWODS solute mixture and power level of MVD drying method. Prior studies had mostly focused on application of sucrose solution during MWODS process and evaluate the effect of MWODS pretreatment on MVD finished drying process (Wray & Ramaswamy, 2015b, 2015d). Whereas in current study the focus was mostly on application maltodextrin moderated sucrose solution during MWODS pretreatment and then evaluated and optimized the MWODS-MVD combined process. Furthermore, the optimized conditions were used for an overall comparison between commonly used sucrose solution and maltodextrin combined sucrose solution on various drying methods such as MVD, freeze, vacuum, air drying techniques.

7.2 Materials and method

7.2.1 Sample preparation

Frozen mango cubes were obtained from a local food freezing company (Nature's Touch, Canada) and kept frozen (-21°C to -27°C) until use. Prior to use, the mango cubes were thawed overnight (8-10 h) in a refrigerator (4°C - 7°C). The dimensions of the mango pieces were approximately 15 ± 2 mm. Commercial grade sucrose (Redpath Canada Ltd., Montreal, QC) and maltodextrin (10DE) (Univar Pvt. Ltd, Canada) were used and solutions were made with tap water. The AOAC method (AOAC, 1975) was used to determine the moisture content of fresh, MWODS treated and dried samples, gravimetrically. The experiments were replicated three times and the average of the moisture ratio at each value was used for preparing the drying

curves. The moisture content of fresh (frozen thawed) mango cube was determined as 86.09% (wb) on average.

7.2.2 Microwave osmotic dehydration pretreatment

The microwave osmotic dehydration pretreatment assembly is described in Chapter 3.

7.2.3 Finished drying

Finished drying process was applied on post-MWODS samples to obtain 20% moisture content (db) using microwave vacuum drying method. Also, other finished drying methods such as air drying, vacuum drying and freeze drying were employed for comparing the results between the microwave vacuum drying. Each of these methods are described briefly below.

7.2.3.1 Microwave vacuum drying

The finished drying was achieved using a bench top microwave vacuum dryer for all the CCRD design runs. The experimental setup used is illustrated in Figure 7.1 and was the same as previously described by Wray and Ramaswamy (2015a) (Wray & Ramaswamy, 2015a), which consisted of (A) a domestic microwave (Model AMW8113ST, Samsung, Mississauga, ON, Canada; 1kW nominal magnetron output, cavity dimensions 33 x 32 x 21 cm) in which (B) a custom-made cylindrical glass vacuum chamber (12 cm ID x 14 cm tall) was suspended from (C) an analytical balance (model TSK4D, Ohaus Corporation, Parsippany, NJ, USA). The vacuum chamber was connected to a cold water condenser/ water trap (D) and (E) a vacuum pump (Model M100EX, Emerson Motors, Markham, ON, Canada) on one side and (F) a standard analog vacuum gauge (Wika Instruments LP, Edmonton, AB, Canada) on the other side. All tubing used was small gauge Masterflex Norprene A-60-F food-grade tubing (McMaster-Carr, Cleveland, OH, USA). The consumer-grade silicone baking sheets (Wilton Industries, Etobicoke, ON, Canada) was used to seal the sample glass chamber and maintain approximately 93% vacuum (≈ 6 kPa abs) (Wray & Ramaswamy, 2015a).

Before the microwave vacuum process, the sample chamber was dried and placed into the desiccator prior to use. The empty sample chamber was then depressurized inside the microwave cavity and zeroed the balance. Further the vacuum was momentarily released and the pre-weighed samples (fresh or MWODS pretreated) were placed in a 10 cm diameter Pyrex Petri

dish lined with food grade silicon to prevent sticking of the samples to the glass and was kept inside a glass chamber. The samples were kept in a single layer on the petri dish and the chamber it was depressurized to the operating pressure (for about 1 min), the mass was recorded. The microwave was then run at the prescribed power level to dry the mangoes up to 20% moisture content (db).

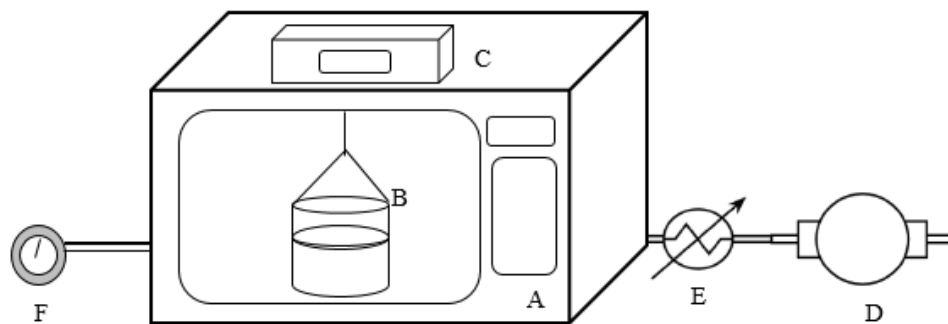


Figure 7.1: Schematic diagram of microwave vacuum drying assembly

7.2.3.2 Air drying

The post-MWODS mangoes were subjected for finished air drying process to obtain final moisture content of 20% (db). A domestic drying oven (Equi-Flow Food Dehydrator, Marysville, Wash., U.S.A.) was used with some modification with a digital thermostat to maintain conditions of 60 ± 1 °C, 0.64 ± 0.02 m/s and a RH of approximately 15%. The dryer was warmed up for 1 h before starting the experiment to achieve stable conditions. The post-MWODS mangoes were arranged in a single layer on a metal mesh which was suspended from a balance (Haus TS4KD MFD, Haus Corporation Florham Park, NJ) inside the drying chamber. The initial mass of the test samples were measured and kept inside the drying chamber, where the samples were subjected to a constant horizontal airflow inside the hot air-drying chamber. The door of hot air chamber was quickly opened 4 times during the process to rotate the sample mesh bed about 90° to alternate the side of the sample exposed to the oncoming hot air. The target weight of dried samples was calculated based on the initial mass and moisture content of the test samples. The resulting samples are referred as fresh-AD and MWODS-AD.

7.2.3.3 Vacuum drying

The vacuum drying method was performed on MWODS pretreated mangoes. The samples were weighed and transferred to a Fisher Scientific IsoTemp vacuum oven which had

been preheated to 60 °C and the pressure was reduced to 6 kPa abs using a model 100EX laboratory vacuum pump (Emerson Motors, Markham, Ont., Canada). The pretreated and fresh samples were vacuum dried to obtain 20% (db) moisture content. The resulting samples are referred as fresh-VD and MWODS-VD.

7.2.3.4 Freeze drying

The laboratory scale freeze dryer (SP Scientific/Virtis MR-145BA, Warminster, PA., U.S.A.) was used at -30°C and 13 to 20 Pa pressure. The process was continued approximately for 24 hours to achieve 20% (db) moisture content in dried samples. The resulting sample is denoted as fresh-FD and MWODS-FD.

7.2.4 Experimental design

The selection of variable factors for experimental runs were based on the combined effect of microwave osmotic dehydration and microwave vacuum drying process. Therefore, it was decided to choose temperature and concentration as two variable constraints in the experimental design to represent the MWODS process. On the other hand, initial power level (10% to 50%) of MVD drying was considered as the third variable constraints. Therefore, to obtain a desired range of experiments, a response surface methodology was applied using central composite rotatable design at five coded levels (-1.68, -1, 0, +1, +1.68) with three different process variables such as osmotic solute temperature, concentration and MVD initial power level (IPL), as shown in Table 7.1 and each run with three replicates was demonstrated. Since, the studies in previous chapters (Chapter 5, Chapter 6) have focused mostly on MWODS processing factors and therefore, the current studies were focused on combined effect of MWODS factors and MVD power level, on finished dried mangoes. The concentration and temperature were ranged numerically from 33.7-66.7, since it was notified that the concentration and temperature could be best studied in the range of 40-60 (Shinde & Ramaswamy, 2019c). In addition, the osmotic solute was prepared using two solute mixture of sucrose+maltodextrin (10DE) in 85:15 proportion. For preparing this, first the appropriate concentration of solute was selected and then the solute portion was adjusted to 85 proportion of sucrose and 15 proportion of maltodextrin. This proportion was adopted from the previous study where it was concluded that the S:MD proportion of 85:15 facilitated high water reduction along with better ML/SG ratio (Shinde &

Ramaswamy, 2019a). Contact time, flow rate was set at 30min and 2L/min which was derived from previous chapter (Chapter 4) where the air-drying process was used to obtain finished dried product.

During MVD process, the finished drying was carried out in steps using two power levels. The initial power level (IPL) was used to reduce the moisture content of post-MWODS samples by 50%, whereas final power level (FPL) was kept constant at 10% until the product reached to 20% moisture content (db). These power levels was adopted from prior work by Wray and Ramaswamy (2015) where the initial power level was set at 20% until the product reached to 50% moisture content (db) after which the power setting as reduced to 10% to achieve 20% moisture content product (Wray & Ramaswamy, 2015b). On the similar ground, in current study the initial power setting was employed using CCRD design ranging from 10% to 50%, while keeping final power level (FPL) constant at 10%. One of the reasons of selecting the two step power level process (initial and final power levels) was that, a constant application of higher power levels would initiate the product charring and damage the product quality (Wray & Ramaswamy, 2015b). Also, the previous work was mostly on a pre-tested the power levels at the given time interval, however in current study the power level was considered as one of the processing factors in CCRD design, which was further used for optimization of whole MWODS-MVD process.

During MVD, the sample mass was measured at selected times without disturbing the MVD assembly or without breaking the vacuum by observing the balance on top of the microwave from which the sample chamber was suspended and subtracting the mass of the petri dish. Once the required mass was achieved after applying initial power level (IPL), the MVD was then set to 10% power level to finally dry the sample to 20% moisture content (db). The microwave was then stopped once the sample was reached to a target mass and the vacuum was partially relieved to a level of ≈ 13 kPa abs for 5 min in order to allow the sample to cool slightly. Further the pump was disengaged from the system in order to fully restore atmospheric pressure. At this point the sample was removed, weighed, and kept in a sealed glass container for further analysis. the initial power level of MVD was studied in 10% to 50% range and ultimately all variable constrains were optimized based on the required constraints.

Table 7.1: CCRD experimental design for MWODS in real and coded values

Std Order no.	Temperature (°C)	Concentration (%)	Microwave power input
1	40(-1)	40(-1)	20(-1)
2	60(+1)	40(-1)	20(-1)
3	40(-1)	60(+1)	20(-1)
4	60(+1)	60(+1)	20(-1)
5	40(-1)	40(-1)	40(+1)
6	60(+1)	40(-1)	40(+1)
7	40(-1)	60(+1)	40(+1)
8	60(+1)	60(+1)	40(+1)
9	33(-1.68)	50 (0)	30 (0)
10	67(+1.68)	50 (0)	30 (0)
11	50 (0)	33(-1.68)	30 (0)
12	50 (0)	67(+1.68)	30 (0)
13	50 (0)	50 (0)	10(-1.68)
14	50 (0)	50 (0)	50(+1.68)
15	50 (0)	50 (0)	30 (0)
16	50 (0)	50 (0)	30 (0)
17	50 (0)	50 (0)	30 (0)
18	50 (0)	50 (0)	30 (0)
19	50 (0)	50 (0)	30 (0)
20	50 (0)	50 (0)	30 (0)

7.2.5 Mass transfer kinetics

Moisture loss (ML), solids gain (SG), moisture loss to solids gain ratio (ML/SG), and weight reduction (WR) were obtained using the following equations:

$$\% \text{ Moisture Loss (ML)} = 100 \frac{M_0 X_0 - M_t X_t}{M_0} \quad (1)$$

$$\% \text{ Solids Gain (SG)} = 100 \frac{M_0 S_0 - M_t S_t}{M_0} \quad (2)$$

$$\text{ML: SG ratio} = \frac{\%ML}{\%SG} \quad (3)$$

$$\% \text{ Weight Reduction (WR)} = 100 \frac{M_0 - M_t}{M_0} \quad (4)$$

where M_0 and M_t are the total mass of the fruit sample at time 0 and time t , respectively; X_0 and X_t are the moisture fractions (kg/kg, wet basis) at time 0 and time t , respectively; S_0 and S_t are the solid fractions (kg/kg, wet basis) at time 0 and time t , respectively.

The above Eqs. (1-4) were used assuming a uniform mass transfer of solids into the product and considering that there is no significant loss of solids from the sample into the solution.

7.2.6 Quality Analysis

Separate test runs were made for each run type, under the same conditions for quality analysis and measured the quality parameters as elaborated below. The quality of each experimental run type was examined after the finished drying process.

7.2.6.1 Texture analysis

TA.XT Plus Texture Analyzer (Stable Microsystems, Surrey, UK) was used for texture profile analysis (TPA) of finished air dried samples after MWODS treatments. Two-cycle compression test was performed to obtain TPA, using a flat bottom probe of 25 mm diameter, with a pretest speed of 5 mm/sec, the test speed of 5 mm s⁻¹ and post-test speed of 5 mm s⁻¹. The target compression was about a distance of 3 mm into the sample during two consecutive cycles to target a 25% deformation from the average height of samples. These settings with minor modifications were used with guidance from Banjongsinsiri et al. (2004), who used TPA on mango cubes (Banjongsinsiri et al., 2004). The analysis was performed with six replicates, and the average values (with standard deviation) were used. A wide range of responses such as hardness, chewiness, adhesiveness, cohesiveness, factorability, gumminess, and springiness can be obtained from TPA analysis (Bourne, 2002). Hardness and chewiness were selected in this study as parameters to determine the texture influence of osmotically processed mango cubes. The peak force defined hardness during the first compression cycle, and chewiness was obtained from the product of gumminess and (Bourne, 2002). The chewiness was calculated using Equation 6.5.

$$\text{Chewiness} = \text{Gumminess} \times \text{Springiness} \quad (6)$$

7.2.6.2 Color

The color of MWODS-air dried samples was analyzed in the L^* , a^* , b^* system using a tristimulus Minolta Chroma Meter (Minolta Corp., Ramsey, NJ, USA). The Chroma Meter was warmed up 20 min prior to use, and the color was calibrated against a white standard. Six measurements were made with each sample, and the values were averaged to obtain the L^* (lightness), a^* (green (-) to red (+)), and b^* (blue (-) to yellow (+)) values of the individual trials. The ΔE (total color change) was also measured where the total color change of processed samples was determined in comparison with the color of freeze-dried mangoes (without MWODS pretreatment). Since many studies have concluded that freeze-dried products can produce highly acceptable quality of the dried product (Wray & Ramaswamy, 2015d). Hence for this study the freeze-dried mangoes were chosen as a control sample to measure the color change of dried mangoes. It can be measured using the following equation 6 (Maftoonazad & Ramaswamy, 2008).

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

7.2.7 Scanning electron microscopy

To understand the microscopic structural behavior, the scanning electron microscopy (SEM) was employed for MWODS pretreated and untreated samples- finished dried samples. Each drying method was utilized to understand the SEM analysis and structural makeup of dried tissues. The dried samples were mounted on aluminum stubs with conductive adhesive in a scanning electron microscopy (SEM), (TM3000, Hitachi, USA). The imaging was performed at an accelerating voltage of 5 kV, from a working distance of 6 mm. The images were obtained under 200um to 1000um magnification to detect the possible structural changes of the dried fruit tissue.

7.2.8 Comparison of MWODS-solutes and finished drying methods

The commonly used sucrose solute was employed for MWODS process and the results are compared with the maltodextrin moderated sucrose solution (S+MD). Each solute was used for MWODS process which were further subjected to one of the four finished drying methods such as air drying, vacuum drying, freeze drying and MVD were employed to understand the

qualitative difference between each drying method and each solute type. The results were compared for color, texture, bulk density and rehydration capacity.

7.2.9 Statistical analysis

A commercial statistical package of Design-Expert version 6.01 (Statease Inc., Minneapolis, MN) was employed to calculate the RSM values. The statistical significance of the terms in the regression equations was examined by analysis of variance (ANOVA) for each response. The adequacy of the model was checked by the coefficient of determination, R^2 , adjusted- R^2 and coefficient variation (CV) (Myers et al., 2002). Similarly, for the comparative effect of drying kinetics the statistical analysis was carried out using JMP[®] v-13 (SAS Institute Inc., Cary, NC., U.S.A) by employing Tukey grouping method to understand the statistical difference between each method and solute combinations.

7.3 Results and discussion

7.3.1 Mass transfer kinetics

The response surface methodology was employed using CCRD design to optimize the MWODS-MVD process and the results (with standard deviation) of each experimental run is presented in Table 7.2. The mass transfer parameters were measured for MWODS process and each of the mass transfer responses were analyzed and the significant model terms are presented in Table 7.3. The effect of process variables such as temperature, concentration of MWODS process as well as power level of MVD is presented in the form of response surface plots as shown in Figure 7.2.

The quadratic model was found to be highly significant ($p < 0.0001$) for ML, WR and significant for SG repose. The temperature and concentration were found to be highly significant factor for ML and WR; and significant for SG, ML/SG responses. On the contrary, no model was found to be significant for ML/SG, however the linear model was found to have better sum of square and therefore linear model was selected for ML/SG response, as shown in Table 7.3. The power level of MVD was found to be a non significant variable for any of the mass transfer responses. This makes sense, as the mass transfer parameters are dependent on mass transfer variables which are temperature and concentration instead of finished drying variable such as power level of MVD.

Table 7.2: CCRD run numbers with results for mass transfer and texture, color parameters, RHC and bulk density

Std Order no.	ML (%)	SG (%)	WR (%)	ML/SG	Hardness (N)	Chewiness (N mm)	L*	a*	b*	Δ E	RHC (%)	Bulk density (Kg/m ³)	Drying time
1	36.3(±2.5)	5.12(±1.4)	31.2(±1.1)	7.31(±1.5)	48.4(±1.2)	40.8(±1.1)	27.6(±1.5)	10.7(±1.3)	27.4(±2.0)	13.2(±2.2)	84.4(+1.2)	249(±9.8)	46.0(±3.0)
2	48.7(±2.4)	6.23(±1.9)	42.5(±4.3)	8.25(±2.9)	58.2(±1.4)	54.2(±1.5)	27.3(±1.9)	9.49(±1.0)	25.2(±1.7)	14.7(±2.5)	79.7(+1.7)	253(±10)	35.0(±1.9)
3	42.8(±1.1)	7.61(±1.7)	35.2(±2.8)	5.78(±1.4)	63.1(±1.8)	55.2(±1.5)	29.7(±2.3)	11.6(±1.0)	27.8(±1.4)	11.8(±1.6)	92.1(+1.3)	250(±8.9)	41.0(±3.0)
4	55.3(±1.6)	8.93(±1.2)	46.4(±2.8)	6.27(±1.0)	101(±2.1)	61.6(±1.8)	26.5(±2.1)	11.7(±1.1)	32.1(±1.9)	11.9(±2.0)	88.1(+1.8)	255(±12)	31.0(±1.3)
5	34.5(±2.3)	5.42(±1.4)	29.1(±0.9)	6.54(±1.3)	87.4(±1.5)	72.7(±1.7)	25.6(±1.4)	10.9(±1.2)	28.3(±3.2)	14.8(±1.6)	103(+2.3)	216(±11)	34.0(±1.8)
6	46.4(±1.4)	6.69(±1.3)	39.8(±2.7)	7.10(±1.6)	90.3(±2.0)	69.5(±1.9)	26.6(±3.3)	6.64(±1.0)	21.2(±1.7)	18.6(±2.5)	89.2(+1.9)	231(±8.3)	26.0(±1.0)
7	41.9(±1.2)	7.17(±1.8)	34.7(±3.0)	6.05(±1.7)	111(±2.5)	76.2(±2.0)	27.4(±1.8)	12.0(±2.1)	32.0(±2.4)	11.4(±1.8)	110(+2.3)	218(±8.4)	30.0(±1.2)
8	54.9(±1.3)	8.73(±1.2)	46.2(±2.5)	6.37(±1.0)	128(±2.9)	81.3(±2.1)	26.2(±1.8)	10.0(±1.2)	30.1(±2.1)	12.7(±1.3)	98.6(+1.3)	240(±10)	21.0(±1.0)
9	36.2(±2.0)	6.11(±0.3)	30.1(±1.7)	5.92(±0.1)	73.1(±1.2)	55.0(±1.2)	30.0(±2.0)	9.38(±1.4)	27.3(±1.8)	11.6(±2.5)	127(+2.1)	214(±8.7)	40.0(±2.6)
10	53.8(±1.6)	7.40(±1.2)	46.4(±2.8)	7.38(±1.4)	107(±2.6)	78.0(±1.9)	28.0(±1.9)	10.0(±1.6)	28.1(±1.9)	12.4(±2.3)	108(+1.8)	245(±9.3)	26.0(±1.0)
11	33.2(±1.4)	6.28(±1.0)	26.9(±2.5)	5.38(±1.1)	76.2(±1.6)	63.0(±2.1)	27.2(±2.0)	11.0(±1.9)	30.2(±2.1)	12.1(±2.0)	84.1(+1.9)	227(±6.9)	43.0(±3.0)
12	46.4(±1.7)	8.94(±0.8)	37.4(±2.5)	5.22(±0.7)	130(±2.8)	73.0(±2.1)	29.7(±1.9)	9.23(±1.1)	27.8(±1.2)	11.4(±1.7)	97.0(+2.1)	216(±7.9)	33.0(±1.2)
13	47.4(±0.9)	7.01(±0.8)	40.4(±1.7)	6.81(±0.9)	40.3(±1.3)	42.0(±1.7)	28.9(±3.3)	12.0(±1.8)	23.4(±1.6)	15.9(±1.4)	78.3(+1.2)	261(±6.8)	44.0(±3.0)
14	48.4(±1.5)	8.13(±0.6)	40.3(±0.9)	5.96(±0.3)	137(±2.6)	72.0(±2.1)	24.1(±1.6)	10.3(±1.8)	21.4(±1.9)	19.9(±1.6)	75.6(+1.8)	228(±6.2)	17.0(±1.2)
15	47.6(±1.6)	7.87(±0.9)	39.7(±2.5)	6.10(±0.9)	83.1(±1.9)	52.4(±1.4)	25.0(±2.0)	7.50(±1.1)	27.2(±1.8)	15.3(±2.5)	128(+2.7)	260(±8.3)	32.0(±1.9)
16	48.6(±1.3)	7.50(±0.7)	41.1(±2.0)	6.51(±0.8)	77.5(±1.8)	53.8(±1.1)	27.1(±2.0)	11.0(±2.0)	28.8(±1.3)	12.8(±1.9)	155(+3.2)	243(±7.8)	31.0(±1.0)
17	46.6(±2.5)	7.60(±1.1)	39.0(±1.4)	6.17(±0.5)	73.9(±1.4)	54.5(±1.9)	26.0(±1.9)	10.2(±1.4)	28.5(±1.7)	13.8(±1.8)	134(+2.6)	244(±8.2)	32.0(±1.6)
18	49.0(±3.9)	8.07(±1.2)	40.9(±5.1)	6.18(±1.4)	80.2(±1.7)	58.9(±1.7)	27.8(±2.0)	9.22(±1.2)	31.1(±1.9)	10.8(±2.3)	121(+2.9)	250(±7.4)	30.0(±1.0)
19	48.9(±2.4)	7.33(±1.1)	41.6(±3.5)	6.78(±1.4)	70.8(±1.2)	52.8(±1.6)	26.3(±1.9)	11.0(±1.2)	29.0(±1.7)	13.3(±1.0)	143(+3.0)	246(±7.9)	31.0(±1.4)
20	48.2(±1.4)	7.93(±1.1)	40.3(±2.5)	6.15(±1.0)	74.8(±1.7)	50.4(±2.0)	27.9(±2.1)	10.3(±1.3)	30.5(±1.8)	11.0(±2.6)	130(+2.6)	255(±8.4)	32.0(±1.2)
FD							37.0(±3.4)	8.94(±2.0)	36.4(±3.1)				
Mango													

Table 7.3: Predicting equation and compiled ANOVA results for CCRD responses

Response s	Model	Predicting equations in terms of actual variables	Lack of fit	R ²
ML	Quadratic	ML= -78.7+ 1.39 *T+ 2.88 *C-0.22*P-8.87E ⁻⁰⁰³ *T ² - 0.03 *C ² +4.09E ⁻⁰⁰⁴ *P ² +1.50E ⁻⁰⁰³ *T*C+9.69E ⁻⁰¹⁷ *T*P+3.50E ⁻⁰⁰³ *C*P	0.1565 (NS)	0.9806
SG	Quadratic	SG= -13.8+0.41*T+ 0.22 *C+0.12*P-3.99E ⁻⁰⁰³ *T ² -1.03E ⁻⁰⁰³ *C ² -7.03E ⁻⁰⁰⁴ *P ² +6.25E ⁻⁰⁰⁴ *T*C+5.000E ⁻⁰⁰⁴ *T*P-1.75E ⁻⁰⁰³ *C*P	0.1118 (NS)	0.9222
WR	Quadratic	WR= -64.8+ 0.97 *T+ 2.67 *C-0.34*P-4.80E ⁻⁰⁰³ *T ² - 0.03 *C ² +1.19E ⁻⁰⁰³ *P ² +8.75E ⁻⁰⁰⁴ *T*C-3.75E ⁻⁰⁰⁴ *T*P+5.13E ⁻⁰⁰³ *C*P	0.1653 (NS)	0.9731
ML/SG	<i>Linear</i>	ML/SG= +7.10+0.04*T-0.04*C-0.02*P	0.0264 (S)	0.4281
Hardness	Quadratic	Hardness =+260-3.71*T- 8.78 *C+ 2.33 *P+0.03*T ² +0.07*C ² +0.02*P ² +0.05*T*C-0.04*T*P+5.00E ⁻⁰⁰³ *C*P	0.0800 (NS)	0.9636
Chewiness	Quadratic	Chewiness =+182.9-3.36*T-4.36*C+ 1.96 *P+0.04*T ² +0.04*C ² +8.03E ⁻⁰⁰³ *P ² +1.62E ⁻⁰⁰³ *T*C-0.02*T*P-8.12E ⁻⁰⁰³ *C*P	0.0988 (NS)	0.9306
L*	Linear	L* =+30.1-0.05*T+0.05*C- 0.09 *P	0.3970 (NS)	0.4573
a*	<i>Linear</i>	a* =+12.1-0.04*T+0.03*C-0.04*P	0.5091 (NS)	0.2049
b*	<i>Quadratic</i>	b* =+34.3-0.07*T-0.96*C+1.28*P-2.89E ⁻⁰⁰³ *T ² +1.44E ⁻⁰⁰³ *C ² - 0.02 *P ² +0.01*T*C-0.01*T*P+6.63E ⁻⁰⁰³ *C*P	0.0743 (NS)	0.6996 (NS)
ΔE	Quadratic	ΔE =-5.49+0.42*T+ 0.66 *C- 0.59 *P-2.47E ⁻⁰⁰³ *T ² -3.34E ⁻⁰⁰³ *C ² + 0.01 *P ² -4.87E ⁻⁰⁰³ *T*C+4.37E ⁻⁰⁰³ *T*P-6.37E ⁻⁰⁰³ *C*P	0.6928 (NS)	0.7923
RHC	Quadratic	RHC =-626.9+7.26*T+16.8*C+10.4*P- 0.07 *T ² - 0.16 *C ² - 0.15 *P ² +3.87E ⁻⁰⁰³ *T*C-0.02*T*P+3.75E ⁻⁰⁰⁴ *C*P	0.7626 (NS)	0.8952
Bulk density	Linear	BD =+237+ 0.72 *T-0.03*C- 1.05 *P	0.0799 (NS)	0.5455
Drying time	Quadratic	DT = +150- 0.97 *T- 2.35 *C- 0.66 *P+3.71E ⁻⁰⁰³ *T ² +0.02*C ² -3.13E ⁻⁰⁰³ *P ² -1.93E ⁻⁰¹⁶ *T*C+5.00E ⁻⁰⁰³ *T*P-1.36E ⁻⁰¹⁶ *C*P	0.0543 (NS)	0.9813

Where T is Temperature (°C), C is Concentration (%), P is the microwave power levels (%). Note that highly significant (p < 0.0001) models and variables are in bold, significant (p < 0.05) are normal type, while non-significant (p > 0.05) factors are italicized.

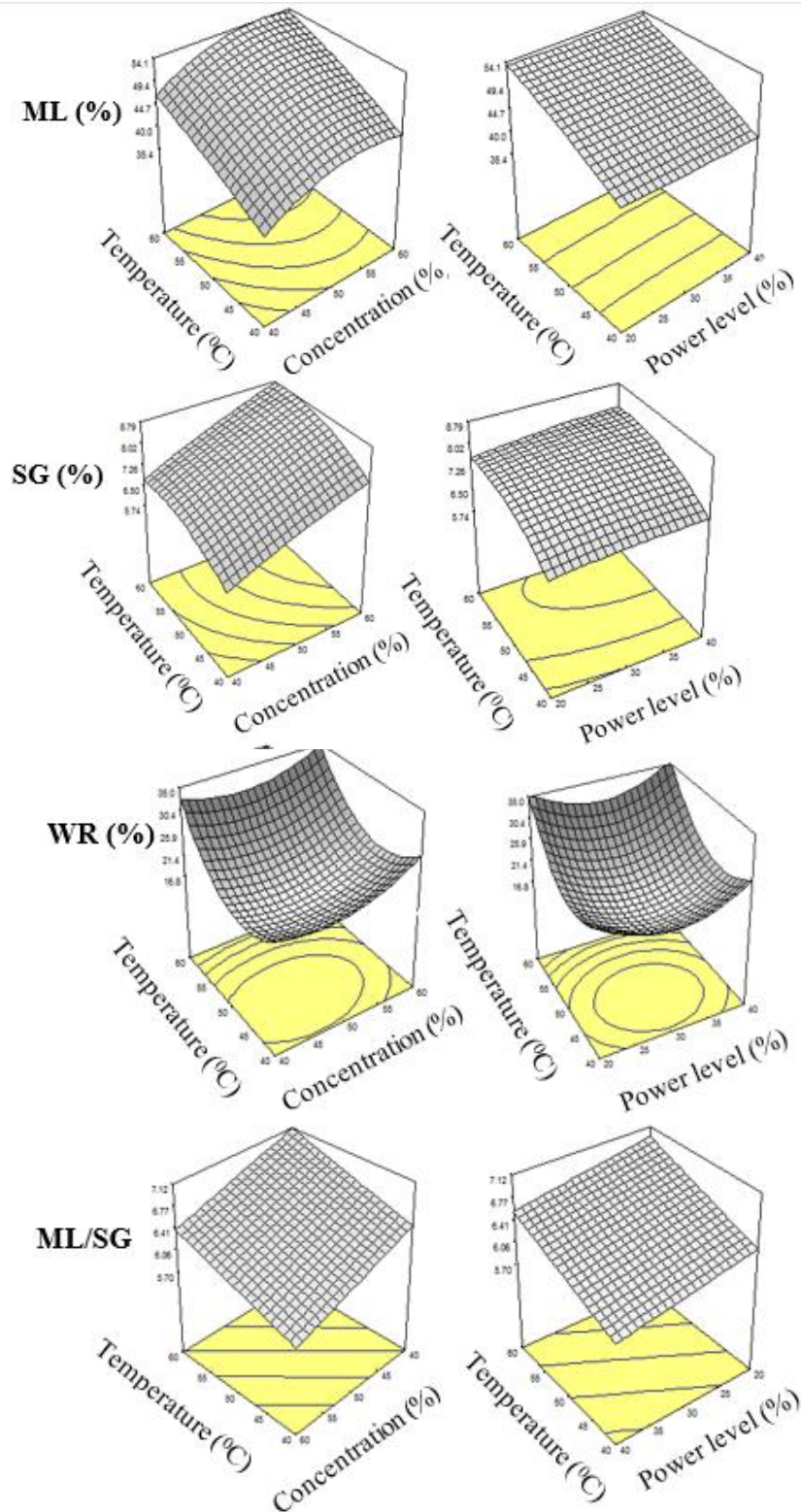


Figure 7.2: Response surface plots for mass transfer for ML, SG, ML/SG, WR. (When not a variable, inputs were fixed at their center point: Conc. 50%, contact time 30min, flow rate 2.0 L/min)

In addition, the effect of individual variables on each response were analyzed and tabulated in Table 7.3. It was observed that the variable temperature and concentration had positive coefficient for ML, SG and WR which attributed to previously proven fact that an increased MWODS temperature and concentration produces high ML, SG and WR (Azarpazhooh & Ramaswamy, 2012a; Shinde & Ramaswamy, 2019a; Wray & Ramaswamy, 2013). On the other hand, the temperature had positive and concentration had negative coefficient for ML/SG response. This attributed to the fact that any increase in solute concentration (maltodextrin moderated solute concentration) would have negative impact on solids gain. Since the ratio of ML/SG itself is the impact of both ML and SG together. Therefore, the concentration showed entirely opposite trend for SG response and for ML/SG ratio. The similar results were also confirmed previously (Shinde & Ramaswamy, 2019c) which proves that maltodextrin restricts the solids intake during MWODS pretreatment.

7.3.2 Color analysis

The mango fruit is widely popular due to its organoleptic characteristics, where color is considered as one of the important factor during quality analysis, especially for processed mangoes. Current study was focused on implementing the MWODS process using complex solutes, along with MVD drying method, where the effect of solute mixture on color parameters was the concern. In addition, the microwave processes itself has been widely discussed for the overheating and charring of samples, which is one of the challenges during this process (Gunasekaran, 1990). Hence, any changes in the appearance of the product due to destruction or leakage of color pigment was monitored through color analysis, while analyzing the optimum conditions for MWODS- MVD process.

The major concern during OD was the leaching of color pigment, which can be monitored through L^* value (light or dark color). The response plots indicating effect of each processing variable is presented in Figure 7.3. The L^* , a^* and b^* value was significantly affected by concentration of MWODS solute mixtures and power level of microwave vacuum drying as shown in Table 7.3.

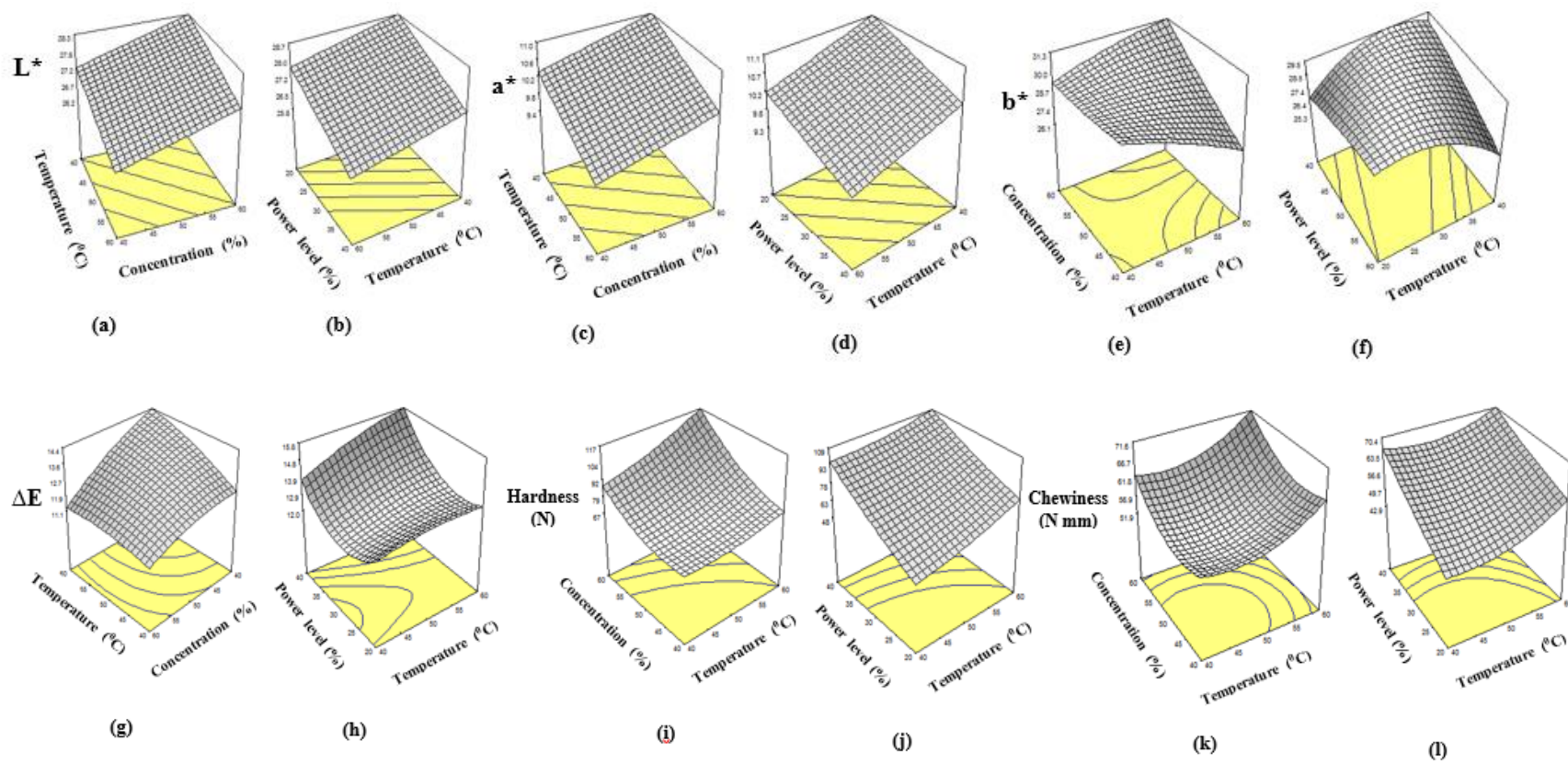


Figure 7.3: Response surface plots for color (L^* , a^* , b^* , ΔE) and texture (hardness, chewiness) analysis. (When not a variable, inputs were fixed at their center point: Conc. 50%, contact time 30 min, flow rate 2.0 L/min)

The negative coefficient of temperature and concentration for L^* , a^* shows that the product will be lighter in color and declined its redness with any increase in temperature and concentration. This attributed to the fact that, intensifying MWODS process parameters such as concentration and temperature, have a deleterious effect on its color pigment, the result which was also confirmed in prior studies (Wray & Ramaswamy, 2015b, 2015d). On the contrary, temperature and concentration had positive coefficient for b^* and ΔE , which reflects that with increased temperature, the product will become more yellow due to the concentrated color pigments during dehydration process and also increases the overall color change (ΔE). Since it is not uncommon during drying process that the product color intensifies due to the concentration of color pigment (Chiralt & Talens, 2005).

The power level of MVD drying was also evaluated in CCRD with color analysis, where it was observed that the power level had negative effect on L^* and a^* which reflects that the microwave power makes the product darker and also increases the redness. This confirms previously published reports (Sunjka et al., 2004) that the redness of the sample declined as power level of microwave increases. On the other hand, any increase in the MVD power level increases yellowness (b^*) and total color change (ΔE) of the dried product, which supports the prior discussions that increase in yellowness was due to concentration of natural color pigments due to dehydration (Chiralt & Talens, 2005).

7.3.3 Mechanical properties

Texture profile is considered as one of the important characteristics in processed fruits. Hence the MWODS- MVD dried mangoes were subjected to texture analysis to analyze the hardness and chewiness of the samples. The hardness and chewiness was selected instead of focusing on other textural characteristics, as most of the dried products are of concern due to their hard texture as well as its poor chewing properties. The mechanical property of the dried fruit has been discussed due to the effect of drying process on the physical state as well as the structure changes caused due to the typical deformations such as cell shrinking or swelling, changes in intercellular spaces (volume), and rupturing of cell bonds (Contreras et al., 2007). Since many studies have commented on the mechanical properties after finished drying of OD pretreated fruits, however the interest here is to identify if any significant changes could take place when complex solute mixture was employed during MWODS process. Also, these results

produced various combination of concentration and temperature of MWODS along with different power settings of MVD.

The results obtained from CCRD responses have presented in Table 7.3 and reflected in response plots as shown in Figure 7.3. The quadratic model was found to be the best fit for both hardness and chewiness responses, where for hardness the model was found to be the highly significant ($p < 0.0001$) model. All the factors were found to be the significant ($p < 0.05$) model terms for hardness and chewiness, where MVD power level was reported to be highly significant factor ($p < 0.0001$) for hardness. Amongst all, the concentration of S:MD solute mixture had negative effect on these responses, which has proven the previously confirmed fact that the MD moderated solution had a protecting effect on structural deformation which restricts the product to become harder (Shinde & Ramaswamy, 2019a; Shinde & Ramaswamy, 2019c). In addition, the factors such as temperature of MWODS and power level of finished drying process have also shown negative effect on hardness and chewiness, indicated that any increase in temperature and power level decreases the hardness and chewiness.

7.3.4 Rehydration capacity

The rehydration capacity reflects the physical and chemical changes occurred during osmotic dehydration, and therefore it can be used as a quality index in many research studies. In current study, the influence of process variables on RHC is presented in the form of model, represented in Table 7.3 and the influence of process variables on RHC is presented in shown in Figure 7.4 (a and b). The quadratic model term as found to be significant ($p < 0.05$) for the rehydration capacity (RHC) and the positive coefficient of all the variables shows that elevated temperature, concentration and power level will increase the RHC. However, the higher degree polynomial of each factor shows negative correlation with RHC as observed in Table 7.3. The positive coefficient of temperature and concentration shows the discrepancy with the prior work where mangoes were subsequently treated with air drying after MWODS process (Article 4). One of the reasons behind the discrepancy was the type of finished drying method used. The previously confirmed results with lower RHC of air-dried fruit due to excessive heat during air drying thereby affecting the ability of the tissue to absorb and to retain water which reverse the positive effect occurred during osmotic process (Azarpazhooh & Ramaswamy, 2012a). However, during this study the subsequent MVD drying had improved the RHC of the dried

mangoes due to its heating mechanism. It was confirmed that application of microwave causes increased permeabilization of the cell membranes which facilitate faster water loss during drying (Azarpazhooh & Ramaswamy, 2012a). Hence any increase in temperature and concentration during MWODS as well as increase in power level during subsequent MVD drying improves the rehydration ability of dried samples.

7.3.5 Bulk density

The bulk density has been considered as one of the essential parameters from logistic point of view. Also, BD attributed to the amount of destruction of cell structure during drying process which leads to the high shrinkage and ultimately high BD. The higher BD value specifies the ability of dried product to store in minimum space area with maximum mass balance. Therefore, studying BD during MVD drying is one of the concerns as the heating mechanism of microwave differs than a traditional air-drying process. The response plots indicating the behavior of process variables on bulk density is given in Figure 7.4 (c) and (d). The linear model was found to be significant for BD and the temperature of MWODS and power level of MVD was found to be the significant ($p < 0.05$) model terms, as shown in Table 7.3. While discussing individual variable it was found that temperature had positive effect on BD, indicating that the increase in temperature increases water removal which tended to the destruction of cellular structure of the fruit tissues making it more compact. As a result, the product shrinkage takes place and the bulk density tended to increase. On the contrary, the negative coefficient of concentration and power level attributed to the reduction in BD. The reason behind the effect of concentration is unknown while power level increases the puffiness of the dried sample, which means it prone to increase the porosity of the sample tissues. The rapid formation of steam in presence of microwave radiation is the main cause of puffed appearance of MVD samples, which was also evident in prior work (Andrés et al., 2004; Wray & Ramaswamy, 2015b). Hence there is reduced BD upon increase in power level during MVD.

7.3.6 Drying time

The quadratic model was found to be the most significant ($p < 0.0001$) model for drying time where temperature, concentration and power level of MVD was found to be highly significant ($p < 0.0001$) terms for this response. The response surface plots represented in Figure

7.4 (e,f) shows the effect of each variable on finished drying time of MWODS pretreated mangoes.

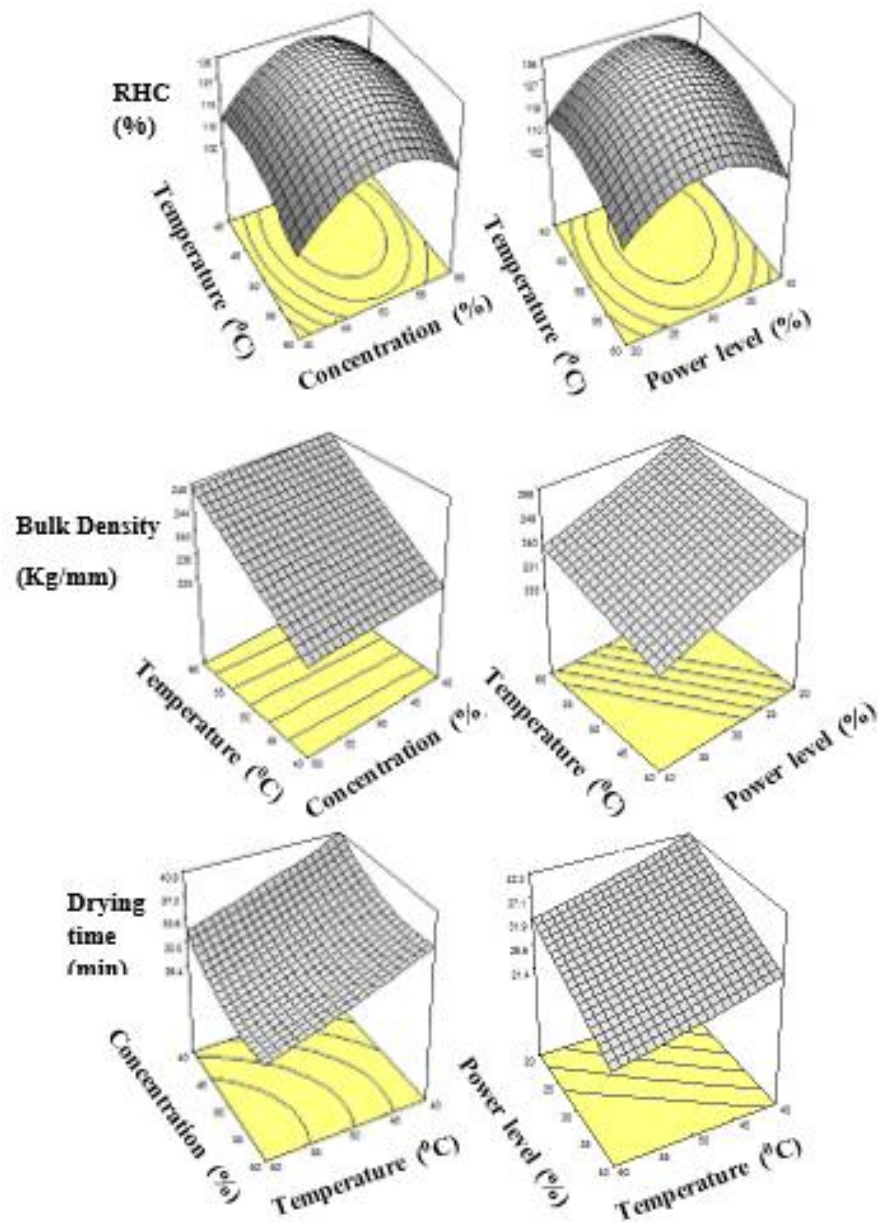


Figure 7.4: Response surface plots for color (L^* , a^* , b^* , ΔE) and texture (hardness, chewiness) analysis. (When not a variable, inputs were fixed at their center point: Conc. 50%, contact time 30 min, flow rate 2.0 L/min)

The negative coefficient of each variable indicates that increase in process intensity will reduce the finished drying time that is MVD drying time. The prior studies have also found the similar outcome where increase in MVD power level was found to have reduced drying time (Wray & Ramaswamy, 2015a). In current study, the various combination of MWODS temperature and concentration was studied together with MVD power level. The highest negative coefficient of concentration attributed that the concentration of solute mixture was the highly affected variable amongst all. Since high concentration prone to increase in ML, especially when the solute mixture is moderated with maltodextrin content gives better ML and hence the finished drying time was short when concentration increases. This fact was supported by Azuara where MD content in OD solute found to promote ML (Azuara et al., 2002; Shinde & Ramaswamy, 2019a).

7.3.7 Optimization and process validation

The optimization of the process MWODS-MVD was achieved by keeping all process variables such as temperature (40-60°C), concentration (40-60%) and MVD initial power level (20-40%) in the experimental range whereas, ML, ML/SG, WR, BD, RHC was set at maximum level and E, hardness and chewiness was kept at minimum level. During the optimization few of the responses such as L*, a* and b* were kept in experimental range, because the total color change was kept at minimum level, which includes all the individual color parameters. The optimization was achieved by keeping SG in the experimental range, as the response ML/SG was already kept at maximum level, which means that the SG was indirectly set at minimum level. The optimization solution which obtained higher desirability (0.675) as 58.5°C-50.0%-23.3IPL was chosen. The optimum conditions were rounded to 60°C, 50% and 20%IPL, to make it easier to perform the experiments. The process MWODS-MVD using maltodextrin moderated sucrose solution was validated and the predicted along with experimental results were tabulated in Table 7.4. It indicates that most of the responses were in the given range of the prediction.

Table 7.4: Predictive model validation

Constraints/ Responses	Optimization	Model validation		
	solution and predicted values	CI Low	CI High	Observed value
Temperature	60	-	-	-
Concentration	50	-	-	-
MVD-IPL	20	-	-	-
ML	53.3	51.7	55.0	53.8(± 1.2)
SG	7.58	7.04	8.12	7.74(± 1.0)
ML/SG	6.96	6.43	7.50	7.02(± 1.1)
WR	45.8	44.1	47.4	46.1(± 2.2)
Hardness	73.0	63.9	82.2	74.8(± 2.1)
Chewiness	56.4	50.8	62.0	59.4(± 1.9)
L*	27.7	26.5	28.8	29.0(± 1.3)*
a*	10.2	8.99	11.4	10.7(± 1.0)
b*	28.7	25.7	31.8	29.4(± 2.3)
ΔE	13.2	11.1	15.2	11.0(± 1.2)*
RHC	106	92.7	120	85.8(± 2.5)*
Bulk density	258	247	268	250(± 8.66)
Drying time	32.4	30.5	34.2	33.0(± 1.0)

Mean values with (standard deviation shown). CI: Confidence Interval (95%), ‘*’ Denotes observed value falls outside of predicted value confidence interval

7.3.8 Effect of solute mixtures on finished drying methods

The overall product quality was compared between different drying methods, with or without MWODS pretreatments. As discussed earlier, the two solutes such as commonly used sucrose and maltodextrin modified sucrose (S+MD) were used to evaluate the difference between each solute mixture as well as finished drying methods. As analyzed before, the optimized condition of 60⁰C/50% for MWODS was used. For MVD drying the optimized value

of 20% initial power level and 10% final power level was applied to dry the samples. The drying methods such as air drying, freeze drying, vacuum drying and microwave vacuum drying (MVD) was employed to compare the results as presented in Table 5.

The color of each drying method was compared with the color of fresh freeze dried samples, where it was observed the L^* , a^* and b^* value of MWOD-MD-MVD was found to be closer to FD samples whereas the MWODS-sucrose-AD was found to be more deviating from FD. For total color change (ΔE), MWODS-sucrose-AD was found to have higher ΔE than any other method. One of the reasons behind the higher color change was due to presence of high amount of sucrose during the MWODS treatment, which might initiate the browning effect during the process and produce darker samples. Secondly the AD method is the slower which makes it difficult to sustain the original color of the samples. For textural characteristics, the hardness of the MWODS-sucrose-AD was found to be higher due to the fact that the SG during MWODS process in presence of sucrose was higher which might damage the cell wall structure of the plant cell and makes it harder in comparison. Whereas, FD was found to have comparatively lower hardness followed by MWODS-MD-MVD.

In contrast, the bulk density of MWODS-sucrose-AD was higher whereas, fresh FD had the lowest BD followed by MWODS-MD-MVD. The RHC of FD was higher and MWODS-MD-FD was second highest than other drying methods. Overall, the quality characteristics of FD was found to be the best followed by MWODS-MD-FD, which attributed the fact that MD has a protective nature to minimize the quality damage than the commonly used sucrose solute.

7.3.9 Structural properties

Scanning electron microscopic (SEM) studies were carried out to visualize the structural changes in dried mangoes with or without the pretreatment with sucrose or maltodextrin solute mixture. Figure 7.5 (a to i) shows scanning electron micrographs to distinguish the difference in the structure of the dried samples. It was observed that the freeze dried (with and without pretreatments) samples were porous in structure which is widely open and less dense. This makes sense while looking back at the bulk density, where FD samples have shown minimum BD due the porous structure of the FD samples.

Table 7.5: Comparison of drying methods with/without MWODS pretreatment using different solute mixtures

Sample Type	Hardness	Chewiness	L*	a*	b*	ΔE	Bulk density	RHC
Fresh	62.2(1.99) ^d	50.7(1.86) ^{a,b,c}	37.0(2.47) ^a	8.94(1.12) ^b	36.4(1.30) ^a		215(9.22) ^e	134(6.75) ^a
FD								
Sucrose	70.2(2.62) ^a	52.5(1.80) ^a	14.8(1.52) ^e	11.9(1.29) ^e	16.8(1.59) ^e	29.8(1.40) ^a	392(8.45) ^a	81.5(8.47) ^c
MWODS AD								
MD	68.1(2.85) ^{a,b,c}	48.1(1.61) ^{c,d}	16.0(1.80) ^e	10.7(1.27) ^{d,e}	19.7(1.60) ^{d,e}	27.0(1.98) ^{a,b}	384(9.67) ^a	92.8(2.67) ^c
MWODS AD								
Sucrose	68.2(2.44) ^{a,b}	51.4(1.74) ^{a,b}	17.3(2.03) ^e	11.2(1.39) ^{a,b}	19.2(1.76) ^e	26.4(2.24) ^b	312(7.66) ^b	90.0(8.33) ^c
MWODS VD								
MD	67.2(2.26) ^{a,b,c}	46.0(1.63) ^{d,e}	16.2(1.80) ^e	10.4(1.29) ^{a,b}	23.5(1.22) ^c	24.6(1.03) ^b	304(10.2) ^b	112(6.99) ^b
MWODS VD								
Sucrose	64.4(2.19) ^{b,c,d}	46.2(1.82) ^{d,e}	27.1(2.02) ^d	9.88(1.71) ^{a,b}	24.0(1.65) ^c	16.1(1.62) ^c	245(6.68) ^{c,d}	122(7.62) ^{a,b}
MWODS FD								
MD	63.7(2.22) ^{c,d}	42.2(1.82) ^f	34.4(2.41) ^{a,b}	9.20(1.17) ^b	31.1(1.46) ^b	6.54(0.89) ^e	230(9.13) ^{d,e}	129(8.77) ^{a,b}
MWODS FD								
Sucrose	66.1(2.47) ^{a,b,,c,d}	48.2(1.54) ^{b,c,d}	31.1(2.46) ^{b,c}	10.7(1.22) ^{a,b}	22.2(1.53) ^{c,d}	15.6(1.74) ^c	260(9.90) ^c	116(7.72) ^b
MWODS								
MVD								
MD	64.5(2.08) ^{b,c,,d}	43.7(1.92) ^{e,f}	29.2(2.05) ^{c,d}	10.1(1.03) ^{a,b}	29.4(1.68) ^b	10.8(1.24) ^d	250(8.66) ^{c,d}	120(6.48) ^{a,b}
MWODS								
MVD								

Similar outcomes were reported in prior studies where FD have shown more open structure than air drying (AD) and vacuum drying (VD) (Wray & Ramaswamy, 2015b). While comparing the pretreated samples such as MWODS with sucrose (MWODS-S-FD) and MWODS with maltodextrin (MWODS-MD-FD) freeze dried mangoes, it was found that the untreated FD samples were comparatively less porous (Figure 7.5 a) than treated, amongst which the pretreated samples with sucrose solute (MWODS-S-FD) was more compact than those treated with maltodextrin (MWODS-MD-FD) as shown in Figure 7.5 (b) and (c), respectively. Firstly, the porous structure in pretreated samples were attributed to the microwave heating mechanism where water removed from the inner core of the sample towards the outer layer, through the cellular channels and which makes it more porous after the dehydration process. Since this observation was also notified with conventional OD where the researchers have proven that the reduction in moisture content before drying, is needed to improve the texture characteristics of fruits, irrespective of the dehydration method used (Maestrelli et al., 1997). On the other side of the spectrum, the application of maltodextrin solute mixture during the pretreatment (MWODS) makes the product highly porous and open in structure than sucrose samples, because the samples treated with maltodextrin attributed to the samples with reduced moisture content when subjected to finished drying process and likely it produced more porous structure of dried samples. The similar outcomes were also published where the osmotic solutes containing high molecular weight solute restricts the process of plasmolysis due to reduced solids uptake during OD treatment (Chauhan et al., 2011). Whereas lower molecular weight solutes might lead to plasmolysis in the cells resulting in deformation, contraction, and collapse, due to high solids intake during OD process. Therefore, the similar effect was recorded in all other drying methods such as air drying (AD), vacuum drying (VD) and microwave vacuum drying (MVD), where pretreated samples with sucrose produced less porous samples than those treated with maltodextrin.

The air drying method yielded packed structure when compared with all other drying methods as shown in Figure 7.5 (d) and (e). Difference in microstructure between MWODS-S-AD and MWODS-MD-AD was not clearly visible, as the conventional hot air drying damaged the cell structure and formed more shrunken and compressed cellular structure. The insignificant difference in structure was also reported on dried cooked rice that was dried at 50⁰C, 80⁰C and 120⁰C, respectively (Luangmalawat et al., 2008). The VD samples were more open in structure

than AD, however it is less porous than FD and MVD as shown in Figure 7.5 (f) and (g). Ultimately, the high porosity with comparatively less dense and compressed structure was noticed in the samples dried by microwave vacuum drying method (MVD) as shown in Figure 7.5 (h) and (i). The pore development after microwave-vacuum drying is presumably because of tissue expansion from the internal water vapor pressure (Argyropoulos et al., 2011). The vapor bubbles could increase total pressure gradient inside the mango fruit tissue and therefore enhanced the porosity. The structural features of MVD samples were more like a freeze dried samples which was observed previously with cranberries (Wray & Ramaswamy, 2015b).

7.4 Conclusions

Overall, it was observed that the process of MWDS-MVD can be implemented with the optimized conditions of 58.5⁰C-50.0%-23.3IPL, within given constraints. It was also found that the pretreatment of MWODS had substantial effect on finished dried product especially, the finished drying time. The MVD power level was found to be the significant factor in quality attributes' textural properties and other physical properties of the dried product. The comparison between the solute mixture on finished dried product found that the microwave vacuum dried samples treated with maltodextrin had the product quality which was closest to freeze dried samples. Also, the structural properties of the MVD samples treated with maltodextrin MWODS were found to be porous in structure compared to samples treated with sucrose. Hence, it can be concluded that the maltodextrin moderated sucrose solution during MWODS treatment had positive impact on microwave vacuum dried mangoes greater than sucrose solution. Also, the MVD was found to be the best finished drying method after the freeze drying.

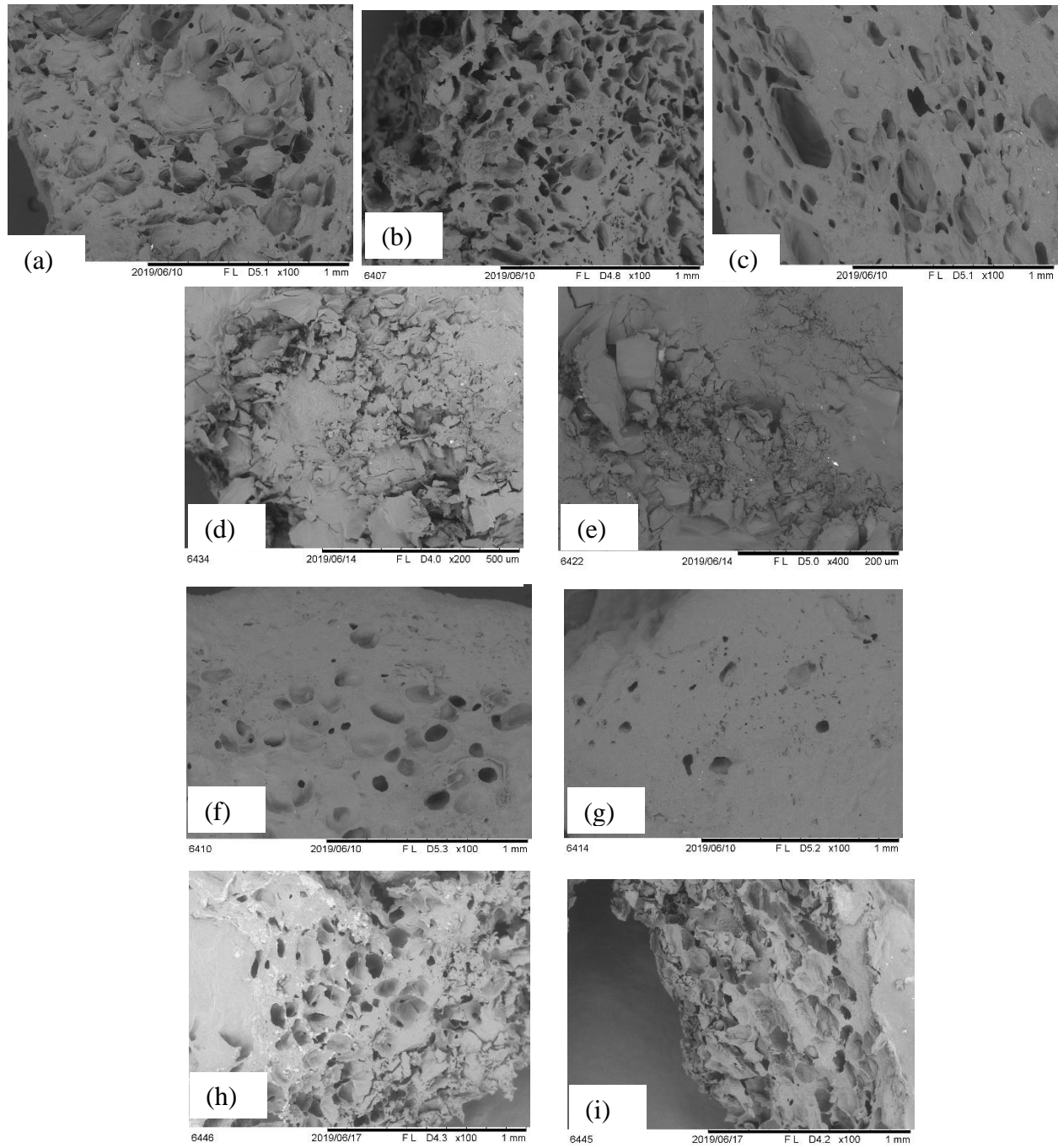


Figure 7.5: Cellular structure of dried Mangoes; Where (a) Fresh-FD; (b) MWODS with MD-FD; (c) MWODS with S-FD; (d) MWODS with MD-AD; (e) MWODS with S-AD (f) MWODS with MD-VD; (g) MWODS with S-VD (h) MWODS with MD-MVD; (i) MWODS with S-MVD.

CHAPTER 8

SUMMARY, CONCLUSIONS AND SUGGESTIONS FOR FUTURE RESEARCH

The application of solute mixtures using maltodextrin in sucrose solution was evaluated and optimized in the microwave osmotic dehydration of mango cubes. The effect of solute mixture was studied on finished drying process as well and various finish drying techniques were compared. The summary research finding from these studies are listed below:

- The application of complex solute mixture such as sucrose+dextrose (S:D), sucrose+maltodextrin (S:MD) was successfully demonstrated on microwave osmotic dehydration under continuous flow medium spray (MWODS) condition. This is the first study on the use of these solute mixtures with a MWODS set-up.
- The better ML/SG performance was recorded with S:MD when compared with other solute mixtures such as sucrose (S), sucrose+dextrose (S:D) while using the MWODS system. ML/SG is an indicator of the quality of osmotically dehydrated products, and higher values indicate better quality retention. Again this is the first finding of this kind with MWODS.
- The research findings also concluded that the maltodextrin enriched solute limited the solids uptake when compared with S:D and S solutes. This phenomenon by itself is a confirmation of earlier studies, however this was evaluated for the first time with MWODS. Quantitative superiority with MWODS combination was established for the first time.
- The qualitative analysis showed minor destruction of the pigments along with reduced hardness and chewiness, which can be considered as an advantage for the dehydrated product. The loss of quality characteristics was minimal despite the presence of microwave energy. These results were confirmatory of earlier MWODS, but first time in combination with solute mixtures.
- The use of maltodextrin 10DE along with sucrose improves mass transfer rates during MWODS process. S+MD 10DE gave the best performance leading to highest moisture loss and lowest solids gain and promoting better drying efficiency.
- Upon comparing different grades of maltodextrin solutes, it was found that the solute mixture sucrose+maltodextrin 10 DE (S+MD 10DE) had highest influence on water transfer (k_w) and solids transfer (k_s) coefficient and also it resulted in high NMC along with low NSC at any

given temperature/concentration/time combinations, when compared S+MD 15DE and S+MD 18DE. MD10DE is the grade with highest molecular weight and highest viscosity enhancing influence. Hence this grade was used for further work.

- Both modified first order empirical and Azuara models were effective in modeling the moisture loss and solids gain patterns in MWODS using more complex solute mixtures. These models have been successful in other MWODS applications.
- The CCRD was used successfully to optimize the process of MWODS, where it was concluded that the process can be optimized under then given constraints at 56⁰C, 46% solute concentration and 16% of MD content in sucrose solution.
- Both temperature and concentration of osmotic solution were found to be significant in influencing all outcome responses. Whereas, the MD supplement to sugar in the osmotic solution was found to be a highly significant positive factor for the mass transfer parameters ML, WR, and ML/SG.
- Most importantly, MD incorporation had a negative coefficient or reciprocal influence on SG, which was the significant finding of this study. It was clearly demonstrated that MD incorporation has the ability to restrict solids uptake as well as enhance ML, which in combination helped further enhance the ML/SG ratio, a desirable feature for osmotic dehydration. In addition, the inclusion of MD supplements resulted in better quality products retaining both color and textural attributes.
- The effect of S:MD solute mixture on finished air dried product quality was also studied using CCRD, where each processing factor (osmotic solution temperature, concentration, contact time and flow rate) was optimized to the required processing constraints, where it was found that the solute concentration had significant effect on product quality.
- Different solute mixtures such as S:D, S:MD and S, were also compared at optimum processing conditions and it was recognized that the mangoes treated with S:MD under MWODS followed by air drying produced better results than S:D and sucrose solutes. This is the first time study demonstrating the advantage of using MD moderated sucrose solution under MWODS followed by finish air drying to produce shelf stable dried products.

- The dehydrated product with minimum color change, comparatively firm texture along with high rehydration capacity was obtained by MWODS-air dried samples treated with S:MD solute mixture.
- The application of microwave vacuum drying process was studied on post-MWODS mangoes using S:MD solute, where it was concluded that the MVD power level was found to be one of the significant factor affecting textural and physical properties of the MVD mango product. The application of MD moderated sucrose solution under MWODS with finish vacuum drying technique was studied first time in this research series. The study demonstrated superior product qualities compared to other methods.
- The comparison between the sucrose solute S:MD using various finished dried techniques (air drying, freeze drying, vacuum drying) found that the microwave vacuum dried samples treated with maltodextrin had the product quality which was closest to freeze dried samples. This is the first time study of such comparisons.
- Also, the structural properties of the MVD samples treated with maltodextrin MWODS were found to be porous in structure compared to samples treated with sucrose. Hence, it can be concluded that the maltodextrin moderated sucrose solution during MWODS treatment had more positive impact on microwave vacuum dried mangoes than sucrose solution. Also, the MVD was found to be the best finished drying method after freeze drying.

SUGGESTIONS FOR FUTURE RESEARCH

Although, this research work has demonstrated many interesting findings, there are several topics of studies for future work, as:

- Exploring several other products and solutes while employing MWODS, which includes:
 - a. Explore the application of MWODS on vegetable-based products
 - b. The application of salt-based solutes such as NaCl in ternary or higher order solutions
- Scaling up the process to study the incorporation of bioactive component in the product for nutritional enrichment purpose
- Further investigate the coating application using maltodextrin or other polysaccharides on the samples while applying MWODS process
 - a. The use of various edible coating solutions and investigate the effect on mass transfer behavior as well as product quality
- Further scale up the MWODS-MVD process for more continuous, semi-continuous process to understand the energy utilization during the process. Also, design the process for continuous movement of product or controlling the output of magnetron power for equal energy distribution and better energy consumption.
- Development of the by product from the reusable osmotic solute, such as using the OD solute as a sweetener in confectionary product. Furthermore, investigating the nutritional content in OD solute and reuse it as an ingredient in value added product.

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