DRIED BROCCOLI (*BRASSICA OLERACEA* L. VAR. *ITALICA*) STALK THROUGH APPLICATION OF OSMOTIC DEHYDRATION AND MICROWAVE-ASSISTED HOT AIR DRYING

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ABSTRACT

This thesis focuses on the valorization of broccoli stalk as dried food product rather being discarded in the field. Drying can increase the shelf life of food products by removing the water content to a point where the product is shelf-stable. There are various drying technologies in food processing and recent advent of hybrid system such as microwave-assisted hot air drying is very promising. A study was conducted to compare the drying performance of broccoli stalk slices dried under hot air drying and microwave-assisted hot air drying. The results indicated that the microwave-assisted hot air drying was more efficient since it allowed faster moisture removal. Different treatments of preconditioning drying are suggested to improve the quality of dried product.

Osmotic dehydration is a method of removing water from a product with minimum input of thermal energy. This method is generally used as a pre-treatment before the drying process. It helps improve the product quality and reduce the energy requirements for drying. In this part of the study, we sought to optimize the conditions of osmotic dehydration process on broccoli stalk slices to maximize the water loss while minimizing the solute (sugar) gain. The first series of tests were conducted in a water bath without agitation. The best results were obtained with a solution containing 56% sucrose (w/v) maintained at 42 °C and immersion time of four hours. Under these conditions, broccoli stalk slices showed nearly 61.5% of water loss with a solute gain of 6.7%.

Thereafter, drying characteristics of osmotically dehydrated broccoli stalk slices under microwave-assisted hot air drying were investigated. The results demonstrated that the osmotic dehydration pre-treatment remarkably reduced the drying time while maintaining the dried product’s color and shape as that of the untreated dried broccoli samples. Among the different mathematical models studied, Page model was the best to describe the thin layer drying kinetics of osmotically dehydrated broccoli stalk slices.

During the osmotic dehydration with no agitation, the mass transfers were low resulting in long processing times. Therefore, a dewatering apparatus equipped with a pump for controlling the speed of the solution was designed and constructed. The device was used to study the effects of the flow velocity on the performance of osmotic dehydration process. The results showed that the flow velocity helps in faster rate of water removal while reducing the amount of sucrose gain. The optimum operating conditions were found to be at a temperature of 30 °C with a sucrose concentration of 54 °Brix and it was moving at a speed of 3.5 mm/s. Under these conditions, it only took 120 minutes to remove 65% of the water from the broccoli stalk slices, and sucrose gain was 3.9%.

Osmotic dehydration results in loss of water and sucrose gain in the broccoli stalk slices. These compositional changes alter the dielectric properties (ε' and ε'') of the product and its ability to convert the electromagnetic energy of the microwaves into heat. In this study, it was found that ε' and ε'' of broccoli stalk were dependent on the osmotic dehydration processing parameters. Compared to fresh sample, the ε' decreases and ε'' increases when broccoli stalk is subjected to the osmotic dehydration pre-treatment, resulting in better conversion of microwave energy into heat.

Further study was conducted to evaluate the effects of osmotic dehydration and microwave-assisted hot air drying on the quality of the dried broccoli stalk slices. The parameters used for the evaluation were: vitamin C content, chlorophyll content, total phenolic content, color and texture. It has been demonstrated that when compared to that of the fresh sample, osmotic dehydration pre-treatment resulted in a significant decrease ($p < 0.05$) in vitamin C content, chlorophyll content, and total phenolic content. In addition, the osmotically dehydrated product has led to minimal color change and softer texture. When compared to varying drying temperatures, a drying temperature of 40 °C resulted in arriving at the best quality of the finished product.

The last part of the study focused on the economic aspects of the production process of dried broccoli stalk slices. The parameters selected were raw material availability, process design, energy input, capital inputs, operation costs and environmental benefits. The economic analysis indicated that the proposed production process had a return on investment of 34.3% with a two-year payback period. Considering the positive results of the analysis and environmental benefits, the production of dried broccoli stalk slices seems to be viable on a commercial scale. Furthermore, this concept of osmotic dehydration followed by microwave-assisted hot air drying could eventually be used to process other biological materials that have the potential at the marketplace.

RÉSUMÉ

Cette thèse porte sur la valorisation de brocoli tige en tant que produit alimentaire séché plutôt d'être jeté sur le terrain. Le séchage peut augmenter la durée de vie du produit en supprimant la teneur en eau à un point où le produit est stable. Il existe différentes technologies de séchage dans la transformation des aliments et récente apparition des systèmes hybride tels que le séchage à air chaud assisté par micro-ondes sont très prometteurs. Une étude a été menée pour comparer les performances de séchage de brocoli tige tranches séchées sous séchage à l'air chaud et séchage à air chaud assisté par micro-ondes. Les résultats indiquent que le séchage à air chaud assisté par micro-ondes est plus efficace car il a permis l'élimination rapide de l'humidité. Différents traitements de séchage de préconditionnement sont proposées pour améliorer la qualité des produits séchés.

La déshydratation osmotique est un procédé qui permet de retirer l'eau d'un produit en minimisant l'apport d'énergie thermique. Ce procédé est généralement utilisé comme prétraitement avant le processus de séchage. Il permet d'améliorer la qualité du produit fini et de réduire les besoins en énergie pour le séchage. Dans cette partie de l'étude, nous avons cherché à optimiser les conditions du procédé de déshydratation osmotique sur des tranches de tiges de brocoli afin de maximiser la perte en eau tout en minimisant le gain en soluté (sucre).La première série d'essais a été effectuée dans un bain d'eau sans agitation. Les meilleurs résultats ont été obtenus avec une solution contenant 56% de saccharose (w/v) maintenue à 42 °C et un temps de trempage de quatre heures. Sous ces conditions, les tranches de tiges de brocoli ont perdues près de 61.5% de leur eau avec un gain en saccharose de 6.7%.

Par la suite, les caractéristiques de séchage de osmotique déshydratées tranches brocoli tige a été étudiée sous séchage à air chaud assisté par micro-ondes. Les résultats ont démontré que la déshydratation osmotique du prétraitement réduit

notablement le temps de séchage tout en maintenant la couleur des produits séchés et la forme par rapport à des échantillons de brocoli séchées non traitées. Parmi les différents modèles mathématiques étudiés, le modèle de la page était le meilleur pour décrire les minces cinétiques couche de séchage osmotique déshydratées tranches brocoli tige.

Pendant la déshydratation osmotique sans agitation, les transferts de masse sont bas ce qui résulte en des temps de traitement longs. Par conséquence, un appareil de déshydratation équipée d'une pompe permettant de contrôler la vitesse de la solution a été conçue et construite. L'appareil a été utilisé pour étudier les effets de la vitesse d'écoulement sur la performance du procédé de déshydratation osmotique. Les résultats ont montré que la vitesse d'écoulement contribue à la vitesse plus rapide d'élimination de l'eau, tout en réduisant la quantité de saccharose absorbée. Les conditions optimales d'opération étaient lorsque la solution de saccharose était à 54 °Brix, à une température de 30 °C et qu'elle se déplaçait à une vitesse de 3.5 mm/s. Sous ces conditions, il n'a fallu que 120 minutes pour retirer 65% de l'eau des tranches de tiges de brocoli, et l'absorption de saccharose était de 3.9%.

La déshydratation osmotique résulte en une perte d'eau et un gain en saccharose dans les brocolis traquent les tranches. Ces changements de la composition modifient les propriétés diélectriques (ε' et ε") du produit et son habilité à convertir l'énergie électromagnétique des micro-ondes en chaleur. Dans cette étude, on a constaté que ε' et ε" de brocoli tige était dépendante des paramètres osmotiques de traitement de déshydratation. Par rapport à l'échantillon frais, l'ε 'diminue et ε" augmente lorsque le brocoli tige est soumise à un prétraitement de déshydratation osmotique, résultant en une meilleure conversion de l'énergie micro-ondes en chaleur.

Une étude plus poussée a été menée pour évaluer les effets de la déshydratation osmotique et séchage à air chaud assisté par micro-ondes sur la qualité des tiges séchées de brocoli tranches. Les paramètres retenus pour l'évaluation étaient : la teneur en vitamine C, la teneur en chlorophylle, la teneur en composés phénoliques totaux, la couleur et la texture. Il a été démontré que lorsque comparer au brocoli frais, la déshydratation osmotique, utilisée en prétraitement, entraînait une diminution significative ($p < 0.05$) de la teneur en vitamine C, la teneur en chlorophylle et la teneur en composés phénoliques totaux. En outre, le produit osmotique déshydraté a conduit à un changement de couleur minimale et une texture plus douce. Lorsqu'on les compare à des températures variant de séchage, une température de séchage de 40 ° C a donné lieu à arriver à la meilleure qualité du produit fini.

La dernière partie de l'étude a porté sur les aspects économiques du procédé de production de tranches de tiges de brocoli séchés fais à partir de résidus de brocoli. Les paramètres retenus étaient : la disponibilité des matières premières, la conception des processus, l'apport d'énergie, les entrées de capitaux, les coûts d'exploitation et les bénéfices pour l'environnement. L'analyse économique a indiqué que le procédé de production proposé avait un retour sur investissement de l'ordre de 34.3 % avec une période de récupération de deux ans. En tenant compte des résultats positifs de l'analyse et des avantages pour l'environnement, la production de tranches de tiges de brocoli séchés fais à partir de résidus de brocoli semble être viable à l'échelle commerciale. De plus, ce concept de déshydratation osmotique suivie d'un séchage à air chaud assisté par micro-ondes pourrait éventuellement être utilisé pour traiter d'autres matières biologiques qui ont le potentiel du marché.

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NOMENCLATURE

M	Moisture content
k	Drying rate constant
n	Drying model unit coefficient
a	Drying model unit coefficient
b	Drying model unit coefficient
c	Drying model unit coefficient
t	Time
R^2	Coefficient of determination
χ^2	Reduced chi-square
RMSE	Root mean square error
MR	Moisture ratio
m	Number of variables
D_{eff}	Effective Moisture Diffusivity (m^2/s)
ΔE	Total color difference
L*	Color Properties (Lightness)
a*	Color Properties (Green–red coordinate)
b*	Color Properties (Blue–yellow coordinate)
WL	Water loss
SG	Solute gain
Re	Reynolds number
ρ	Density (kg/m$^{3)}$
v	Velocity (m/s)
D	Diameter (m)
μ	Viscosity (Pa.s)
Q	Flow rate (m^3/s)
A	Cross sectional area (m^2)
w_{wo}	Mass of water in the sample before dehydration (g)

w_t	Mass of sample after dehydration (g)
w_{so}	Mass of the solids in the sample before dehydration (g)
w_{st}	Mass of solids in the sample after dehydration (g)
Rpm	Revolutions per minute
ε'	Dielectric constant
ε''	Dielectric loss factor
$\tan\delta$	Loss tangent
d_p	Penetration depth
λ_0	Wavelength in free space (cm)
Na_2CO_3	Sodium carbonate
CO_2	Carbon dioxide
Q	Heat energy (J)
m	Mass (kg)
C_p	Specific heat capacity (Jkg^{-1}°C$^{-1)}$
Δ	Difference
T	Temperature (°C)
λ	Latent heat of water vaporization (kJkg^{-1})

CONTRIBUTION OF AUTHORS

The research work presented in this thesis was performed by the candidate, including the design of experiment, experimental work, data analysis and preparation of manuscripts for publication under the supervision of Prof. Vijaya Raghavan, who provided the constructive inputs, reviewing and editing of the manuscripts. Mr Yvan Garièpy assisted in the design and construction of the osmotic dehydration equipment system and provided the technical support throughout this study. Dr. Jiby Kudakasseril Kurian helped in results analysis for techno-economical aspects.

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4. Md Salim, N.S., Garièpy, Y., Raghavan, V. 2014. Microwave-assisted Hot Air Drying Characteristics of Osmotically Dehydrated Broccoli Stalk Slices. *Proceedings of the 19th International Drying Symposium,* Lyon, France August 24-27. (ISBN 978-2-7598-1631).

5. Md Salim, N.S., Garièpy, Y., Raghavan, V. 2016. Design of Continuous Flow Osmotic Dehydration and its Performance on Mass Transfer Exchange during Osmotic Dehydration of Broccoli Stalk Slices. *Food Bioprocess and Technology*. **9**(9), 1455-1470 (DOI: 10.1007/s11947-016-1732-z).

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CHAPTER 1

INTRODUCTION

1.1 Motivation of Study

About 1.3 billion tonnes per year of food is lost or wasted, starting at the production stage and ending at the consumer domain (FAO, 2011). At the same time, United Nations predicts that food demand must be increased by 70% to feed the projected world population by 2050. Presently, the world population of 7.3 billion is expected to be increased to more than 9.7 billion by 2050 and according to the *2015 Revision* (United Nations), the population seems to continue to grow by almost 30 million per annum now. Figure 1.1 illustrates the current and the projected world population at major areas. From this figure, it is seen that nearly all the population growth is taking place in the developing countries. According to this population data, the world continues to face the challenge to have an adequate supply of food to feed the fast growing population. This means, we need to produce more food with the existing limited natural resources (land, water, energy and fertilizer).

The potential of reducing waste to combat the food security issues are high, as Lipinski et al. (2013) estimates that by 50% reduction in current food waste, the world would be saving 1314 trillion kcal per year. This represent a reduction of about 22% of the number of additional calories needed to feed the projected population by 2050. Moreover, reduction in food waste leads to more efficiency in economic productivity and reductions in emissions of greenhouse gases that contribute to climate change (WRAP, 2015). Furthermore, Foley et al. (2011) highlighted the reduction of waste and utilization of resources more efficiently; if one can utilize food produced effectively, it would help in world food security in addition to contributing towards the reduction of environmental impacts of agriculture.

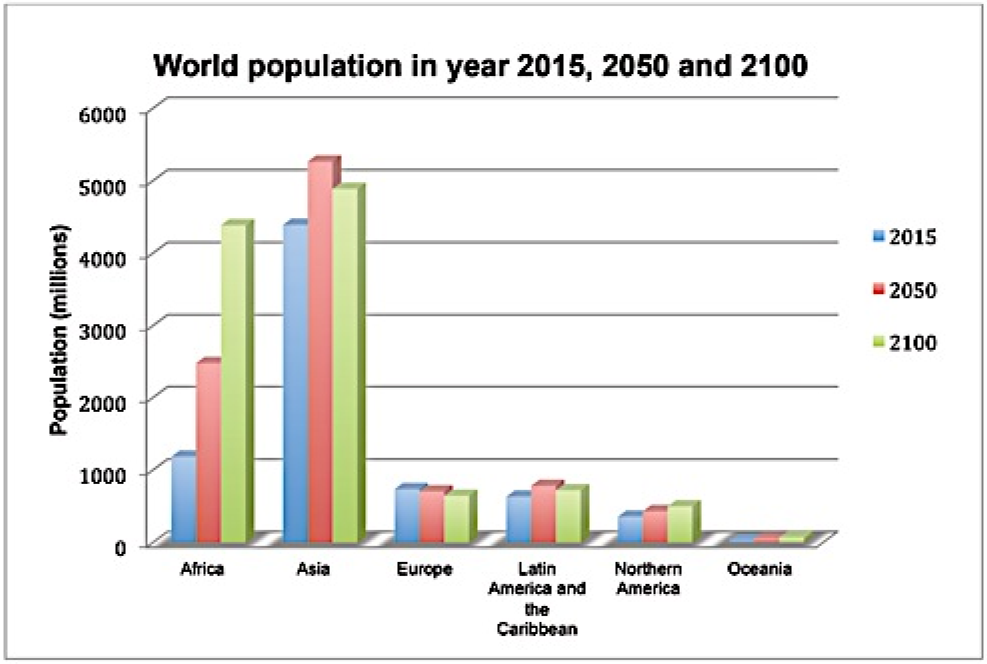


Figure 1.1: World population at different major areas in the year of 2015, 2030 and 2100 (Source: United Nations (2015)).

The amounts of fruits and vegetables are not only increased due to the demand in consumption but it was also found to be the largest food waste contributor. Figure 1.2 presents the estimated global food waste from different food commodities. Fruits and vegetables waste represent 44%, roots and tubers by 20%, cereals by 19% and other commodities also produce significant amount of waste. For example, fruits and vegetables are commonly produced seasonally and overproduction during a favorable season without proper utilization leads to the food waste, especially in tropical countries. In addition to that, there is a significant amount of crop remains were plowed back to the field although they contain sufficient nutritional ingredients. As in food processing industry, when the raw materials enter the production process it produces the desired products and also the waste. An estimated 25 - 30% waste is produced from the processing of fruits and vegetables in the form of pomace, peels, and seeds (Galanakis, 2013).

Due to these compelling circumstances, researchers are working towards reducing the food waste and identifying the potential crop remains and by-products that contain nutritional ingredients which are needed for the population; this leads to the domain of new value added products at the market place (Ayala-Zavala et al., 2011; Đilas et al., 2009; Laufenberg et al., 2003; Peschel et al., 2006). In other words, transformation of these wastes to food can have positive effects towards a sustainable future.

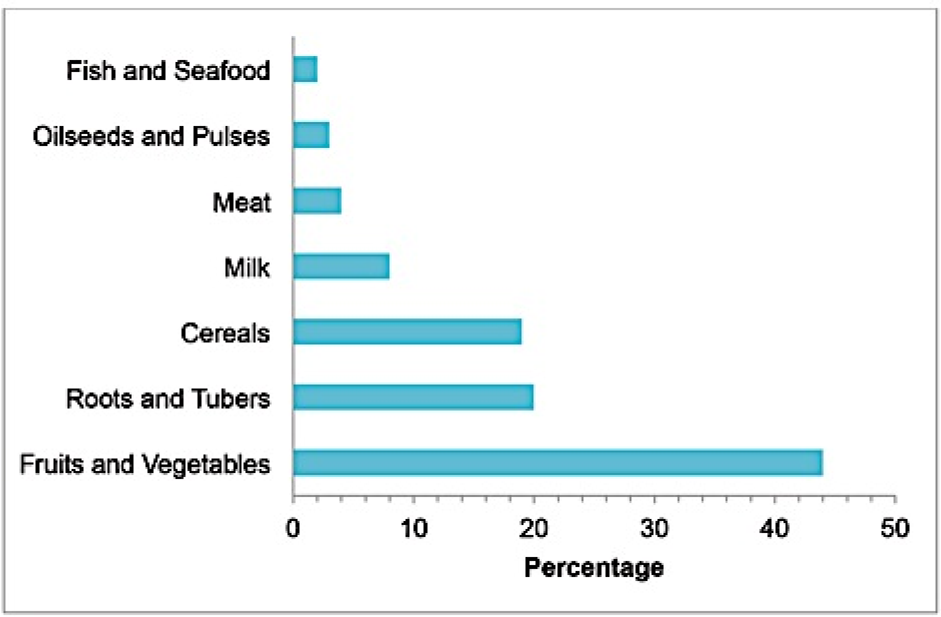


Figure 1.2: Global Food Waste by Commodity (Source: FAO (2011)).

Stem, stalks, and leaves of plants are the common crop remains in fruits and vegetables production. Such examples are common in broccoli industry. Broccoli is a nutritious vegetable belonging to the *Cruciferae* family and has been consumed worldwide. It has been conclusively shown that consumption of broccoli can help reduce the risk for the development of certain forms of cancer (Finley, 2003; Latté et al., 2011).

World statistics on broccoli production are difficult to obtain because it often mixed with cauliflower production. In Canada, broccoli was harvested on 10,025 acres in 2015 with a total production of 41,456 tonnes. About 47.6% of this broccoli's production is from the province of Quebec (Statistics Canada, 2015). Commonly, broccoli products are predominantly from the florets part, which only represent about 25% of the total mass of plant. The leaves and stalks are left as crop remains. Campas-Baypoli et al. (2009) found that the stalks have higher total carbohydrate and crude fiber compared to leaves and florets. Table 1.1 shows the comparative nutritive value between floret and stalk of broccoli and it can be seen that the stalk still contains nutritional value in them that can be further explored for product development rather than farmers' plowing back the crop remains into the soil.

Table 1.1: Nutritional value of broccoli floret and stalk (Singh et al., 2011).

Nutrients	Broccoli Floret (Quantity per 100g)	Broccoli Stalk
Ascorbic acid	100.75 ± 3.10 mg	30.04 ± 1.95 mg
β-caretenoids	7.76 ± 0.73 mg	3.02 ± 0.40 mg
Phenolics	30.07 ± 1.39 GAE	15.04 ± 1.04 GAE
Total antioxidant activity	4.23 ± 0.45 μmol	2.61 ± 0.21 μmol

*GAE=Gallic acid equivalent

Over the recent years, wide variety of snack products such as dehydrated chips made from fruits and vegetables are being produced in conjunction with the usually produced snack products to meet the demands of the society. Consumer behavior market research conducted by NPD Group reported in 2010 that the snacking trends among Canadians has increased significantly since 2000 due to the customers' hectic lifestyle, which makes snack food becoming a meal replacement as it is more convenient and saves time. From the economic point of view, revenues from Canada's snack food industry have increased from $1.5 billion in 2001 to $2.2 billion in 2010

(Canadian Industry Statistics, 2010). Hence, promotion of broccoli stalk as a ready-to-eat product could be an alternative marketing option for the broccoli industry which in turn leads to the minimization of losses and help better economic return for the producers.

To address the contemporary issues of the food security and environment, drying is one of the key unit operation required for producing a newer, acceptable and excellent edible food products. This water removal process is aimed to reduce the water activity of the perishable biological materials in order to produce a shelf-stable product. Commercially, dried products from fruits and vegetables are used as confectionary products, flour, flakes, granulated, powder, additional ingredient of ready-to-eat soup, salads, energy bars, and cereals, and also as snacks products. The diversity of food products has spurred the development of various type of dryers around the world. The classifications of the dryer systems are as listed in Table 1.2 and the most prevalent ones are based on the mode of heat transfer. However, there is no one technique of drying that is applicable for all products because each biological material has their own unique properties and hence the requirements are varied. Thus, there are many studies conducted for various fruits and vegetables using different drying techniques to produce desired dried products as listed in Table 1.3.

Table 1.2: Classification of dryer system (Monica & Cristina, 2008).

Factor	Type
Mode of operation	Batch or continuous
Number of stages	Single or multi-stage
Mode of heat transfer	Conduction, convection, radiation, dielectric heating or combination of different modes
Residence time	Short = below 1 min, medium = 1-60 min, higher = more than 60 min
State of material in dryer	Stationary, moving, agitated, fluidized, and atomized

Table 1.3: Different drying techniques used for production of a variety of dried products.

Drying methods	Products	Reference
Open sun	Raisins	(Doymaz, 2012; Kelebek et al., 2013)
	Dried banana	(Fudholi et al., 2013)
Solar	Jackfruit leather	(Chowdhury et al., 2011)
	Dried chillies	(Fudholi et al., 2014; Kaewkiew et al., 2012)
	Tomato slices	(Kulanthaisami et al., 2010)
Solar-spouted bed	Dried peas	(Sahin et al., 2013)
Hot air	Jackfruit slices	(Saxena et al., 2012)
	Apple leathers	(Demarchi et al., 2013)
Spray	Orange powder	(Cano-Chauca et al., 2005)
	Watermelon powder	(Quek et al., 2007)
	Tomato powder	(Goula et al., 2004)
Fluidized bed	Dried green peas	(Hatamipour & Mowla, 2006)
	Dried green bean	(Senadeera et al., 2000)
Vacuum	Kiwifruit slices	(Orikasa et al., 2014)
	Dried eggplant	(Wu et al., 2007)
Freeze	Freeze-dried broccoli	(Mahn et al., 2012)
	Freeze-dried carrot	(Voda et al., 2012)
Microwave-hot air	Dried okra	(Kumar et al., 2014)
	Carrot slices	(Zhao et al., 2014a)
	Tomato slices	(Workneh et al., 2011)
Microwave-fluidized bed	Apple cubes	(Askari et al., 2013)
Microwave-freeze	Banana chips	(Hao et al., 2014)
	Apple slices	(Duan et al., 2012)
Microwave-vacuum	Mango slices	(Pu & Sun, 2015)
	Dried Lychee	(Duan et al., 2015)
	Dried strawberries	(Borquez et al., 2015)
Osmotic - Microwave-vacuum	Dried Pineapple	(Corrêa et al., 2011)
	Potato cubes	(Sutar et al., 2012)
	Dried Strawberries	(Changrue et al., 2008a)

Over the recent years, microwave drying is one of the potential methods that have been implemented in food industry as it offers a fast volumetric heating and increases the drying rate. From the economic point of view, stand-alone microwave drying is very costly. Hence, to complete the drying process and improve the energy efficiency, a combination of microwaves with other drying techniques is recommended (Chou & Chua, 2001; Orsat et al., 2007). In general, the developments of new attractive dehydrated products using the innovative drying technologies will increase and diversify its availability, minimize the food losses and create additional income for the producing community.

1.2 Research Hypotheses

The broccoli stalk has a potential to be utilized as a dried food product rather being discarded in the field. Microwave hybrid drying can be a promising option to produce good quality of dried broccoli stalk with less energy input. This drying approach will be economically feasible, which in turn help better economic return for the broccoli producers.

1.3 Research Objectives

The main objective of this research was to evaluate the processing conditions that provide positive benefits for the development of dried broccoli stalk product in terms of drying performance, energy consumption and product quality. In accomplishing this, the following specific objectives were determined:

- To study the influence of drying conditions on drying kinetics, moisture diffusivity and physical changes of broccoli stalk slices under hot air drying and microwave-assisted hot air drying. The results are presented in **Chapter 3**.

- To study the effects of operating parameters on the mass transfer exchange during osmotic dehydration of broccoli stalk slices in a conventional static bath method. The results are presented in **Chapter 4**.
- To evaluate the drying characteristics of osmotically dehydrated of broccoli stalk slices under microwave assisted hot air drying. The results are presented in **Chapter 5**.
- To design and construct osmotic dehydration equipment system for continuous flow condition. The results are presented in **Chapter 6**.
- To investigate the mass transfer exchange during osmotic dehydration of broccoli stalk slices under continuous flow condition. The results are presented in **Chapter 6**.
- To determine the effects of osmotic dehydration operating variables on the dielectric properties of osmotically dehydrated broccoli stalk slices. The results are presented in **Chapter 7**.
- To evaluate the drying characteristics and quality changes during the processing of osmo-dried broccoli stalk slices. The results are presented in **Chapter 8.**
- To evaluate the techno-economical aspects of the development of dried broccoli stalk slices using combined osmotic dehydration and microwave-assisted hot air drying. The results are presented in **Chapter 9**.

CONNECTING STATEMENT FOR CHAPTER 2

The motivation for the development of a dried broccoli stalk product and the division of research works that have been implemented to achieve the research goal was presented in **Chapter 1**. In this following chapter, an overview of microwaves, microwave-assisted drying, the osmotic dehydration concept and recent progress in this hybrid drying (osmotic, microwave-assisted drying) are highlighted.

CHAPTER 2

DRIED PRODUCTS THROUGH APPLICATION OF HYBRID DRYING TECHNOLOGY USING COMBINED MICROWAVES AND OSMOTIC PRE-TREATMENT: A REVIEW

Abstract

Hybrid drying has gained interest in the food industry to improve the quality of dried products while substantially saving the energy cost in drying process. Amongst combination drying techniques available nowadays, dried food products produced through combined osmotic dehydration and microwave-assisted drying has a better potential to enhance the energy efficiency of the drying process and provide food at the market place that is acceptable by the consumers. This review provides an overview on principle of microwave heating, microwave-assisted drying, the osmotic dehydration concept, and recent progress in this hybrid drying (osmotic, microwave-assisted drying) in production of dried products. Indeed, preventing losses at all levels of food system could reduce carbon dioxide (CO_2) load loss to the atmosphere.

Keywords: Dried products, Osmotic dehydration, Microwaves, Hybrid drying.

2.1 Introduction

" Simply by removing water from food you can save money, eat better, reduce waste, minimize crop loss, help stabilize the world's food supply, utilize locally grown food, and have a reliable supply of food from one harvest to the next.
Food drying is revolutionary "

~Bell (2008)

Most agricultural products are highly perishable materials and have a short shelf life, commonly only a few days. To prolong the shelf life of a product, it needs to be stored in a proper storage otherwise it easily deteriorates. Improper storage is a major source of food wastage. Statistically, Food and Agriculture Organization of the United Nations estimated that 1.3 billion tonnes of food is wasted per year, globally. This situation is becoming a growing concern as the world is now facing huge challenges to provide more food to feed the projected population by 2100. In dealing with the concern over the food security issue, it is important to reduce food waste and thus leading to more available food.

Drying of food is an important and effective way not only for preservation, but also to provide easy handling and lessening the transportation cost by reduction of bulk density (Raghavan & Orsat, 2008). Moreover, different drying technologies stipulate different drying mechanisms, which result in a variety of food products with different qualities. In that case, drying also provides the opportunity in the creation of various value added products to harvested commodities. For example, wide variety of snack products such as dehydrated chips made from fruits and vegetables are produced in conjunction with the usually produced snack products to meet the demands of the society (NPD Group, 2010). The competition in the market place drives the food processor to explore the possibility of employing new and improved drying technologies for the development of high quality products to meet consumer demands.

On top of that, the selection of a suitable dryer plays an important role in the development of dried products to achieve the desired goal in the drying process as listed in Table 2.1. Meanwhile, the "structural approach" suggested by Baker (1997) for dryer selections are: (1) List all key process specifications; (2) Conduct preliminary selection; (3) Carry out bench scale tests including quality tests; (4) Make economic evaluation of alternatives; (5) Conduct pilot scale trials; and (6) Select the most appropriate dryer types. However, there is no one technique of drying that is applicable for all products because each biological material has its own unique properties and hence the technical requirements are varied.

Table 2.1: The objectives in drying process of food industries (adapted from Chou and Chua (2001)).

Economic consideration	Environmental concern	Product quality aspects
• To reduce operating costs. • To improve the capacity per unit of drying equipment. • To develop simple and reliable drying equipment that requires minimal labor. • To minimize the off-specification product • To develop a stable process that is capable of continuous operation.	• To minimize energy consumption during the drying operation • To reduce environmental impact by reduction of product loss in waste streams.	• To have precise control of the product moisture content at the end of the drying process. • To minimize chemical degradation reactions. • To reduce change in product structure and texture. • To obtain the desired product color. • To control the product density. • To develop a versatile drying process that can produce products of different physical structures for various end-uses.

During drying, exposure of biological products to high temperatures for long periods of time often results in quality degradation. Therefore, there exists varied improved drying technologies for food industries nowadays and hybrid microwave drying is one of the promising options to meet the demands of the food industry. Hybrid drying is commonly defined as a combination of two or more drying methods and recently, various combinations of drying methods for the development of dehydrated fruits and vegetables have been reported. Hybrid microwave drying is the combination of microwave drying with the conventional drying methods which has drawn the attention of researchers in industrial food processing as it offers an efficient and rapid drying process (Raghavan & Orsat, 2008).

Conventional thermal methods such as hot air drying, vacuum drying and freeze drying have low drying rates causing longer drying times, which subsequently influences the energy consumption and product quality. Contrarily, microwave drying could offer a decrease in drying time and improve the final quality of the dried products (Zhang et al., 2006). The rapid energy dissipation throughout the material during microwave drying is an advantage to overcome the limitation of other slow drying processes and improves the final quality of the dried products (Zhang et al., 2006). In addition to that, there has been an increasing interest in combination of osmotic dehydration pre-treatment process with microwave drying to enhance the drying performance and the quality of the dried product (Changrue et al., 2008a; Corrêa et al., 2011; Md Salim et al., 2014; Wray & Ramaswamy, 2013).

Therefore, the objective of this article is to present the overview of microwave-assisted drying, the principle of osmotic dehydration process and the recent progress in the combination of osmotic dehydration with microwave-assisted drying in the production of dehydrated food products.

2.2 Principle of Microwave Drying

The term microwaves are used to describe electromagnetic waves with a frequency range of 300 MHz to 300 GHz with a wavelength range of 1 mm to 1 m. Federal Communications Commission (FCC) allocated the frequency of 915, 2450 and 5800 MHz for heating purposes; to avoid interference between radio and TV waves (Venkatesh & Raghavan, 2004). In North America, 2450 MHz is the most used frequency for microwave processing equipment as it is widely used for domestic application. Microwaves propagate through a material in many ways; part of the energy is reflected, part is transmitted, and part is absorbed by the material (Venkatesh & Raghavan, 2005). It is important to understand the relationship between microwave energy and the properties of material that need to be dried. When the material is exposed to microwaves, two major mechanisms are involved namely ionic conduction and dipole rotation (Orsat et al., 2007). In ionic conduction, the ions oscillate back and forth under the influence of electric field. The movement of the ions collide with the other ions adjacent to them resulting in heat which is generated throughout. While, in dipole orientation, the dipole molecules attempt to follow the rapidly changing electric field. The fast rotating molecules lead to energy loss in the form of heat through molecular friction.

As heat is generated throughout the material in both processes, a temperature gradient occurs where the centre temperature is greater thus forcing the moisture vapour towards the surface of the material (McLoughlin et al., 2000). Hence, these effects depend on the dielectric properties of the material. The dielectric properties normally described in terms of complex permittivity:

$$\varepsilon^* = \varepsilon' - j\varepsilon'' \quad (2.1)$$

The real part, ε' is the dielectric constant that reflects the ability of material to store energy and directly related to strength of polarization, while the imaginary part, ε'' is the dielectric loss factor that measures the energy absorbed from the applied field. The ratio of the dielectric loss factor to the dielectric constant is called the loss tangent or dissipation factor. Dissipation factor is an indicator of material's ability to generate heat (Mudgett, 1990) and calculated as:

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} \tag{2.2}$$

The energy absorption in a unit volume of a dielectric material (Equation 2.3) shows that the power dissipation in microwave heating is proportional to the dielectric loss factor. Briefly, material with high ε'' (known as lossy material) facilitates the absorption of energy during the microwave drying process.

$$P = 2\pi f \varepsilon_o \varepsilon'' E^2 \tag{2.3}$$

Where P is the power dissipated per unit volume (Wm^{-3}), f is the frequency (Hz), ε_o is the absolute permittivity of free space (8.854×10^{-12} Fm^{-1}), ε'' is the loss factor and E is the electric field strength (Vm^{-1}).

Another important factor in microwave drying is the penetration depth, d_p. It is defined as the depth, where the dissipated power is reduced to 37% of the power entering the material surface. The d_p is expressed as:

$$d_p = \frac{\lambda_0}{2\pi\sqrt{2\varepsilon'}}\left(\sqrt{1+\left(\frac{\varepsilon''}{\varepsilon'}\right)^2}-1\right)^{-\frac{1}{2}} \tag{2.4}$$

Where, λ_0 is wavelength in free space.

Dielectric properties of material are dependent on many factors such as frequency, temperature, moisture content, and composition. Table 2.2 provides the values of dielectric properties of various fruits and vegetables at 2.45 GHz reported in the temperature range of 20-25 °C. Although the literature value can be used as guideline, actual measurements are often required because of the variability of composition of the materials.

Table 2.2: Dielectric constant (ε'), dielectric loss (ε''), dissipation factor ($\tan\delta$), and penetration depth (d_p) of various foods at 2.45 GHz.

Fruits or vegetables	Moisture content, % (w.b.)	ε'	ε''	$\tan\delta$	d_p (cm)	Reference
Grapes	80	69.8	17.5	0.25	0.93	(Tulasidas et al., 1995)
Carrots	87.7	66.1	16.3	0.25	0.98	(Changrue et al., 2008b)
Strawberries	93.5	69.1	18	0.26	0.90	(Changrue et al., 2008b)
Mango	86	61	14	0.23	1.09	(Nelson et al., 1993)
Lemon	91	71	14	0.20	1.17	(Nelson et al., 1993)

The progress in drying of food products solely in microwave drying at the industrial level has been relatively slow when compared to laboratory level due to its high initial capital investment (Chou & Chua, 2001; Piotrowski et al., 2004). Besides that, non-uniformity of the electromagnetic field and low penetration depth were also found as drawbacks in applying microwaves as stand-alone drying technique (Shivhare et al., 2009). Thus, combination of microwaves with other conventional drying technique has been extensively studied for lowering the cost and thus overcome the limitation.

2.3 Microwave-assisted Drying

Hot air drying is commonly used method in drying process due to its simple operation. There have been several studies on the development of dehydrated food product using hot air drying (Falade & Solademi, 2010; Tunde-Akintunde & Afon, 2010). During hot air drying, the heat has to diffuse in from the surface of the material. Thus, rapid reduction of the surface moisture may act as water barrier that may lead to case hardening and shrinkage (Gunasekaran, 1999; Zhang et al., 2006). Furthermore, drying with only hot airflow takes a long time and has low energy efficiency and subsequently, may results in quality degradation of dried products especially for heat-sensitive food products. Qing-guo et al. (2006) reported that edamame (vegetable soybean) dried under hot air drying at 70 °C results in degradation of vitamin C and chlorophyll contents, large shrinkage and poor rehydration due to high temperature and longer drying time.

Thus, applying microwave with hot air drying seems to be an applicable approach that can minimize the retreating wet front, maintaining the evaporating surface and perhaps permitting entrainment-evaporation (Raghavan & Orsat, 2008). This will lead to reduction in drying time, improved drying efficiency and minimization of product degradation. The mechanism of heat transfer distribution is illustrated in Figure 2.1. Comparative study conducted by Alibas (2007) shows that combined microwave with hot air drying of pumpkin slices offer a convenient drying method in terms of energy consumption, drying period and color criteria compared to stand alone microwave drying and hot air drying. Dev et al. (2011) studied the drying kinetics of drumstick (*Moringa oleifera*) under microwave-assisted hot air drying. They found that the drying time were reduced more than 80% compared to hot air drying and the product quality being better in terms of color, rehydration rate and retention of volatiles.

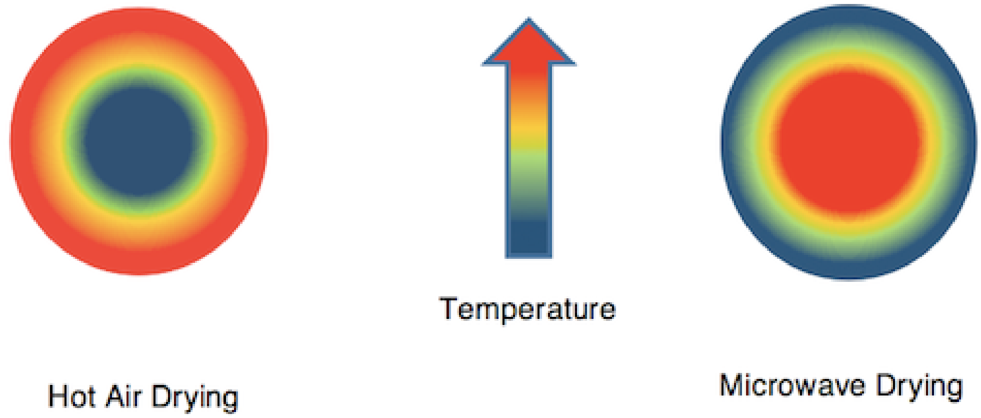


Figure 2.1: Mechanism of heat transfer distribution of hot air and microwave drying.

In vacuum conditions, moisture evaporation proceeds at lower operating temperatures and this condition is favorable for heat sensitive products. Instead of it being used as a stand-alone drying system, combination of vacuum drying with microwave has been successfully employed in drying of various agricultural products. Previous study reported that potato and carrot dried under microwave-assisted vacuum drying showed high rehydration capacity (Markowski et al., 2009; Nahimana & Zhang, 2011). Common parameters to be considered in this drying system are the microwave power and vacuum pressure. Song et al. (2009) reported that in microwave-assisted vacuum drying of potato slices; drying rate was strongly influenced by microwave power but slightly affected by the vacuum pressure. This finding matches previous work reported by Cui et al. (2004) of carrot slices. Combination of hot air and microwave-assisted vacuum drying are also being reported extensively. Recently, application of pulsed spouting on microwave vacuum drying were investigated by Mothibe et al. (2014) for apple cubes. The results showed that the apple cube products were good in color and had higher sensory evaluation scores attributed to microwave-assisted vacuum drying. This hybrid technique could be a promising drying method for developing a high quality dried snacks.

Further drying improvement can be obtained by combination of microwave with fluidized bed drying. Fluidized bed concept is used extensively for the drying of particulate products by passing airflow through a bed of particles. At relatively low velocity, the air can only percolate through the interparticle voids; which is known as packed bed state (Chauk & Fan, 1998). As the air velocity increases, the pressure drop across the bed increases. At a certain air velocity, the bed is fluidized as the drag exerted on the particles counterbalances the weight of the bed. This state is known as minimum fluidization and the corresponding velocity is called the minimum fluidization velocity, U_{mf}. As the gas velocity increased further the U_{mf}, the pressure drop across the bed remains the same. The U_{mf} is obtained from experiments that are dependent on the particle size, column dimension and the operating parameters. Fluidization of particles in drying bed facilitates heat and mass transfer due to constantly renewed boundary layer at the particle surface, subsequently enhancing the product quality. However, the energy efficiency is found to be lower in the falling rate period, which becomes the major drawback of fluidized bed dryers (Chen et al., 2001). Therefore, combining microwave with fluidized bed provides effective way to overcome this limitation. Kaensup and Wongwises (2004) reported that drying time of peppercorn under microwave-assisted fluidized bed was reduced by 80-90% compared to fluidized bed drying along with higher retention of physical texture and color making the process to be more attractive; such results are limited in the literature.

Freeze drying is well known as an excellent dehydration technique for heat-sensitive fruits and vegetables compared to other drying techniques due to its low drying temperature and almost no oxygen involved in the process (Zhang et al., 2006). Freeze drying maintains the structural rigidity of samples during dehydration through the sublimation of frozen water (ice). Thus, the rigidity prevents the structure collapse and results in preserved porous structure and non-shrunken dehydrated products (Ibarz & Barbosa-Canovas, 2003). The freeze-dried products were also reported to have 4-6 times higher rehydration ratio compared to air-dried products (Ratti, 2001). However, this drying technique is commonly recognized as the most expensive drying technique due to longer processing time which required higher energy consumption (Louka & Allaf, 2002). Therefore, an attempt has been made to overcome this mitigation by combination with microwave drying (Hao et al., 2014; Jiang et al., 2010; Wang et al., 2009). Wang et al. (2010b) reported that potato slices dried under microwave-assisted freeze drying reduced 33% of drying time required in stand alone freeze drying process. Interestingly, both methods provide the same quality in terms of vitamin C, sugar and starch losses. Nevertheless, plasma discharge was found to be the limitation in the combination of microwaves with freeze drying (Duan et al., 2010). Plasma discharge happens when the strength of the applied electric field exceeds the threshold value in the vacuum chamber. Ionization of the residual gases present in the vacuum chamber causes in burning of product surface, excessive heating, and energy loss. Hence, controlling the process parameters (vacuum pressure and microwave power levels) can prevent the event during the drying step (Duan et al., 2010).

2.4 Osmotic Dehydration: Pre-treatment Step

In drying process, pre-treatments were applied with aims to accelerate its drying kinetics and improve the final product quality (Lewicki, 1998). Various pre-treatment methods such as blanching, osmotic dehydration, chilling and freezing are used in conjunction with drying process which may exhibit diverse effects on final products. Apparently, osmotic dehydration has gained much attention to assist in drying performance for the development of dehydrated products. Osmotic dehydration is a process that utilizes osmosis phenomenon when samples are immersed in a hypertonic solution. During osmotic process, the two major mass transfer mechanisms occurs where the water outflows from the product and the solute inflows from solution to product concurrently as illustrated in Figure 2.2. In addition of these two major mass transfers, natural substances such as organic acids, vitamins or minerals are also leached out from the food product (Lewicki & Lenart, 1995). This mass transfer exchange can influence the quality of the finished product.

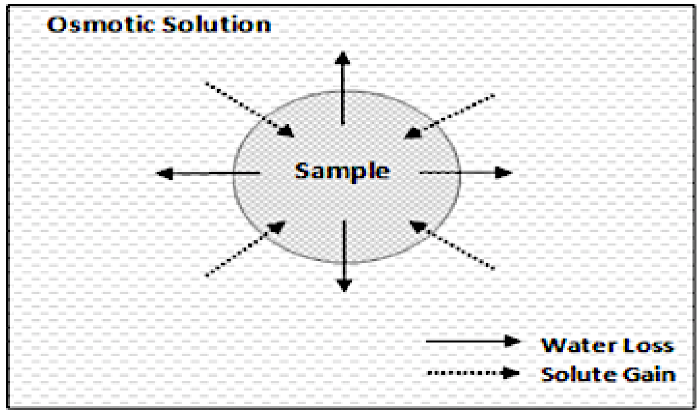


Figure 2.2: Two major mass transfer mechanisms during osmotic dehydration process.

When considering transferring water from solid/liquid to a gas, high-energy consumption is involved. Therefore, by applying osmotic dehydration, water is reduced by up to 50% of the initial mass without a phase change. Lewicki and Lenart (1995) reported that convective drying needs about 5 MJ per kg of evaporated water; while for osmotic dehydration it needs only 0.1-2.4 MJ per kg of removed water. Thus, by applying this process it can enable less energy requirement that has becomes a major concern in drying process in terms of economic consideration (Raghavan & Orsat, 2008). Besides lowering the energy consumption, osmotic dehydration pre-treatment prior to drying can shorten the drying time and improve overall product quality such as color, texture, and aromas in addition to nutritional integrity.

2.4.1 Factors Affecting Mass Transfer During Osmotic Dehydration

There are numerous publications in different types of food materials that have been carried out to evaluate the mass transfer, but it is still difficult to generalize the operating conditions as they are dependent not only on the operating variables but also on the cellular tissue type of the sample itself (Eren & Kaymak-Ertekin, 2007). Under conventional osmotic dehydration system, osmotic agent, solution concentration, temperature and immersion time are the important variables in osmotic dehydration process and combination of these process variables influences the mass transfer (Alam et al., 2010; Changrue, 2006; Fasogbon et al., 2013; Jain et al., 2011).

When food products are immersed in a hypertonic solution, the cell wall of the food products act as semi permeable membrane, which allows smaller molecules to pass through it. The pressure acts as a driving force across the cell wall of the food products called as osmotic pressure, which can be calculated from Van't Hoff equation:

$$\pi = iMRT \tag{2.5}$$

Where *i* is the Van't Hoff's coefficient, *M* is the molarity, *R* is the gas constant (0.082057 L.atm/mol.K), *T* is the absolute temperature (273 + °C) K.

The molarity and moles can be calculated as:

$$Molarity\ (M) = \frac{moles\ (mol)}{volume\ (liter)} \tag{2.6}$$

$$moles\ (mol) = \frac{mass\ (grams)}{molecular\ weight\ \left(\frac{grams}{mol}\right)} \tag{2.7}$$

As osmotic pressure is a colligative property, it means that the osmotic pressure is dependent on the number of dissolved particles. Sugar and salt are the common osmotic agents used in the osmotic dehydration process. Recently, researchers have shown interest in evaluating the effects of sugar alcohol in osmotic dehydration process. Sugar is chemically related to sweet-flavored substances that commonly used as food. When sugar alcohol was introduced as an alternative sweetener, it led to its use in various applications (Mitchell, 2006; Nabors, 1986): (i) To assist in caloric value that may contribute towards a lower risk of over-consumption, obesity and improved survival; (ii) To provide more variety of food and beverage choices; (iii) To aid the management of diabetes; (iv) To reduce risk of dental caries; (v) To enhance the usability of pharmaceuticals and cosmetics; and (vi) To assist in the cost-effective use of limited resources.

However, it is useful to note that in osmotic dehydration process the choice is not simply a matter of it being healthier or healthiest; but the technological properties and economics of sugars and sugar alcohols are important which would impact the mass transfer during the dehydration process. Table 2.3 shows a list of several sugars and sugar alcohols with different molecular weight and solubility properties. Solubility and molecular weight of the osmotic agent have a significant effect on mass transfer during osmotic dehydration process.

Table 2.3: List of molecular weight and solubility of different sugars and sugar alcohols (Davis, 1995; Schiweck et al., 1994).

Sugar and sugar alcohol	Formula	Molecular weight (g/mol)	Solubility ($g/g\ H_2O$)
Sucrose	$C_{12}H_{22}O_{11}$	342.30	0.67
Glucose	$C_6H_{12}O_6$	180.16	0.47
Fructose	$C_6H_{12}O_6$	180.16	0.80
Sugar alcohol			
Xylitol	$C_5H_{12}O_5$	152.15	0.63
Sorbitol	$C_6H_{14}O_6$	182.17	0.69
Mannitol	$C_6H_{14}O_6$	182.17	0.18
Maltitol	$C_{12}H_{24}O_{11}$	344.17	1.50

As shown in Table 2.3, overall the sugars and sugar alcohols have good solubility attributes. Fructose has a higher solubility when compared to sucrose. Among the sugar alcohols, maltitol shows a higher solubility and mannitol has the lowest. On the other hand, it is noteworthy that lower molecular weight of solutes penetrates more easily and rapidly into food products (İspir & Toğrul, 2009; Khan, 2012; Ooizumi et al., 2000; Phisut et al., 2012). As the primary objective of osmotic dehydration process is to maximize the water loss and minimize the solute gain, therefore the sucrose and maltitol was ideal as osmotic agents that can be used in the osmotic dehydration process.

Nevertheless, food products have their own compositional and microstructural complexity that can significantly affect the mass transfer during osmotic dehydration process (Falade & Igbeka, 2007). The shape / geometry of the food products also affect mass transfer due to the variation of the surface area to volume ratio of the product. İspir and Toğrul (2009) evaluated the effect of geometry (whole, half and cube) of apricot and found that the water loss and solute gain was higher for cube shape due to the decrease in dimension and increase in contact surface area.

The solution temperature also markedly affects the osmotic pressure, thus affecting the kinetics of mass transfer during the osmotic dehydration process. Study reported by Eren and Kaymak-Ertekin (2007) stated that higher temperatures result in increase of water loss and solute gain in potato cubes. Similar results are reported by Azoubel and Da Silva (2008) on mangoes. Earlier, Moreira and Sereno (2003) conducted the osmotic dehydration of apple at lower temperatures and found that the mass transfer was higher at 25 °C compared to 5 °C.

Apparently, the mass transfer exchange will proceeds until both water and solute concentrations attain their equilibrium conditions, which can exceed 24-hour immersion time. Most studies on osmotic dehydration have been reported in non-equilibrium conditions, which is about 3 to 5 hours immersion time (Azoubel & Murr, 2004; Ganjloo & Bimakr, 2015; Mundada et al., 2011; Singh et al., 2007). Nevertheless, the initial period of time has the dramatic impact on mass transfer phenomena resulting in higher water removal with minimal solute uptake (Tortoe, 2010).

2.4.2 Methods to Enhance the Rate of Mass Transfer

Despite its simple operating process, osmotic dehydration conducted under conventional static bath commonly results in longer processing time due to low rate of mass transfer. This happens owing to localized area of increased water content around the sample, which creates the external resistance during the osmotic process. Thus, many techniques have been attempted to decrease the boundary layer around the sample and to enhance the mass transfer rate during the process such as by shaking, stirring, flow circulation, ultrasound, high hydrostatic pressure, and centrifugal force.

Ertekin and Cakaloz (1996) conducted a study on osmotic dehydration of peas under static and with an agitation rate of 200 rpm under shaker bath and found that agitation through shaking enhanced the mass transfer compared to static conditions. Moreira et al. (2007) reported the mass transfer kinetics of osmotic dehydration of chestnut conducted at different stirring levels (0, 40 and 100 rpm) of glycerol at 20 °C. This study also reported that the mass transfer was improved under agitated condition. However, the level of stirring shows no significant dependency on the mass transfer process except at low concentration solution. It was explained in this study that the different levels of glycerol concentration might change the hydrodynamic characteristics and thus affect mass transfer process.

Effects of laminar and turbulent flow on mass transfer kinetics of apples has been reported by Mavroudis et al. (1998). This study was operated using agitating vessel with rotation range of 10 to 1400 rpm and the Reynolds number (*Re*) studied was ranging between 350 and 18 500. The results show that, the water loss was found to be higher in turbulent flow compared to laminar flow. Interestingly, the solute gain was not significantly affected when both flows were compared. Further studies conducted on apple were investigated by Moreira and Sereno (2003). In their studies, flow rates between zero to 6.67×10^{-5} m^3/s, corresponding to laminar flow at $Re < 100$ were investigated at temperatures of 5 and 25 °C, respectively. The findings led to the conclusion that osmotic dehydration conducted under continuous flow conditions enhanced the rate of water removal without affecting the solute uptake. However, Li and Ramaswamy (2006) found that mass transfer exchange during osmotic dehydration of apple conducted at flow rates ranging from 330 to 670 ml/min, corresponds to $Re < 135$

was similar to that observed in osmotic dehydration under conventional static conditions. To date, studies on quantification of agitation in osmotic dehydration especially in laminar flow conditions are still limited in the literature.

The use of ultrasound to accelerate the mass transfer process without significant heating has been extensively studied in osmotic dehydration process. Ultrasound is a mechanical wave with frequency ranging from 20 kHz to 100 MHz and the majority of studies are limited to the frequency of 20 to 40 kHz, which is the common frequency used in conventional ultrasonic equipment. In a solid-liquid system, sound waves produce rapid series of alternative compression and expansion of the material called 'sponge' effect. This process is responsible for creation of microscopic channels in material, which reduce the diffusion boundary layer and substantial improvement in the mass transfer (De la Fuente-Blanco et al., 2006; Fernandes & Rodrigues, 2007; Knorr et al., 2004). Kek et al. (2013) reported that application of ultrasound had increased the mass transfer during osmotic dehydration of guava slices. It was found that the water loss of 34% with solute gain of 0.89% could be achieved at a sucrose concentration of 70 °Brix for 60 min of immersion time under ultrasonic bath at 1.75 kW. The influence of ultrasound had also been investigated by Oliveira et al. (2010) during osmotic dehydration of Malay apple. It was found that solute gain and water loss was higher when ultrasound was used compared to non-ultrasound.

Centrifugal force has been found as a technique to improve the mass transfer during osmotic dehydration process. Although still scarce, the most detailed study is reported by Azuara et al. (1996) on potato slices and apple slices. The results show that the samples treated under centrifugal condition during osmotic dehydration process have higher water loss compared to conventional static conditions. Interestingly, solute gain was reduced under centrifuged condition. Thus, these findings provide ideal conditions as a pre-drying stage. It is useful to note that an excessive solute uptake during the osmotic process can block the surface layer of the product and this additional resistance can lower the water diffusion rate during the finish drying process (Menting et al., 1970).

Another interesting technique is through application of high hydrostatic pressure (HPP). HPP is a non-thermal process of food by the application of high pressure commonly applied in the range of 100-800 MPa (Lopes et al., 2010; Nuñez-Mancilla et al., 2013). Compression and decompression taking place during the high pressure process alters the cell wall structure, generates cell permeabilization and cell disruption (Ahmed et al., 2016). In that event, the mass transfer rate was enhanced. In addition, the amount of energy to compress the food is relatively low, thus this technique is found to be more energy efficient than other methods that required heat (Ghani & Farid, 2007). Dalla Rosa et al. (1997) studied the influence of HPP on mass transfer kinetics during osmotic dehydration of kiwifuit slices. The results of their study show that the water loss and solute gain were higher under HPP than at atmospheric pressure. Meanwhile, Sopanangkul et al. (2002) have found that the application of high pressure significantly contributed to acceleration of solute uptake in potatoes. However, it is noteworthy in their study that higher pressure leads to starch gelatinization in potatoes that hinders the mass transfer process.

2.5 Microwave-assisted Drying of Osmotically Dehydrated Product

Application of osmotic dehydration as a pre-treatment prior to microwave-assisted drying is scanty in the literature. By selecting the ideal conditions to achieve higher water loss and minimal solute uptake during the osmotic dehydration process results in modification of dielectric properties and thus offers the possibility of influencing the drying performance and the properties of the dried products. Hence, a considerable amount of literature has been published on determination of optimum conditions for osmotic dehydration using response surface methodology (RSM).

RSM is an effective experimental strategy to optimized conditions for multivariable responses according to the target specifications. With RSM, empirical model for performance prediction that relate the response to the process parameter can be built (Song & Hwang, 2003); and the number of experimental runs needed to evaluate the various operating factors and their interactions can be reduced, which may leads to substantial reduction in the cost and experiment time (Giovanni, 1983; Mohsen et al., 2013). Table 2.4 provides the optimum process conditions reported for various fruits and vegetables to achieve higher water loss and minimal solute gain in osmotically dehydrated products. In general, the optimization of this dehydration process becomes an important strategy to achieve energy conservation for further drying.

This hybrid (osmotic, microwave-assisted) drying is capable of producing better quality dehydrated product with lower energy consumption due to decrease in drying time. Table 2.5 shows the effects of osmotic dehydration pre-treatment on different microwave-assisted drying methods. Parameters such as microwave power, air velocity and temperatures of osmotically dehydrated bananas have been investigated by Pereira et al. (2007). The results demonstrated that increasing the microwave power level results in reduction in drying time. However, it is necessary to control the microwave power during the finish drying as higher microwave power leads to rapid temperature rise in the product and thereby, resulting in charring of the dried products. Meanwhile, lower air temperature with higher air velocity is suggested to improve the quality of dried products. While the study of Therdthai et al. (2011) showed that an increase in microwave power from 4.8 to 6.8 W/g improved the drying rate of osmotically dehydrated mandarin cv. (*Sai-Namphaung*); but the quality retention in terms of *β*-carotene was significantly decreased. Further, Beaudry et al. (2003) reported that high power density (125 W/g) resulted in the burning of some osmotically dehydrated cranberries. However, it should be noted the power rating used was very high and it could be detrimental to the quality of the product.

Table 2.4: Optimum conditions for osmotic dehydration as pre-drying stage of various food products.

Produce	Optimum conditions	WL (%)	SG (%)	Reference
Papaya cube	60 °Brix, 37 °C, 4.25 hours, solution to product ratio of 4:1 (w/w)	28	4	(Jain et al., 2011)
Peach slices	69.9 °Brix, 37.56 °C, 3.97 hours	28.42	8.39	(Yadav et al., 2012)
Button Mushroom	16.5% brine concentration, 44.89˚C, 47.59 min.	44.55	2.98	(Mehta et al., 2012)
Ginger slices	50 °Brix + 7.31% NaCl, 30°C, 102 min, solution to product ratio of 8:1 (w/w)	58.8	12.56	(An et al., 2013a)
Carrot cubes	50 °Brix + 15% NaCl salt (w/v), 54.8 °C and 120 min.	42.86	12.9	(Singh et al., 2010)
Aonla Slices (Indian gooseberries)	59° Brix, 51°C, 60 min, solution to fruit ratio of 4:1 (w/w)	44	4.59	(Alam et al., 2010)

Table 2.5: Effects of osmotic dehydration on different microwave-assisted drying.

Drying methods	Produce	Osmotic dehydration effects	Reference
Microwave-assisted hot air drying	Apple	Drying time is reduced; improved appearance; softer rehydrated texture; lower rehydration capacity.	(Prothon et al., 2001)
	Mushroom	Drying time is slightly reduced; uniform heating; improved rehydration properties; less shrinkage and higher porosity.	(Torringa et al., 2001)
	Red sweet pepper	Faster drying time; enhanced the moisture diffusion.	(Swain et al., 2012)
Microwave-assisted vacuum drying	Pineapple	Reduced shrinkage; improved rehydration capacity; softer texture.	(Corrêa et al., 2011)
	Apple and Strawberries	Drying time is reduced; superior quality in terms of color, taste, structure and volume; vitamin C retention was around 60%.	(Erle & Schubert, 2001)
	Strawberries	Drying time was same with untreated sample but product quality are better in terms of color, sensory, rehydration and toughness.	(Changrue et al., 2008a)
	Papaya	Rapid water removal during drying; highly porous microstructure; higher rehydration rate.	(Nimmanpipug et al., 2013)
Microwave-assisted freeze drying	Potato	Drying time is reduced; lower rehydration capacity; increased in hardness and crispness.	(Wang et al., 2010a)

Another concern when applying osmotic dehydration as a pre-treatment in the processing line of dried product is the osmotic solution management after the process. If the diluted solution is discarded as waste at the end of the process, it could greatly increase the cost of osmotically dehydrated products. Besides, it also results in loss of valuable natural substances such as vitamins, pigments, and minerals that leaches from the food sample to the solution during the osmotic process. In addition, diluted sucrose solution are susceptible to fermentation when it is kept for more than one week at room temperature (Shi & Xue, 2009). Thus, it is necessary to find a variety of applications for the used osmotic solution to make the process profitable with minimum waste.

Reconstitution of the diluted osmotic solution by either evaporation or addition of solute has been suggested previously to make the process economically feasible (Shi & Xue, 2009; Valdez-Frugoso et al., 1998). Furthermore, reusing osmotic solution over a number of osmotic cycles has also been investigated. Valdez-Frugoso et al. (1998) reused osmotic solution for 20 batches of apple cubes, by reconcentrating the diluted solution to its initial concentration. According to the results of this study, to keep the initial mass of the solute in the solution constant for each batch, the evaporation method could be advantageous and it could bring more savings in terms of sugar use. Nevertheless, García-Martínez et al., (2002) reported reuse of osmotic solution of kiwifruit for 10 cycles without being concentrated seems not to have major effects on dehydration process, color properties and acceptable microbial contamination.

Recently, Wray and Ramaswamy (2016) found that the reuse of osmotic solution by 10 cycles resulted in constant drying performance of cranberries under microwave-assisted vacuum dryer. It is also possible to use osmotic syrup in fruit canning, jam making, aromatic flavor production and also as natural additives in food and pharmaceutical industry (Morales et al., 2005; Shi & Xue, 2009). Grabowski et al. (2007) suggested that recycle of osmotic syrup containing anthocyanin from osmotic dehydration of blueberries could be used as table syrup. An attempt has been made by Aachary and Prapulla (2009) to provide better alternatives by adding value to osmotic syrup for production of fructooligosaccharides (FOS). FOS is known to have a prebiotic and other beneficial health effects with high market demand in neutraceutical food sector.

2.6 Conclusion

The development of dried food products using innovative drying technology provide an option for increasing food availability, reduction in food waste and also generating more incomes to the producers. Osmotic pre-treatment has shown a great improvement in reducing the drying time of microwave-assisted drying. In general, energy conservation can be achieved in drying process owing to the fact that the water from the food sample is removed partially without the phase change during the pre-treatment step. So far, in terms of quality aspects, most studies on this hybrid drying have shown better quality products in terms of physical aspects. However, far too little attention has been paid to nutritional content. In addition, the cost of applying this combination technique on industrial scale also needs to be examined because it is critical to its potential for commercial exploitation.

CONNECTING STATEMENT FOR CHAPTER 3

From the review presented in **Chapter 2**, it is clear that there are various pathways to perform the microwave hybrid drying for development of dried products. Thus, in the following chapter, first attempt has been made to evaluate the drying performance of untreated broccoli stalk slices dried under hot air drying and microwave-assisted hot air drying.

CHAPTER 3

HOT AIR DRYING AND MICROWAVE-ASSISTED HOT AIR DRYING OF BROCCOLI STALK SLICES (*BRASSICA OLERACEA* L. VAR. *ITALICA*)

Abstract

Drying characteristics of broccoli stalk slices under hot air drying and microwave-assisted hot air drying at temperatures of 40, 50 and 60 °C is presented. In addition, the color and shape changes of the dried sample were compared. Midilli et al. model shows a good agreement with the experimental data obtained for both drying systems. The calculated effective moisture diffusivity varied from 2.92 x 10^{-8} to 7.91 x 10^{-8} m^2/s under hot air drying and 6.64 x 10^{-8} to 13.31 x 10^{-8} m^2/s under microwave-assisted hot air drying. Less changes in color was observed under microwave-assisted hot air drying owing to its lower temperature condition. Meanwhile, higher circularity of the shape was observed under hot air drying. According to these results, microwave-assisted hot air drying promotes faster drying time with higher moisture diffusion; however, pre-treatment prior to this drying process is suggested to enhance the quality of the final dried product.

Keywords: Broccoli stalk, Drying, Mathematical modelling, Moisture diffusivity, Product quality.

3.1 Introduction

Broccoli is a nutritious cool-weather cultivated crop and it consumption has increased due to its numerous health benefits. Most of broccoli production has been marketed as either fresh or frozen products. However, these products are predominantly from the florets part. Meanwhile the stalks are known to have much food value, which needs to be exploited for further use. Study done by Singh et al. (2011) and Bekhit et al. (2013) found that broccoli stalk contains the nutritional value that can be used for product development. Hence, the expansion opportunity in the marketing of broccoli stalks exists.

Broccoli stalk consists of vascular and parenchyma cells, and these cell walls play a role in the integrity of elastic properties (Müller et al., 2003). A study conducted by Jin et al. (2012) shows that an anisotropic shrinkage occurred during drying of broccoli stalk due to the transport barrier formed by the vascular ring which are more elastic compared to the core. Therefore, anomalous drying behavior occurred during the drying process. Due to this particular circumstance, skin peeling process becomes an important step. During the sample preparation process, elimination of the effect of the area-dependent elasticity attributes is necessary and hence, establishment of a uniform moisture distribution throughout the drying process of the broccoli stalk becomes a challenge.

Conventionally, hot air drying is used for drying food products due to its simple operation. However, hot air drying alone is a time-consuming process caused by higher latent heat of vaporization requirement where heat has to diffuse in from the surface of the material. Whereas, in the microwave drying, heating occurs inside the material that creates a total pressure gradient which leads to a rapid water movement towards the surface (Raghavan & Orsat, 2008). However, from the economic point of view, stand-alone microwave drying is very costly. Therefore, the utilization of both drying methods becomes a practical alternative to take advantages of improved drying efficiency while totally minimizing the product degradation.

Drying is a complex phenomenon that involves simultaneous heat and mass transfer for different materials. Thus, it requires simple representations to predict the drying behavior, and for optimizing the drying parameters. Generally, drying process is modeled as either distributed models or lumped parameter models. Distributed models consider both moisture and temperature gradient of gases and solids during drying. Meanwhile, lumped parameter models assume a uniform moisture and temperature distribution in the solid that equal the air drying temperature. Thin layer drying models are a lumped parameter models that have been widely used to estimate the drying time and establish the drying curves of food slices or a layer of sample particles (Akpinar, 2006; Amiri Chayjan et al., 2015; Doymaz, 2009; Fan et al., 2014). Due to its thin structure, the particle can be assumed to have a uniform temperature distribution during the drying process.

Moisture diffusion strongly depends on moisture content, temperature and product's structure (Saravacos, 2001). In food drying, the moisture diffusion is controlled by several mechanisms such as molecular diffusion, caplillary flow, Knudsen flow, hydrodynamic flow, or surface diffusion (Hui, 2008). For all these possible transport mechanisms, it is difficult to evaluate the effect of an individual mechanism associated with the moisture transport. Hence, a lumped parameter model concept where the rate of moisture movement of these various mechanisms during the entire course of drying is useful which is associated with the effective moisture diffusivity, D_{eff} (Erbay & Icier, 2010; Seth & Sarkar, 2004). The value of D_{eff} have been reported on various vegetables and fruits such as potato (Singh et al., 2013); pineapple (Olanipekun et al., 2014); and peaches (Doymaz, 2014b).

In terms of food quality, color is an important visual sensation that plays an important role in influencing consumer perception and acceptance of a dried product.

Color pigment in agricultural products are strongly dependent on the drying conditions because they are unstable and participate in different chemical and biochemical reactions (Wilska-Jeszka, 2002). Therefore, a suitable drying process is the one which can retain much of the natural color of the fresh products. Food shape is also one of the most common indicators on quality of the dried product. During drying, the rapid water evaporation causes shrinkage thus changing the original shape. The shrinkage can be evaluated by direct measurement using caliper or micrometer. Besides, apparent volume can also be evaluated, which can be achieved by the Archimedes principle. In recent years, application of image analysis for shrinkage evaluation has been of great interest with researchers (Hosseinpour et al., 2014; Nahimana & Zhang, 2011). These tools offered a possibility for an alternative approach for measurement as it can be applied easily to determine the shape of the food.

So far, evaluation on drying characteristics of broccoli stalk is still scarce in the literature. Therefore, the present study was conducted with the following objectives:

(1) To study the effect of drying temperatures on the drying behavior of broccoli stalk slices under hot air drying and microwave-assisted hot air drying;

(2) To evaluate a suitable thin-layer drying model of the experimental drying data;

(3) To calculate the effective moisture diffusivity; and

(4) To evaluate the color and shape changes of the dried broccoli stalk slices.

3.2 Materials and Methods

3.2.1 Sample Preparation

The broccoli (*Brassica oleracea* L. var. *italica*) was purchased from a local market in Sainte Anne de Bellevue, QC, Canada (Figure 3.1). The stalks were separated from the florets, washed and peeled. The peeled stalk was then sliced to a uniform shape with a diameter of 23 mm and a thickness of 6 mm. Moisture content of samples was determined by drying to constant mass in a hot air oven at 105 °C for three hours (AOAC 935.29) (AOAC, 2000).

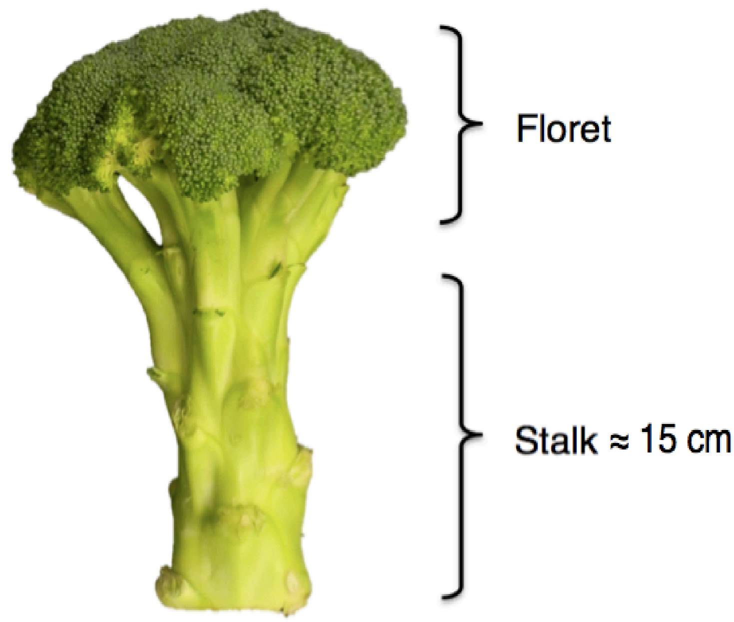


Figure 3.1: The broccoli floret and stalk.

3.2.2 Experimental Setup

Drying experiments were performed in an automated microwave-assisted laboratory scale dryer (Figure 3.2). The dryer consists of a 2,450 MHz microwave generator with 3 tuning screws for adjusting and minimizing the reflected power, air blower connected to the bottom of microwave cavity and fibre optic temperature sensor for monitoring the temperatures of the samples. The dimension of microwave cavity is about 47 x 47 x 27 cm. This dryer is also equipped with a data logger weighing system.

3.2.3 Experimental Procedure

In each experiment, about 50 g of broccoli slices were put in a sample holder, spread in one layer and placed inside the microwave cavity. The power of the dryer was set to 100 Watts and the superficial air velocity to about 1.4 m/s. The blower supplies the air to the microwave cavity. Air supplied by the blower was heated to the required temperature (40, 50 and 60 °C). The temperature of the samples was monitored by fibre optic temperature sensor and the mass of the sample was recorded by the computer via data acquisition system (Hewlett-Packard, USA). For hot air drying, the 50 g of broccoli slices was dried in the same dryer without the application of microwave energy at the set drying temperatures. All samples for both drying methods were dried until it reached moisture content of 20% w.b.

3.2.4 Analysis of Drying Data

The change in moisture during drying will be expressed by the moisture ratio:

$$\text{Moisture Ratio (MR)} = \frac{M_t - M_e}{M_o - M_e} \tag{3.1}$$

Where M_t is the moisture content at time t_i, M_o is the initial moisture content and M_e is the equilibrium moisture content. However, during the drying process, the M_e is negligible because M_e is too small to be quantified. Therefore, MR is determined as:

$$\text{MR} = \frac{M_t}{M_o} \tag{3.2}$$

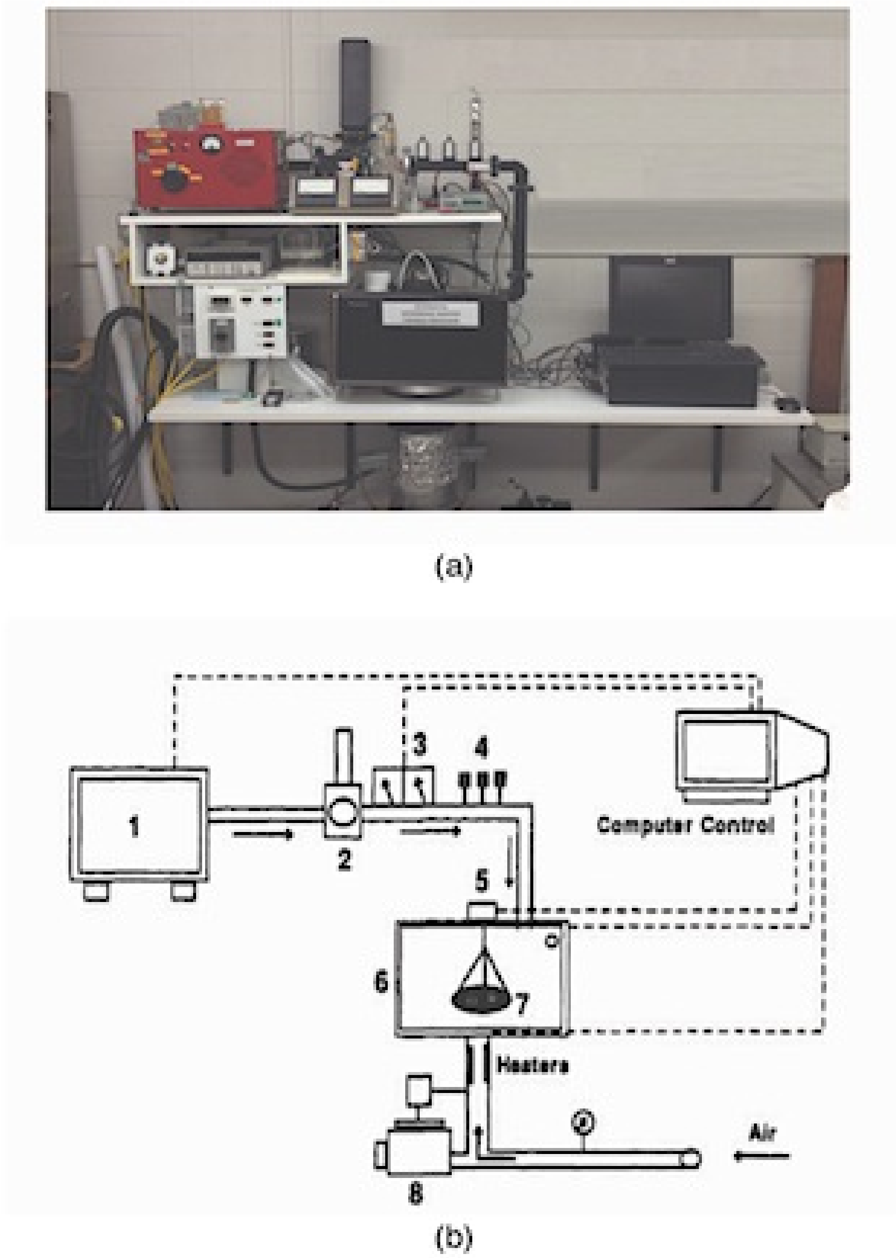


Figure 3.2: (a) Automated microwave-assisted laboratory dryer; (b) Schematic diagram of microwave-assisted laboratory dryer (1: microwave generator, 2: circulator, 3: power meters, 4: tuning screws, 5: strain gauge, 6: microwave cavity, 7: sample holder and 8: blower).

In addition to detailed experimental work, modelling steps were carried out. Thin layer drying model used in the analysis of drying characteristics mainly fall into three categories; theoretical, semi-theoretical or empirical. Some semi-theoretical models that widely used to match in the experimental drying curves are given in Table 3.1 (Akpinar, 2006; Menges & Ertekin, 2006; Nair et al., 2012; Singh et al., 2013; Yaldiz et al., 2001).

Table 3.1: Thin layer drying models used for drying kinetics of broccoli stalk slices.

Model no.	Model Name	Model Equation	Equation no.
1	Newton	$MR=exp(-kt)$	(3.3)
2	Page	$MR=exp(-kt^n)$	(3.4)
3	Modified Page	$MR=exp(-(kt)^n)$	(3.5)
4	Handerson & Pabis	$MR=a\ exp(-kt)$	(3.6)
5	Logarithmic	$MR=a\ exp(-kt)+c$	(3.7)
6	Midilli et al.	$MR=a\ exp(-kt^n)+bt$	(3.8)

Where, *k* is the drying rate constant; *n, a, b* and *c* are the regression coefficient and *t* is the time (min).

The primary criterion for selecting the best equation to describe the drying curve equation is the highest coefficient of determination (R^2) followed by the lowest reduced chi-square (χ^2) and root mean square error ($RMSE$) values obtained from the curve fitting. These comparison parameters calculated are as follows:

$$R^2 = 1 - \frac{\sum_{i-1}^{i=n}\left(MR_i^{exp} - MR_i^{pred}\right)^2}{\sum_{i=n}^{i=n}\left(MR_i^{exp} - \overline{MR}^{exp}\right)^2} \quad (3.9)$$

$$\chi^2 = \sum_{i-1}^{i=n} \frac{(MR_i^{pred} - MR_i^{exp})^2}{n-m} \tag{3.10}$$

$$RMSE = \sqrt{\frac{1}{n}\sum_{i=1}^{n}(MR_i^{pred} - MR_i^{exp})^2} \tag{3.11}$$

Where *n* is the number of observations, MR_i^{pred} is the *i*th predicted dimensionless MR, $MR_i^{exp}$is the *i*th experimental dimensionless MR, *m* is the number of variables and $\overline{MR}^{exp}$ is the mean of the experimental dimensionless MR.

3.2.5 Calculation of Effective Moisture Diffusivity

The experimental drying data for the determination of effective moisture diffusivity were estimated by using Fick's diffusion model. The general series of Fick's second law for slab sample is given as follows (Crank, 1975) :

$$MR = \frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{2n+1}\exp\left(-(2\mathrm{n}+1)^2\frac{\pi^2 D_{eff}t}{4L^2}\right) \tag{3.12}$$

Where D_{eff} is the effective moisture diffusivity (m^2/s), and L is the half-thickness of the broccoli slices (m) and n is the positive integer. Equation 3.12 can be further simplified in logarithmic forms as in Equation 3.13 and the effective moisture diffusivity can be estimated from the slope of linear plot.

$$\ln(MR) = ln\left(\frac{8}{\pi^2}\right) - \left(\frac{\pi^2}{4L^2}D_{eff}t\right) \tag{3.13}$$

3.2.6 Colorimetric Parameters

The color change in the samples was analyzed in terms of L*, a* and b* color scales. The parameters L* (lightness), a* (green–red coordinate) and b* (blue–yellow coordinate) were obtained by using a tristimulus colorimeter (CR-310, Minolta Co. Ltd., Japan). The total color change (ΔE) is a single value which takes into account the differences between the L*, a* and b* values of the dried and fresh sample. ΔE is calculated using the Hunter-Scotfield equation (Equation 3.14); where $\Delta a^* = a^* - a^*_0$; $\Delta b^* = b^* - b^*_0$; and $\Delta L^* = L^* - L^*_0$, subscript 0 represents the fresh sample.

$$\Delta \mathrm{E} = \sqrt{(\Delta \mathrm{a})^2 + (\Delta \mathrm{b})^2 + (\Delta \mathrm{L})^2} \qquad (3.14)$$

3.2.7 Shape Determination

Image analysis was performed to evaluate the final shape of the dried broccoli stalk slices under different drying conditions. Top images of dried samples were captured using a digital camera with high-resolution pixels. The images were then analyzed using the software *ImageJ* version 1.43 (National Institute of Health, USA). Several geometrical parameters that were considered in this study are surface area, perimeter and circularity. In image analysis terms, the area was defined as the number of pixels contained within its boundary and the parameter is the length of its boundary (Yan et al., 2008). Whereas, circularity is defined as;

$$\text{Circularity} = \frac{4\pi \text{ x Area}}{\text{Perimeter}^2} \qquad (3.15)$$

3.2.8 Statistical Analysis

All experiments were conducted in three replicates and the means were analyzed using analysis of variance (ANOVA). Duncan's multiple range test was used to identify significant difference ($p < 0.05$) among samples for each attribute.

3.3 Results and Discussion

3.3.1 Drying Kinetics and Mathematical Modelling

Drying kinetics of broccoli stalk slices at different drying temperatures under hot air drying and microwave-assisted hot air drying are presented in Figure 3.3. For both drying methods, air drying temperatures have a determinant role in drying time of broccoli stalk slices. The decrease in drying time was observed when the air drying temperature was increased. These results are consistent with findings of past studies on carrot (Zielinska & Markowski, 2010); mushroom (Tulek, 2011); and blueberries (López et al., 2010).

Under hot air drying, the drying time required to lower the moisture ratio to a given level at 40 °C was approximately twice that required at drying temperature of 60 °C. The same trend in the relationship between temperature and drying time was observed under microwave-assisted hot air drying and that the drying time at drying temperatures of 40 °C was approximately twice that required at drying temperature of 60 °C. Meanwhile, by comparing both drying methods, the drying time was reduced by 42-55% when broccoli stalk slices dried under microwave-assisted hot air drying compared to hot air drying alone. It is clearly evident that involvement of microwaves energy during drying process increased the moisture evaporation of water molecules. Similar findings have been reported for flax straw (Nair et al., 2012); Moringa oleifera (Dev et al., 2011); and pumpkin slices (Alibas, 2007).

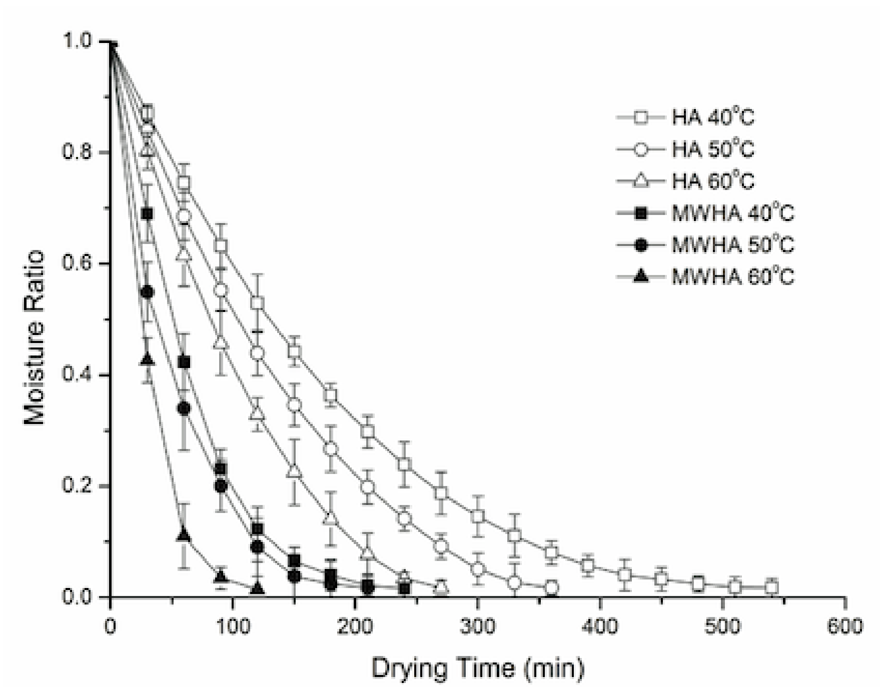
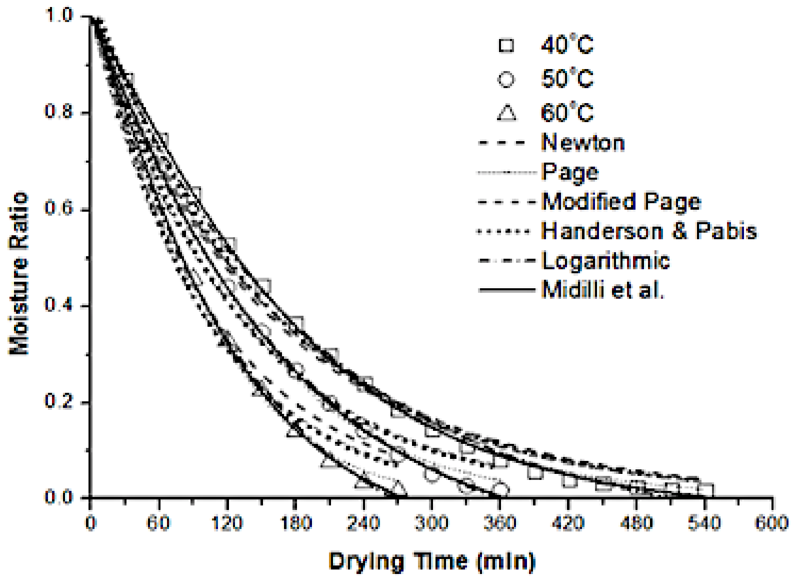
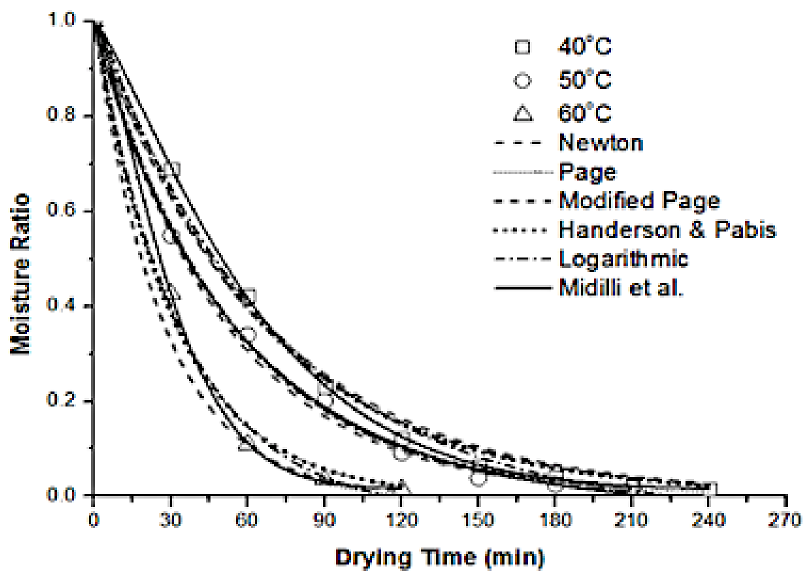


Figure 3.3: Drying kinetics of broccoli stalk slices under hot air drying (HA) and microwave-assisted hot air drying (MWHA) at 40, 50 and 60 °C.

Figures 3.4 (a) and (b) present the graph fitting of non linear regression analysis of six thin layer mathematical drying models to the experimental data. The statistical analysis of this curve fitting are shown in Table 3.2 for hot air drying and Table 3.3 for microwave-assisted hot air drying. For hot air drying, it was determined that the R^2 value was highest and χ^2 and $RMSE$ were lowest for the Midilli et al. model at all drying temperatures. For microwave-assisted hot air drying, all the six models provided an excellent fit to the experimental data with a value of R^2 greater than 0.99. Among them, Midilli et al. model's performed the best fit with R^2 value of 0.9999 with the lowest χ^2 and $RMSE$ at 40, 50 and 60 °C. Based on these results, it can be concluded that Midilli et al. model is the most suitable thin layer drying model used in describing the drying characteristics of broccoli stalk slices



(a)



(b)

Figure 3.4: Mathematical modelling of thin layer drying kinetics of broccoli stalk slices at different temperatures under: (a) Hot air drying and (b) Microwave-assisted hot air drying.

Table 3.2: Model parameters and performance of the thin layer model of hot air drying at different drying temperatures.

Drying Temperature	Model No.	Coefficient		R^2	χ^2	$RMSE$
40°C	*1*	*k=0.0059*		*0.9893*	*0.0010*	*0.0322*
	2	*k=0.0020*	*n=1.2053*	*0.9991*	*9.7526x10⁻⁵*	*0.0099*
	3	*k=0.0062*	*n=1.0000*	*0.9881*	*0.0012*	*0.0350*
	4	*a=1.0498*	*k=0.0062*	*0.9921*	8.1374×10^{-4}	*0.0285*
	5	*a=1.1013*	*k=0.0051*	*0.9981*	2.0721×10^{-4}	*0.0144*
		c=-0.0781				
	6	*a=0.9936*	*k=0.0023*	*0.9996*	5.1181×10^{-5}	*0.0072*
		n=1.1744	$b=-3.9170\times10^{-5}$			
50°C	*1*	*k=0.0075*		*0.9855*	*0.0015*	*0.0021*
	2	*k=0.0023*	*n=1.2305*	*0.9976*	2.7887×10^{-4}	*0.0167*
	3	*k=0.0060*	*n=1.0000*	*0.9393*	*0.0070*	*0.0837*
	4	*a=1.0465*	*k=0.0078*	*0.9988*	*0.0013*	*0.0367*
	5	*a=1.1679*	*k=0.0056*	*0.9993*	8.8652×10^{-5}	*0.0094*
		c=-0.1559				
	6	*a=0.9986*	*k=0.0040*	*0.9997*	3.8536×10^{-5}	*0.0062*
		n=1.1167	$b=-1.8316\times10^{-4}$			
60°C	*1*	*k=0.0097*		*0.9818*	*0.0021*	*0.0461*
	2	*k=0.0024*	*n=1.2880*	*0.9980*	2.6151×10^{-4}	*0.0162*
	3	*k=0.0090*	*n=1.0000*	*0.9769*	*0.0030*	*0.0550*
	4	*a=1.0481*	*k=0.0102*	*0.9850*	*0.0020*	*0.0444*
	5	*a=1.2015*	*k=0.0070*	*0.9985*	2.2435×10^{-4}	*0.015*
		c=-0.1864				
	6	*a=0.9980*	*k=0.0036*	*0.9997*	5.2222×10^{-5}	*0.0072*
		n=1.1829	$b=-2.1382\times10^{-4}$			

Table 3.3: Model parameters and performance of the thin layer model of microwave-assisted hot air drying at different drying temperatures.

Drying Temperature	Model No.	Coefficient		R^2	χ^2	$RMSE$
40°C	1	k=0.0155		0.9959	9.9473×10^{-4}	0.0315
	2	k=0.0055	n=1.2390	0.9999	3.2941×10^{-5}	0.0057
	3	k=0.0151	n=1.0000	0.9957	0.0012	0.0346
	4	a=1.0299	k=0.0159	0.9965	9.8123×10^{-4}	0.0313
	5	a=1.0611	k=0.0143	0.9978	7.288×10^{-4}	0.0270
		c=-0.0409				
	6	a=0.9992	k=0.0049	0.9999	1.6693×10^{-5}	0.0041
		n=1.2655	$b=4.160 \times 10^{-5}$			
50°C	1	k=0.0189		0.9990	2.3778×10^{-4}	0.0154
	2	k=0.0183	n=1.0117	0.9990	1.9863×10^{-4}	0.0141
	3	k=0.0198	n=1.0000	0.9991	2.5606×10^{-4}	0.0160
	4	a=0.9991	k=0.0192	0.9993	2.0202×10^{-4}	0.0142
	5	a=1.0078	k=0.0186	0.9994	2.0009×10^{-4}	0.0152
		c=-0.0116				
	6	a=0.9986	k=0.0037	0.9999	3.8536×10^{-5}	0.0062
		n=1.1167	$b=-1.8316 \times 10^{-4}$			
60°C	1	k=0.0318		0.9973	9.2298×10^{-4}	0.0304
	2	k=0.0096	n=1.3219	0.9998	7.9270×10^{-5}	0.0089
	3	k=0.0372	n=1.0000	0.9929	0.0033	0.0570
	4	a=1.0084	k=0.0032	0.9973	0.0012	0.0035
	5	a=1.0415	k=0.0291	0.9983	0.0012	0.0348
		c=-0.0363				
	6	a=1.0000	k=0.0077	0.9999	3.3932×10^{-5}	0.0058
		n=1.3872	$b=1.2225 \times 10^{-4}$			

3.3.2 Effective Moisture Diffusivity

Figures 3.5 and 3.6 show the values of ln(*MR*) versus drying time for all drying temperatures under hot air drying and microwave-assisted hot air drying, respectively. The value of effective moisture diffusivity, D_{eff} is calculated from the slope of the straight lines and the results of both drying schemes at 40, 50 and 60 °C are presented in Table 3.4. As shown in Figure 3.4, two linear slopes was observed under hot air drying at 50 °C and 60 °C. This corresponds to faster moisture diffusion in the falling-rate period as the temperature increases. The computed D_{eff} of hot air drying is in the range of 2.92 x 10^{-8} and 7.91 x 10^{-8} m^2/s and these results corresponding are comparable to the values obtained in other studies at similar drying temperatures (Sacilik et al., 2006; Unal & Sacilik, 2011).

In addition, these results are in good agreement with the previous work on broccoli florets (Mulet et al., 1999) and broccoli pieces (Doymaz, 2014a). The reported values of broccoli florets dried in hot air dryer at temperature of 35 °C to 70 °C with air velocity of 4 m/s ranged between 3.00 x 10^{-8} to 6.23 x 10^{-8} m^2/s. Whereas, the D_{eff} of broccoli pieces pretreated under different blanching temperature dried under cabinet dryer at 60 °C with air velocity of 2 m/s were in the range of 1.99 x 10^{-8} and 3.58 x 10^{-8} m^2/s. Meanwhile, the D_{eff} values of broccoli stalk slices obtained under microwave-assisted hot air drying are about twice higher than those of hot air drying, which is in the range of 6.64 x 10^{-8} and 13.31 x 10^{-8} m^2/s. For both drying systems, the D_{eff} are within the general order of magnitude of 10^{-8} - 10^{-12} m^2/s for drying of biological materials (Ahmed et al., 2011).

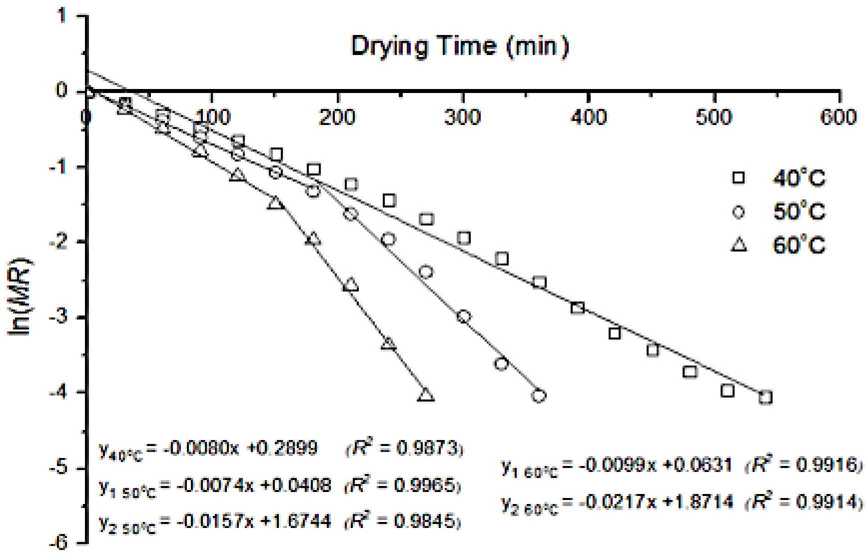


Figure 3.5: Plot of ln(*MR*) versus drying time of broccoli stalk slices under hot air drying at different air drying temperatures.

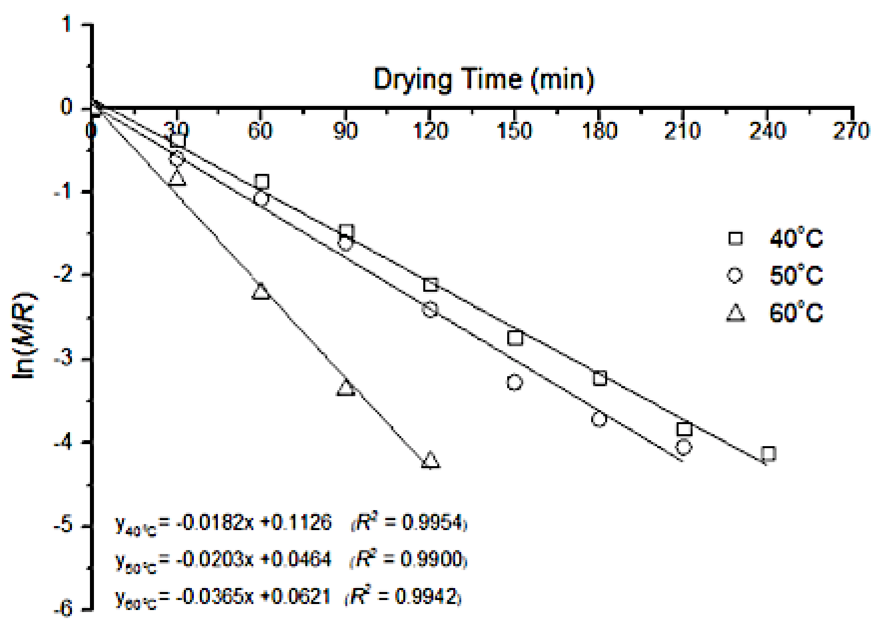


Figure 3.6: Plot of ln(*MR*) versus drying time of broccoli stalk slices under microwave-assisted hot air drying at different air drying temperatures.

Table 3.4: Effective moisture diffusivity of hot air drying (HA) and microwave assisted hot air drying (MWHA) at different drying temperatures.

Drying System	Drying temperature (°C)	Effective moisture diffusivity, D_{eff} (m^2/s)	
HA	40	2.92×10^{-8}	
	50	2.68×10^{-8}	5.72×10^{-8}
	60	3.62×10^{-8}	7.91×10^{-8}
MWHA	40	6.64×10^{-8}	
	50	7.41×10^{-8}	
	60	13.31×10^{-8}	

3.3.3 Product Quality: Color and Shape Changes

Figure 3.7 compared the total color change calculated from Equation 3.14 for hot air drying and microwave-assisted hot air drying at different drying temperatures. It is apparent from this figure that broccoli stalk dried under hot air drying at temperature of 50 °C had greatest changes ($p < 0.05$) in color compared to 40 °C and 60 °C. Samples dried at 40 °C required longer residence time, yet the lower temperature helps to minimize the color changes. While when samples are dried at higher temperature of 60 °C, the residence time is lower compared to 50 °C, which also leads to higher retention of color. Meanwhile, under microwave-assisted hot air drying, the color was more stable at different drying temperatures. Hence, the best color retention was achieved at 40 °C under microwave-assisted hot air drying.

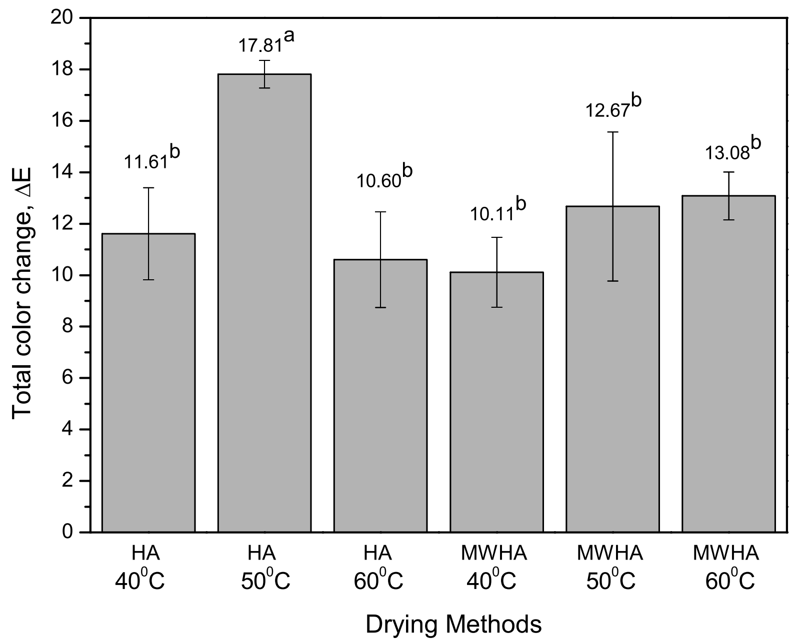


Figure 3.7: Total color change, ΔE of broccoli slices dried under hot air drying (HA) and microwave-assisted hot air drying (MWHA). (Column–wise values followed by the same letter are not significantly different).

Table 3.5 shows the results of shape analysis of dried broccoli stalk slices. The circularity obtained from Equation 3.15 shows that the geometry of the dried broccoli stalk slices were less of a circle under microwave-assisted hot air drying compared to hot air drying which is attributable to the curling effect of the final product. These results are in line with studies reported by Singh et al. (2013) which also produced curled potato slices as observed after drying under microwave-assisted hot air drying. To address this uneven shrinkage of the dried broccoli stalk products, pre-treatment prior to the drying process can be proposed for further research to minimize the tissue collapse during the drying process and strengthen the structure of the dried products. For example, osmotic dehydration as a pre-treatment step has been reported to improve the geometry of dried mushrooms (Torringa et al., 2001); apricot cubes (Riva et al., 2005); and apples (Kowalski & Mierzwa, 2013). Another pre-treatment that have a good potential to reduce the shrinkage and enhance the structural integrity of dried products are by applying high electric field (Singh et al., 2013) or through appropriate edible coatings on the surface of raw materials as reported by Askari et al. (2006) for apple slices and Garcia et al. (2014) for papaya.

Table 3.5: Shape analysis of broccoli slices dried under hot air drying (HA) and microwave-assisted hot air drying (MWHA).

System	Temperature (°C)	Area (cm^2)	Perimeter (cm)	Circularity
HA	40	1.92 ± 0.15^{a}	6.22 ± 0.34^{ab}	0.63 ± 0.05^{a}
MWHA		1.78 ± 0.04^{ab}	6.29 ± 0.36^{ab}	0.57 ± 0.06^{ab}
HA	50	1.82 ± 0.11^{a}	5.98 ± 0.43^{b}	0.64 ± 0.06^{a}
MWHA		1.75 ± 0.10^{ab}	6.80 ± 0.35^{a}	0.48 ± 0.06^{c}
HA	60	1.59 ± 0.02^{b}	5.92 ± 0.19^{b}	0.58 ± 0.05^{ab}
MWHA		1.74 ± 0.09^{ab}	6.49 ± 0.14^{ab}	0.52 ± 0.01^{bc}

Column–wise values followed by the same letter are not significantly different.

3.4 Conclusion

In this study, the drying behavior of broccoli stalk slices was investigated under hot air drying and microwave-assisted hot air drying. The results from this study showed that the drying time decreased considerably with increased air drying temperature for both drying systems. Combined microwave drying with hot air drying can reduce the drying time by up to 55% compared to hot air drying alone. The semi-empirical Midilli et al. model used to describe the drying kinetics of broccoli stalk slices gave an excellent fit of experimental data for all drying conditions. The effective moisture diffusivity, D_{eff}

increased with increase in the product temperature. The D_{eff} values of broccoli stalk slices dried under microwave-assisted hot air drying are higher than those dried under hot air drying alone, and this was harmonious with the theory. In addition, the changes in color were less observed at lower temperatures under microwave-assisted hot air drying. However, the shape of the dried broccoli was less circular compared to hot air drying. Taken together, these results shows that the hybrid drying technique can be used for faster water removal during drying of highly perishable biological material and further studies on pre-treatment could be conducted to enhance the geometry of the final product.

CONNECTING STATEMENT FOR CHAPTER 4

In **Chapter 3**, it was found that microwave-assisted hot air shorten the drying times of broccoli stalk slices than that of hot air alone. In order to improve the quality of dried product under microwave-assisted hot air drying, an attempt to apply osmotic dehydration as pre-treatment was evaluated. In the following chapter, investigation on the effects of operating parameters on osmotic dehydration to achieve higher water removal with minimal solute gain as a pre-drying step is described. The information provided in this study is useful to achieve better energy conservation for further drying in the development of dried broccoli stalk slices.

CHAPTER 4

EFFECTS OF OPERATING PARAMETERS ON OSMOTIC DEHYDRATION OF BROCCOLI STALK SLICES

Abstract

In this study, effects of sucrose concentration (40-60% w/v), immersion time (1-5 hours) and temperature (25-65 °C) on water loss and solute gain during osmotic dehydration of broccoli stalk slices were quantitatively investigated using response surface methodology. It was found that concentration of sucrose solution, immersion time and temperature affected the water loss during osmotic dehydration process. Significant factors affecting the solute gain are the temperature and immersion time. The operating conditions to obtain water removal of 61.5% with solute gain of 6.7% were found to be at a sucrose concentration of 56% w/v, temperature of 42 °C and immersion time of four hours.

Keywords: Broccoli stalk, Osmotic dehydration, Response surface methodology.

4.1 Introduction

Drying is an important process to increase the shelf life of food products by removing the moisture to a point where the product is shelf stable. During drying, exposure of biological products to high temperatures for long periods of time often results in quality degradation. Therefore, there are a various improved drying technologies spurred in food application nowadays to produce better quality product and to improve energy efficiency during the drying process.

In the last few years, there has been a plethora of studies on osmotic dehydration of various agricultural products. This phenomenon indicates that osmotic dehydration has been considered as an important process in food production (An et al., 2013b; Changrue & Orsat, 2009; Corrêa et al., 2011; Kowalski & Mierzwa, 2013; Therdthai & Visalrakkij, 2012; Udomkun et al., 2015; Verma et al., 2014; Wang et al., 2010a; Zhao et al., 2013). Osmotic dehydration utilizes osmotic principles when the food products are immersed in a hypertonic solution. During the immersion process, osmotic pressure acts as a driving force across the cell wall of the food product. This pressure results in cell membrane disintegration that triggers the resulting mass transfer processes (Rastogi et al., 2002). The two major mass transfer mechanism occurring are the water outflow from the product and the solute inflow from the solution to product concurrently. There are also natural solutes (such as minerals, organic acids, pigments and flavor) leaching from the product to the solution during the immersion process (Taiwo et al., 2003; Tortoe, 2010). All these mass transfer exchange might affect the final quality of food product. Thus, it is important to identify the optimum operating conditions to increase the mass transfer and produce a desirable quality product.

Osmotic dehydration is a process where the water is removed without a phase change. Thus, it can facilitate less energy requirement which is a major concern in the drying process (Raghavan & Orsat, 2008). Although this process will not result in sufficiently low moisture content to be considered as a shelf stable product, still it can reduce the mass of fresh sample by up to 50%. Hence, it has often been applied as a pre-treatment step prior to finish drying. Previous study has shown that osmotic dehydration was found advantageous in improving the final quality of food product (An et al., 2013b; Changrue, 2006; Therdthai et al., 2011). In general, food products that undergoes the osmotic step before drying results in modified functional properties of food; which are favorable for drying and offers a various taste of end products that can provide more options to consumers.

Broccoli is considered as the best known cruciferous vegetable that is rich in health-promoting components (Latté et al., 2011; Moreno et al., 2006). Each part of the broccoli plant is known to have significant amount of bioactive compounds (Campas-Baypoli et al., 2009; Singh et al., 2011; Vallejo et al., 2002). In broccoli market, the florets are the main raw commodities. Meanwhile the stalk and leaves are left as crop remains. Therefore, there is a crucial need in adding value to these by-products and thus bring profit to the broccoli industry.

In this study, we are interested to add value to the broccoli stalk by applying osmotic dehydration as a pre-treatment step before drying process. Previous study done by Ohnishi and Miyawaki (2005) reported that broccoli tissue pretreated osmotically in sucrose solutions has higher retention of rheological properties compared to untreated samples. However, little information is available on the statistical modelling of osmotic dehydration of broccoli stalk slices.

Furthermore, the broccoli stalk consists of a woody outer layer that can limit the mass transfer during osmotic dehydration process due to its rigid cell wall. For this particular circumstance, skin peeling becomes an important step to enhance the mass transfer rate (Lewicki & Lenart, 1995). In addition, the rate of mass transfer is also dependent upon various operating variables such as temperature, type of osmotic agent, osmotic solution concentration, immersion time, ratio of solution to product, size and geometry of the food sample.

Response surface methodology (RSM) is an effective statistical tool to evaluate the effects of operating factors even in the presence of complex interactions and to seek the optimum conditions for multivariable response according to the target specification. RSM is also a useful technique in building empirical model for performance prediction that relate the response to the process parameter (Song & Hwang, 2003). Other advantage of employing RSM is associated with the design of experimentation which helps in reducing the number of experimental runs needed to evaluate the various operating factors and their interactions; leading to substantial reduction in the cost and experiment time (Giovanni, 1983; Mohsen et al., 2013).

Therefore, the objectives of this study are to investigate the effects of sucrose concentration, temperature and immersion time on the mass transfer exchange during osmotic dehydration process of broccoli stalk and to find the optimum operating conditions that maximizes the water loss while minimizing the solute gain using response surface methodology.

4.2 Materials and Methods

4.2.1 Materials

The broccoli stalks (*Brassica oleracea* L. var. *italica*) were washed, peeled and sliced to a uniform shape with a thickness of 6 mm and a diameter of 23 mm. Moisture content of samples were determined by drying in a hot air oven at 105 °C (Thermo Scientific Model 6528, Rockford, IL, USA) until a constant mass was reached (AOAC 935.29) (AOAC, 2000). Sucrose (Redpath Sugars, Montreal, QC, Canada), used as an osmotic agent was purchased locally. The osmotic solutions were prepared by dissolving the sucrose with the proper amount of distilled water.

4.2.2 Osmotic Dehydration

Containers filled with sucrose solution are placed in a water bath controlled by heating/cooling unit (Isotemp 1013S, Fisher Scientific Inc., Pittsburgh, PA, USA). All containers were covered with a lid during the experiment in order to prevent evaporation from the osmotic solution. Once the osmotic solution reached the required temperature, the weighed broccoli stalk slices were immersed in the container and kept for the required time period. Samples were then removed from the osmotic solution and rinsed immediately in flowing water, drained on a tissue paper to remove surface moisture. The mass and moisture content of sample before and after osmotic process were determined. For each experiment, fresh sucrose solution was used. A ratio of sample to solution was kept constant at 1:20.

4.2.3 Determination of Water Loss and Solute Gain

Gravimetric approach has been used extensively to analyze osmotically dehydrated products (Tortoe, 2010). The terminology used to quantify the mass exchange of sample and solution between broccoli stalk slices and sucrose solution during osmotic dehydration are the water loss (WL) and solute gain (SG). The WL and SG were calculated using the following equations (Hawkes & Flink, 1978):

$$WL\ (\%) = \frac{w_{wo} - (w_t - w_{st})}{w_{wo} + w_{so}}\ \text{x}\ 100 \tag{4.1}$$

$$SG\ (\%) = \frac{w_{st} - w_{so}}{w_{wo} + w_{so}}\ \text{x}\ 100 \tag{4.2}$$

Where W_{wo} is the mass of water in the sample before dehydration (g), W_t is the mass of sample after dehydration (g), W_{so} is the mass of the solids in the sample before dehydration (g) and W_{st} is the mass of solids in the sample after dehydration (g).

4.2.4 Experimental Design and Statistical Analysis

The response surface methodology was used to estimate the main effects of osmotic dehydration process. The experiments were designed according to central composite rotatable design using the statistical software JMP version 11 (SAS Institute, Cary, NC, USA). The rotatable design provides a constant variance under any rotation of the coordinate axes (Jones, 1996). The design required 20 runs derived from 2^3 full factorial design combined with 6 center points and 6 axial points as illustrated in Figure 4.1. An axial point distance from the center point is 1.68. The 6 center points runs was chosen in this study with the following purposed (Reddy, 2011): (1) Provide a measure of process stability (i.e. reduces model prediction error); (2) To capture any inherent variability; and (3) To provide a check for curvature.

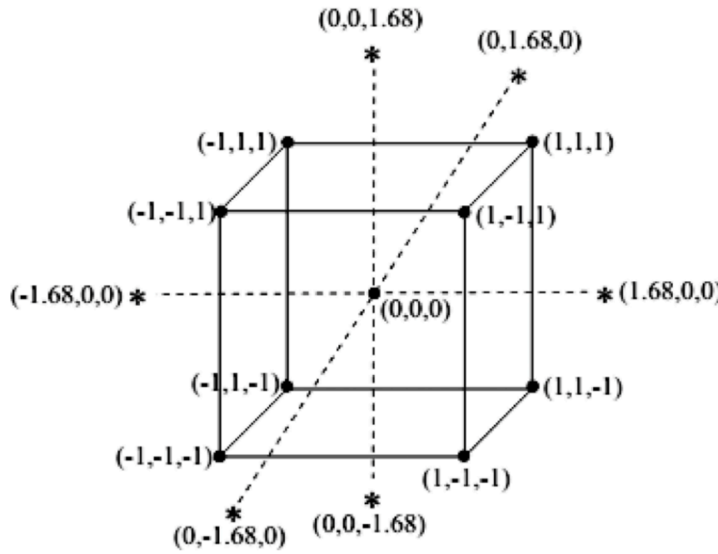


Figure 4.1: Central composite rotatable design for the three variables (Kazemi-Beydokhti et al., 2015).

The three continuous independent variables were sucrose concentration (X_1), temperature (X_2) and immersion time (X_3) which were examined at five different levels as shown in Table 4.1. The column called Pattern identifies the coding of the factors; "+" for high factor, "–" for low factor, "a" and "A" for low and high axial values, and "0" for midrange. The levels of the independent variables were selected based on the preliminary study. The WL (Y_1) and SG (Y_2) obtained from experimental data are the dependent variables, also referred as responses were subjected to an analysis of variance (ANOVA) in order to determine the significant effects of independent variables on each response. The following second-order polynomial model was used for the statistical analysis:

$$Y_i = b_0 + \sum_{i=1}^{k} b_i X_i + \sum_{i=1}^{k} b_{ii} X_i^2 + \sum_{i=1}^{k-1} \sum_{j=i+1}^{k} b_{ij} X_i X_j \quad (4.3)$$

where Y_i is the response (i.e. Y_1 is WL and Y_2 is SG); b_o, b_i, b_{ii}, b_{ij} are the regression coefficients for intercept, linear, quadratic and interaction terms; X_1, X_2 and X_3 are the coded independent variables.

The identification of the operating conditions for osmotic dehydration of broccoli stalk slices as a pre-drying stage, which aimed to achieve higher water removal with minimal solute gain were evaluated using desirability function performed by the same software as mentioned earlier.

Table 4.1: Process variables and the levels for the osmotic dehydration of broccoli stalk slices.

Coded (X_i)	Independent Variables	Pattern and levels				
		a	-	0	+	A
X_1	Sucrose Concentration (w/v %)	40	44.05	50	55.95	60
X_2	Temperature (°C)	25	33.11	45	56.89	65
X_3	Immersion time (hour)	1	1.81	3	4.19	5

Desirability function is a simple and useful technique in optimizing multiple response to provide the desirable product characteristics (Costa et al., 2011; Eren & Kaymak-Ertekin, 2007; Vieira et al., 2012). Different desirability functions can be used depending on particular response whether to be maximized or minimized (Derringer & Suich, 1980). For each response, the desirability functions assign numbers between 0 and 1, and it corresponds to the desirability interpretation such as: very good (1.0 - 0.8); good (0.8 - 0.63); fair (0.63 - 0.37); poor (0.37 - 0.2); and very poor (0.2 - 0) (Harrington, 1965). In practice, fitted response value $\hat{Y}_i$ at operating conditions x are used in place of Y_i. Thus, for WL to be maximized, the desirability function, d_1 is written as:

$$d_1(\hat{Y}_1) = \begin{cases} 0 & if \;\; \hat{Y}_1(x) < L_1 \\ \dfrac{Y_1\,(x) - L_1}{T_1 - L_1} & if \; L_1 \leq \hat{Y}_1(x) \leq T_1 \\ 1 & if \; \hat{Y}_1(x) > T_1 \end{cases} \tag{4.4}$$

where L_1 is a lower value for $\hat{Y}_1$, and T_1 is the target value. For SG to be minimized, the desirability function, d_2 with the target value T_2 and the upper value U_2 has the form:

$$d_2(\hat{Y}_2) = \begin{cases} 1 & if \;\; \hat{Y}_2(x) < T_2 \\ \dfrac{Y_2\,(x) - U_2}{T_2 - U_2} & if \; T_2 \leq \hat{Y}_2(x) \leq U_2 \\ 0 & if \;\; \hat{Y}_2(x) > U_2 \end{cases} \tag{4.5}$$

The individual desirabilities are then combined using the geometric mean, which gives the overall desirability function, D :

$$D = (d_1 d_2)^{1/2} \tag{4.6}$$

4.3 Results and Discussion

4.3.1 Effect of Process Variables on Water Loss and Solute Gain

Experimental data obtained for WL and SG of broccoli stalk slices subjected to different run of osmotic dehydration conditions are presented in Table 4.2. The response for WL and SG are in the range of 37.0% to 65.0% and 3.7% to 15.6%, respectively. The results of multiple linear regressions conducted using second order response surface model for describing the effect of independent variables on the WL and SG are presented in Table 4.3 (a) and (b), respectively.

According to the results given in Table 4.3, it can be seen that WL was affected linearly and quadratically by all the independent variables. The sucrose concentration was the most important effect on WL , followed by immersion time and temperature. However, the sucrose concentration did not significantly influence SG. Only the effect of temperature and immersion time positively affected SG. To visualize the effects of indepedant variables on WL and SG, response surface curves were generated (Figure 4.1 to 4.3). These surface plot reflect the influence of two independent variables on WL and SG while the third variable was kept constant at their center point.

Table 4.2: Experimental conditions with values of response variables.

		Independent variables			Dependent variables	
Run #	Pattern	Concentration (w/v %)	Temperature (°C)	Time (hour)	WL (%)	SG (%)
1	a00	40.00	45.00	3.00	40.76	4.89
2	−−−	44.05	33.11	1.81	36.97	4.55
3	−−+	44.05	33.11	4.19	39.57	6.43
4	−+−	44.05	56.89	1.81	42.14	8.99
5	−++	44.05	56.89	4.19	44.90	14.17
6	0a0	50.00	25.00	3.00	42.49	5.69
7	00a	50.00	45.00	1.00	40.30	3.68
8	000	50.00	45.00	3.00	57.22	5.96
9	000	50.00	45.00	3.00	56.24	5.93
10	000	50.00	45.00	3.00	57.27	6.34
11	000	50.00	45.00	3.00	57.30	5.93
12	000	50.00	45.00	3.00	57.37	6.12
13	000	50.00	45.00	3.00	56.73	5.82
14	00A	50.00	45.00	5.00	61.18	8.83
15	0A0	50.00	65.00	3.00	50.67	15.64
16	+−−	55.95	33.11	1.81	42.40	4.12
17	+−+	55.95	33.11	4.19	54.59	6.55
18	++−	55.95	56.89	1.81	46.6	7.27
19	+++	55.95	56.89	4.19	57.76	13.37
20	A00	60.00	45.00	3.00	65.02	4.94

Table 4.3: Regression coefficients and ANOVA of the second-order polynomial model for the overall effects of the three process variables (sucrose concentration (X_1), temperature (X_2) and immersion time (X_3)) on (a) water loss (WL) and (b) solute gain (SG).

(a) For WL (R^2 = 0.93, Adjusted R^2 = 0.87, Root Mean Square Error (RMSE) = 3.11)

Source	Sum of Square	*df*	Mean Square	F Ratio	Prob > F	Estimate
Intercept						57.15
Concentration	452.03	1	452.03	46.83	<.0001	9.68
Temperature	73.24	1	73.24	7.59	0.0203	3.89
Time	298.29	1	298.29	30.90	0.0002	7.86
Concentration*Temperature	1.22	1	1.22	0.13	0.7291	-1.11
Concentration*Time	40.46	1	40.46	4.19	0.0678	6.36
Temperature*Time	0.09	1	0.09	0.01	0.9231	-0.31
Concentration*Concentration	77.01	1	77.01	7.98	0.0180	-6.54
Temperature*Temperature	297.37	1	297.37	30.81	0.0002	-12.85
Time*Time	135.98	1	135.98	14.09	0.0038	-8.69
Model	1301.37	9	144.60	14.98	0.0001	
Error	96.52	10				
Corrected Total	1397.89	19				
Lack Of Fit	95.52	5	19.10	95.92	<.0001	
Pure Error	1.00	5	0.20			
Total Error	96.52	10				

(*df*= Degree of Freedom)

(b) For SG (R^2 = 0.98, Adjusted R^2 = 0.97, Root Mean Square Error (RMSE) = 0.59)

Source	Sum of Squares	*df*	Mean Square	F Ratio	Prob > F	Estimate
Intercept						5.99
Concentration	0.55	1	0.55	1.57	0.2388	-0.34
Temperature	110.71	1	110.71	314.75	<.0001	4.79
Time	43.06	1	43.06	122.43	<.0001	2.99
Concentration*Temperature	0.61	1	0.61	1.74	0.2171	-0.78
Concentration*Time	0.27	1	0.27	0.77	0.4014	0.52
Temperature*Time	6.07	1	6.07	17.26	0.002	2.46
Concentration*Concentration	0.67	1	0.67	1.90	0.1986	-0.61
Temperature*Temperature	47.62	1	47.62	135.39	<.0001	5.14
Time*Time	0.96	1	0.96	2.74	0.1288	0.73
Model	211.37	9	23.49	66.77	<.0001	
Error	3.52	10	0.35			
Corrected Total	214.89	19				
Lack Of Fit	3.35	5	0.67	19.43	0.0027	
Pure Error	0.17	5	0.03			
Total Error	3.52	10				

(*df*= Degree of Freedom)

Figure 4.2 shows the effect of sucrose concentration and immersion time on WL. It shows that WL is increasing gradually with solution concentration over the entire process due to high osmotic driving force between the sample and solution. In addition, the WL increases rapidly at the beginning stage of the process, after which the rate of water transfer slowes down. These findings match those observed in earlier studies on potato (Eren & Kaymak-Ertekin, 2007). The higher water removal from the sample during the early stage of the process makes the concentration gradient reduction around the sample and subsequently decreases the driving force thus lowering the mass transfer rate.

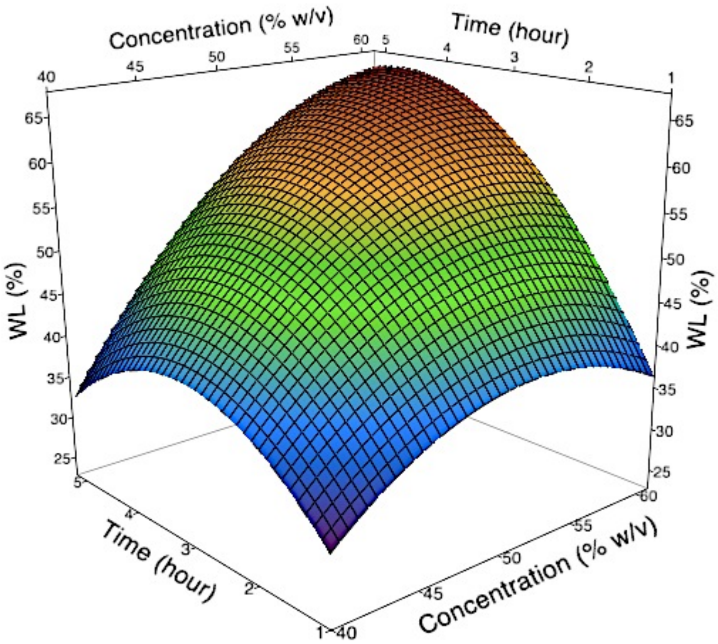


Figure 4.2: 3D surface plot as a function of sucrose concentration and immersion time during osmotic dehydration of broccoli stalk slices on water loss (WL).

On the other hand, WL increased at the lower temperatures, while the rate gradually decreased at higher temperature as shows in Figure 4.3. Higher temperatures seem to increase the solubility of solids in liquids. Therefore, the osmotic solution becomes more dilute (less concentrated) and it reduces the driving force during the osmotic dehydration process. Although the rate of water removal has decreased at the higher temperature, the SG kept increasing (Figure 4.4). This is alluded to the membrane swelling effect which might increase the cell membrane permeability to sucrose molecules (Lazarides et al., 1997). The increases in SG as temperature increased are in line with those of previous studies reported by Azoubel and Da Silva (2008) for mangoes; Lee and Lim (2011) for pumpkin; and (Badwaik et al., 2013) for bamboo shoots. It is to be noted in Figure 4.4 that higher values of temperature in combination with longer immersion time produced sharper increases of SG. Through these findings, it demonstrates that higher temperature applied on osmotic dehydration of broccoli stalk contributes to the decrease of WL performance with increase in SG.

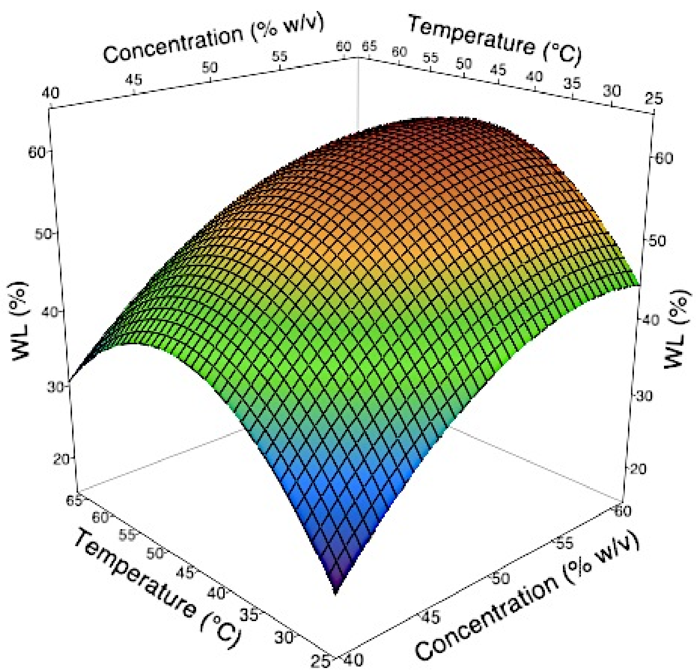


Figure 4.3: 3D surface plot as a function of temperature and sucrose concentration during osmotic dehydration of broccoli stalk slices on water loss (WL).

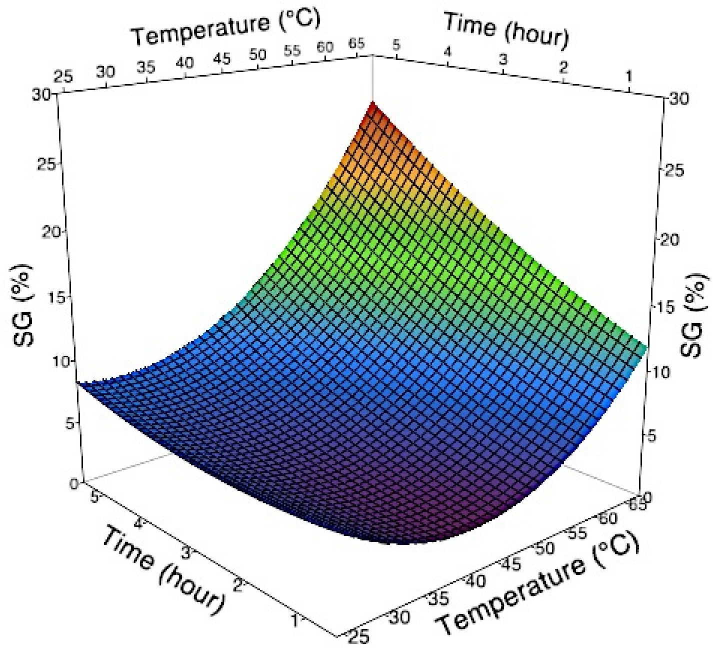


Figure 4.4: 3D surface plot as a function of immersion time and temperature during osmotic dehydration of broccoli stalk slices on solute gain (SG).

4.3.2 Predictive Model for Water Loss and Solute Gain

The predictive model was obtained for each response by stepwise regression (Draper et al., 1966). The correlation value of these models for WL (R^2 = 0.90) and SG (R^2 = 0.96) indicate a good fit of experimental data. The plot of predicted versus of experimental data for WL is shown in Figure 4.4 and SG in Figure 4.5. The mathematical expressions to describe the relationship to the response with variables are shown in Equations 4.7 and 4.8 for WL and SG, respectively. Both models are valid within the applied range of the experimental processing variables used in this study.

$$WL\ (\%) = 57.15 + 9.68\,X_1 + 3.89X_2 + 7.86X_3 - 6.54\,{X_1}^2 - 12.84{X_2}^2 - 8.69{X_3}^2 \quad (4.7)$$

$$SG\ (\%) = 5.99 + 4.78X_2 + 2.99X_3 + 4.14{X_2}^2 + 2.46X_2X_3 \quad (4.8)$$

where;

$$X_1 = \frac{(\,Concentration - 50)}{10} \quad ; \; 40 \text{ w/v \%} \leq Concentration \leq 60 \text{ w/v \%}$$

$$X_2 = \frac{(\,Temperature - 45)}{20} \quad ; \; 25\ °C \leq Temperature \leq 65\ °C$$

$$X_3 = \frac{(\,Time - 3)}{2} \quad ; \; 1 \text{ h} \leq Time \leq 5 \text{ h}$$

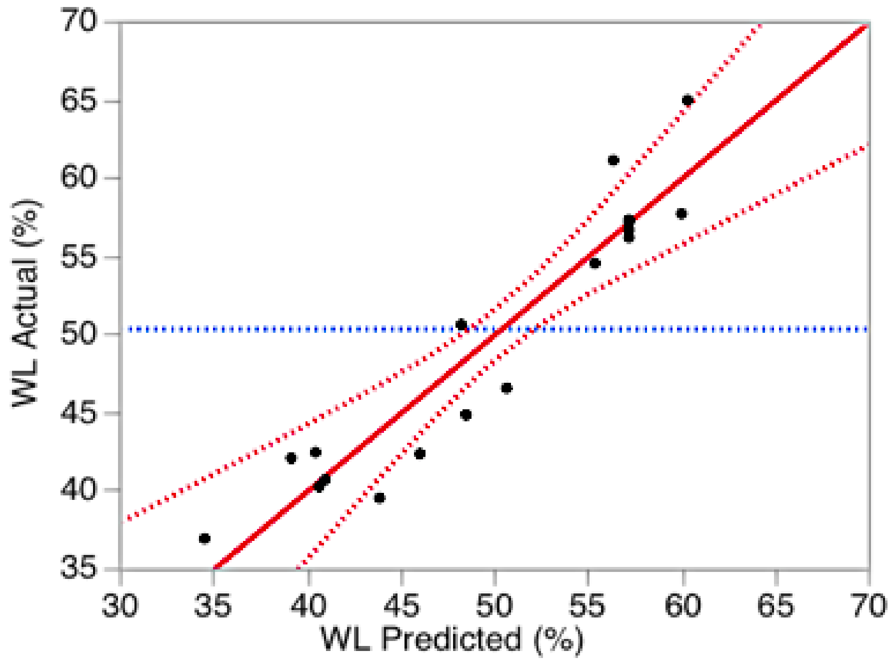


Figure 4.5: Predicted versus experimental values for water loss (WL).

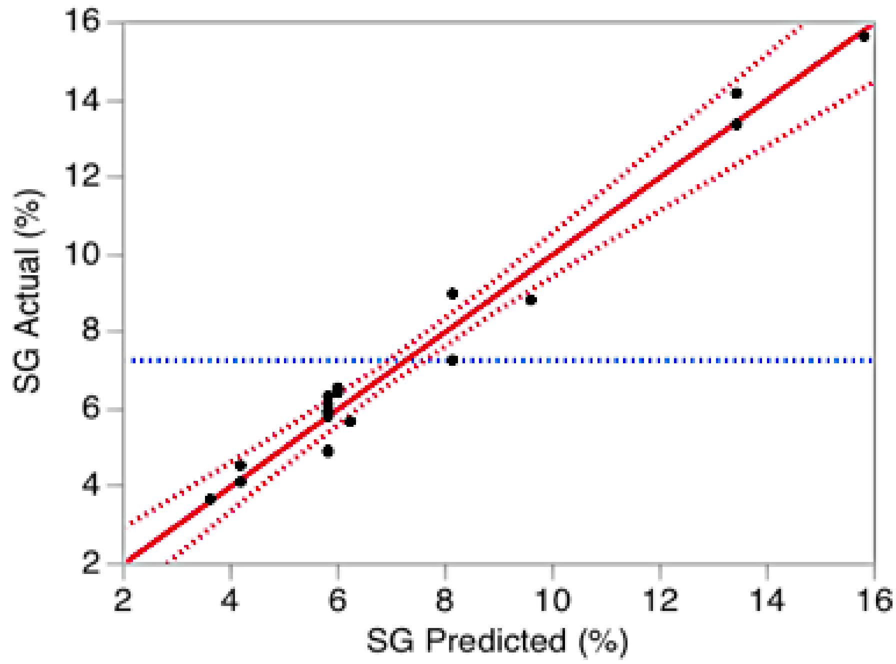


Figure 4.6: Predicted versus experimental values for solute gain (SG).

4.3.3 Operating Conditions for Osmotic Dehydration as Pre-drying Step

The studied osmotic dehydration concept has shown partial water removal from broccoli stalk that can results in less energy use in finish drying. However, the solute uptake would cause a change in sensory perception and acceptability. Moreover, excessive solute uptake during the osmotic process can block the surface layer of the product and this additional resistance can lower the water diffusion rate during the finish drying process (Menting et al., 1970).

For this reason, it is important to determine optimal operating conditions of three independent variables: (1) sucrose concentration at a range of 40-60 w/v %; (2) temperature at a range of 25-65 °C; and (3) immersion time at a range of 1 to 5 hours to achieve higher WL at minimal SG which can be used at the pre-drying stage. By applying desirability function generated by the JMP statistical software, the results indicate that to achieve 61.5 (g / 100 g fresh sample) of WL with corresponding SG of

6.7 (g /100 g fresh sample) the conditions required are: a concentration of 56 w/v % operating at 42 °C for four hours of immersion time. The individual desirability at these conditions were found to be 0.74 for WL and 0.65 for SG; which provides an overall desirability of 0.70. These conditions show that the water content in broccoli stalk can be reduced by more than 50% through the osmotic dehydration. Meanwhile, less than 7% SG are achieved in four hours of immersion time.

4.4. Conclusion

In this study, the effects of sucrose concentration, temperature and immersion time on mass transfer exchange during osmotic dehydration of broccoli stalk slices was investigated using response surface methodology. Analysis of variance shows that sucrose concentration, temperature and immersion time were significantly affected water loss (WL). Whereas, temperature and immersion time significantly affected solute gain (SG). The predictive model for both responses was well fitted to the experimental data with high value of R^2 (≥ 0.9). From the analysis, water removal of 61.5% with only 6.7% gain of solute can be accomplished through osmotic dehydration at its optimum conditions; sucrose concentration of 56 w/v % at 42 °C for four hours immersion time. Also, energy conservation for further drying is achievable through the establishment of these pre-drying conditions.

CONNECTING STATEMENT FOR CHAPTER 5

In **Chapter 4**, the pre-drying stage to obtain higher water removal at minimal solute gain has been determined. In **Chapter 5**, the drying characteristics of the osmotically dehydrated product dried under microwave-assisted hot air drying were evaluated. In addition, the performance in terms of drying time and product quality were compared with untreated products dried under microwave-assisted hot air and hot air drying.

CHAPTER 5

MICROWAVE-ASSISTED HOT AIR DRYING CHARACTERISTICS OF OSMOTICALLY DEHYDRATED BROCCOLI STALK SLICES

Abstract

Osmotic dehydration prior to microwave-assisted hot drying was investigated as potential means for drying the broccoli stalk slices. The drying time and the changes in color and shape were compared with untreated samples dried under microwave-assisted hot air drying and hot air drying at different drying temperatures. The comparison showed that osmotic dehydration pre-treatment remarkably decreases the drying time, improves the shape appearance and better color retention at lower drying temperatures. Among thin layer models, Page model was the best to describe the drying of osmotically dehydrated broccoli.

Keywords: Microwave-assisted drying, Osmotic dehydration, Broccoli, Product quality.

5.1 Introduction

Broccoli (*Brassica oleracea* L. var. *italica)* is a nutritious vegetable that have been consumed worldwide. Based on the database at Statistics Canada in 2014, broccoli was harvested in Canada on 10,427 acres in 2013 with total production of 37,947 metric tonnes. This value has increased by 12 percent compared to 2011. Consumption of broccoli has increased due to their numerous health benefits. It has been conclusively shown that broccoli intake can help reduce the risk for the development of certain forms of cancer (Latté et al., 2011). Habitually, edible floret of broccoli is consumed, leaving the stalk as crop remains. Study done by Singh et al. (2011) found that broccoli stalk still contains nutritional values such as ascorbic acid, β-caretenoids, phenolics and total antioxidant activity. Therefore, these stalks could be used in product development which required drying as one of the key unit operation.

Drying is a process of removal of water or to reduce the water activity in order to produce a shelf stable product. During drying, exposure of biological products to high temperatures for long periods of time often results in quality degradation. Therefore, there are a various improved drying technologies spurred in food application nowadays and hybrid microwave drying is one of the promising options. Microwave energy has been applied successfully to assist in many drying processes (Corrêa et al., 2011; Dev et al., 2011; Singh et al., 2013; Workneh et al., 2011). Microwave generate heat by exciting dipolar molecules and ionic conduction caused by the alternating electromagnetic field resulting rapid heating from within the sample itself, which is known as volumetric heating (Orsat et al., 2007). The rapid energy dissipation throughout the material is an advantage to overcome the limitation of other slow drying processes and improves the final quality of the dried products (Zhang et al., 2006).

Meanwhile, hot air drying is commonly used method in drying process due to its simple operation. However, drying with only hot airflow takes a long time and has low energy efficiency and subsequently, may result in quality degradation of dried products. During hot air drying, the heat has to diffuse in from the surface of the material. Thus, rapid reduction of the surface moisture may act as water barrier that may lead to case hardening and shrinkage (Gunasekaran, 1999; Zhang et al., 2006). Therefore, applying microwave with hot air drying seems to be an applicable approach that can minimize the retreating wet front, maintaining the evaporating surface and perhaps permitting entrainment-evaporation (Raghavan & Orsat, 2008). This will lead to reduction in drying time, improved drying efficiency and minimization of product degradation.

Apparently, osmotic dehydration has gained much attention as a pre-treatment step in drying process. Osmotic dehydration is partial removal of water in liquid form from biological materials by immersion in osmotic solution. When considering transferring water from solid/liquid to a gas, high energy consumption is involved. Therefore, by applying osmotic dehydration, water is reduced by up to 50% of the initial mass without a phase change which is advantageous in the drying process. Besides lowering the energy consumption, osmotic pre-treatment prior to drying can shorten the drying time and improve overall product quality (Corrêa et al., 2011; Kowalski & Mierzwa, 2013).

The aim of this study was to investigate the characteristics of osmotically dehydrated broccoli stalk slices dried under microwave-assisted hot air (Osmo-MWHA) at different drying temperatures. The drying time, color and shape changes of Osmo-MWHA was then compared with the broccoli stalk slices dried under hot air drying (HA) and microwave-assisted hot air drying (MWHA) without osmotic dehydration pre-treatment. In addition, the mathematical modelling and effective moisture diffusivities of microwave-assisted hot air drying of osmotically dehydrated broccoli stalk were determined.

5.2 Materials and Methods

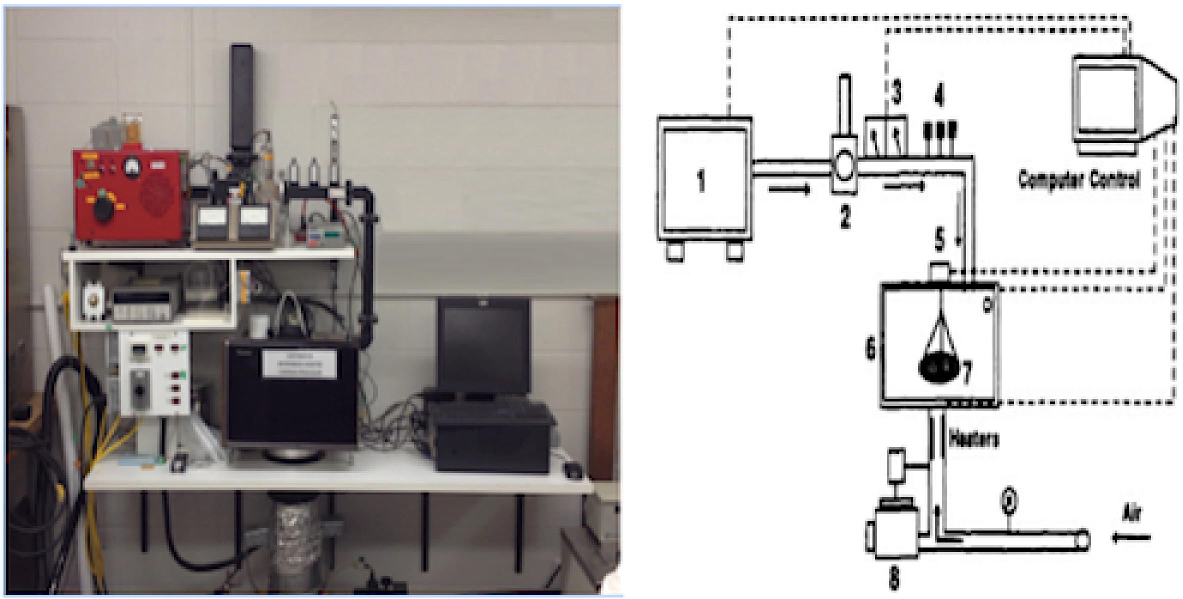
5.2.1 Materials

Broccoli used in this study was purchased from a local market. The stalks were separated from the florets. Then, the stalks were washed, peeled and sliced to a uniform shape with a thickness of 0.6 cm and a diameter of 2.3 cm. Moisture content of samples was determined by drying in a hot air oven at 105 °C (AOAC 935.29) (AOAC, 2000). These experiments were replicated thrice and it was found to have a moisture content of 94% w.b.

5.2.2 Experimental Procedure

Containers filled with 56 w/v % sucrose solution were placed in a water bath controlled by heating/cooling unit (Isotemp 1013S, Fisher Scientific Inc.). The entire containers were covered with a lid during the experiment in order to prevent evaporation from the osmotic solution. Once the osmotic solution reached 42 °C, the 50 g broccoli stalk slices were immersed into the container and kept within four hours. Samples were then removed from the osmotic solution and rinsed immediately in flowing water, drained on a tissue paper to remove surface moisture. The weight and moisture content of sample before and after osmotic process were determined. A ratio of sample to solution was kept constant at 1:20 to minimize the significant dilution and to maintain the potential of dehydration. This given condition was the optimized processing condition evaluated from the preliminary experiment conducted with the aim of maximizing the water loss while minimizing the solute gain.

After osmotic dehydration pre-treatment, the samples were then subjected to microwave assisted hot air drying. The dryer as shown in Figure 5.1 consist of 2,450 MHz microwave generator with 3 tuning screws for adjusting and minimizing the reflected power. The power of the dryer was set to 100 Watts and the superficial air velocity of about 1.4 m/s. Air supplied by the blower was heated to the required temperature (40°C, 50°C and 60°C) until it reached a moisture content of 20% w.b. The temperature of the sample was monitored by fibre optic temperature sensor and the mass of the samples was recorded by the computer via data acquisition system (Hewlett-Packard, USA). The drying characteristics of osmotic-microwave assisted hot air (Osmo-MWHA) was then compared with the untreated broccoli stalk slices dried under microwave-assisted hot air (MWHA) drying and hot air (HA) drying which was previously reported in Chapter 3.



(a) (b)

Figure 5.1 (a): Automated microwave-assisted laboratory dryer; (b): Schematic diagram of microwave-assisted laboratory dryer (1: microwave generator, 2: circulator, 3: power meters, 4: tuning screws, 5: strain gauge, 6: microwave cavity, 7: sample holder and 8: blower).

5.2.3 Analysis of Drying Data

The change in moisture during drying will be expressed by the moisture ratio:

$$\text{Moisture Ratio (MR)} = \frac{M_t - M_e}{M_o - M_e} \tag{5.1}$$

Where M_t is the moisture content at time t_i, M_o is the initial moisture content and M_e is the equilibrium moisture content.

However, MR is simplified to Equation 5.2 because M_e is too small to be quantified.

$$\mathrm{MR} = \frac{M_t}{M_o} \tag{5.2}$$

The experimental drying data obtained were fitted to four well known drying models given in Table 5.1.

Table 5.1: Thin layer drying models used for drying kinetics of broccoli slices.

Model Name	Model Equation	Equation no.
Page	$MR = exp(-kt^n)$	(5.3)
Newton	$MR = exp(-kt)$	(5.4)
Handerson & Pabis	$MR = a\ exp(-kt)$	(5.5)
Wang and Singh	$MR = 1 + at + bt^2$	(5.6)

Where, *k* is the drying rate constant; *n,a,b* is the regression coefficient and *t* is the time (min).

The statistical parameters such as the reduced chi-square (χ^2), root mean square error (RMSE) and coefficient of determination (R^2) are used in selecting the best equation to describe the drying curve equation. The highest R^2 and the lowest χ^2 and *RMSE* values required were used to evaluate the goodness of fit (Erbay & Icier, 2010; Singh et al., 2013). These comparison parameters calculated are as follows:

$$\chi^2 = \sum_{i-1}^{i=n} \frac{(MR_i^{pred} - MR_i^{exp})^2}{n-m} \tag{5.7}$$

$$RMSE = \sqrt{\frac{1}{n}\sum_{i=1}^{n}(MR_i^{pred} - MR_i^{exp})^2} \tag{5.8}$$

$$R^2 = 1 - \frac{\sum_{i-1}^{i=n}\left(MR_i^{exp} - MR_i^{pred}\right)^2}{\sum_{i=n}^{i=n}\left(MR_i^{exp} - \overline{MR}^{exp}\right)^2} \quad (5.9)$$

Where *n* is the number of observations, MR_i^{pred} is the *i*th predicted dimensionless MR, $MR_i^{exp}$is the *i*th experimental dimensionless MR, *m* is the number of variables and $\overline{MR}^{exp}$ is the mean of the experimental dimensionless MR.

5.2.4 Calculation of Effective Moisture Diffusivity

The experimental drying data for the determination of effective moisture diffusivity were estimated by using Fick's diffusion model. The general series of Fick's second law for slab sample is given as follows (Crank, 1975) :

$$MR = \frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{2n+1}\exp\left(-(2\mathrm{n}+1)^2\frac{\pi^2 D_{eff}t}{4L^2}\right) \quad (5.10)$$

Where D_{eff} is the effective moisture diffusivity (m^2 /s), and L is the half-thickness of the broccoli slices (m) and n is the positive integer. Equation 5.10 can be further simplified in logarithmic forms as in Equation 5.11 (Tütüncü & Labuza, 1996) and the effective moisture diffusivity can be estimated from the slope of linear plot.

$$\ln(MR) = ln\left(\frac{8}{\pi^2}\right) - \left(\frac{\pi^2}{4L^2}D_{eff}t\right) \quad (5.11)$$

5.2.5 Color Measurement

The parameters *L** (lightness), a^* (green–red coordinate) and *b** (blue–yellow coordinate) were obtained by using a tristimulus colorimeter (CR-310, Minolta Co. Ltd., Japan). The color of the dried samples were analysed in terms of *ΔL**, Δa^*, *Δb** and ΔE. The values for *ΔL**, Δa^* and *Δb** indicate how much a standard sample differs from another with respect to *L**, a^* and b^*, according to equations:

$$\Delta L^* = L^* - L^*_0 \tag{5.12}$$

$$\Delta a^* = a^* - a^*_0 \tag{5.13}$$

$$\Delta b^* = b^* - b^*_0 \tag{5.14}$$

Where, the subscript 0 represents the fresh sample.

The total color difference (ΔE) is a single value which takes into account the differences between the *L*, a* and b** values of the dried sample and standard are calculated according to the following equation:

$$\Delta \mathrm{E} = \sqrt{(L^* - L^*_0)^2 + (a^* - a^*_0)^2 + (b^* - b^*_0)^2} \tag{5.15}$$

5.2.6 Shape Determination

A digital camera (Nikon Coolpix s80) with high resolution of 14.1 megapixels was used to capture the shape of raw and dried broccoli stalk. The images were analyzed using the software *ImageJ* version 1.43 (National Institute of Health, USA). The radius and area of the samples were obtained by converting the pixels into cm units and the circularity were calculated based on the following equation:

$$\mathrm{Circularity} = \frac{4\pi \text{ x Area}}{\mathrm{Perimeter}^2} \tag{5.16}$$

5.2.7 Statistical Analysis

All experiments were conducted in triplicate and the means were analyzed using analysis of variance (ANOVA). Duncan's multiple range test was used to identify significant difference ($p < 0.05$) among samples for each attribute.

5.3 Results and Discussion

5.3.1 Drying Curves

The graphs of moisture content versus drying time of the broccoli samples with and without osmotic dehydration pre-treatment at various drying temperatures are shown in Figure 5.2. For all drying methods, decrease in drying time was observed when drying temperature was increased. The drying time for untreated broccoli slices dried under HA drying at 40 °C, 50°C and 60 °C took about 540 min, 360 min and 270 min, respectively to reach 20% (w.b.). Meanwhile, drying time was reduced by 42-55% when dried under MWHA drying.

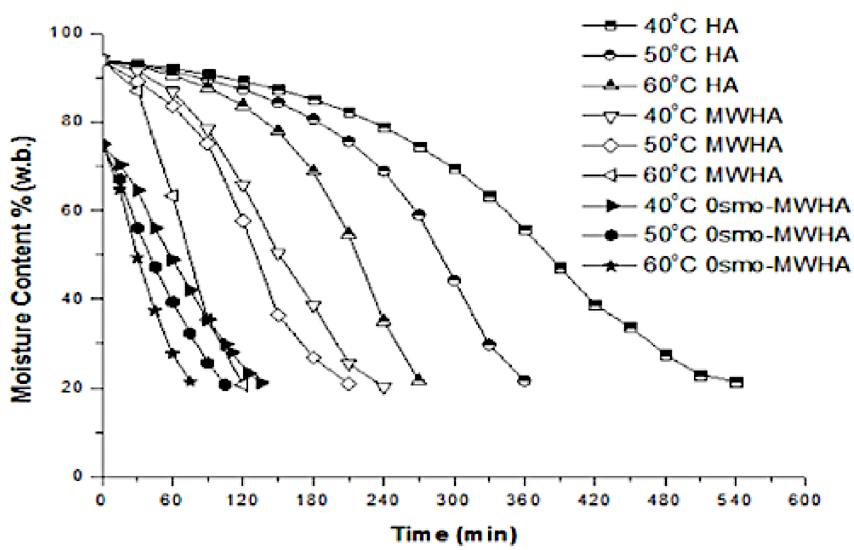


Figure 5.2: Variation of moisture content with time for broccoli slices at different drying temperatures under different drying methods.

The decrease in drying time under microwave-assisted hot air drying compared to hot air drying alone has been reported by Dev et al. (2011) for drumsticks. By using osmotic dehydration as a pre-treatment step, the initial moisture content of broccoli slices was reduced to 75% (w.b.). This water removal has increased the mass transfer rate at early drying period. From the Osmo-MWHA curves, it is clearly shown that osmotic dehydration gives a significant effect on the drying time. The drying time of osmotically dehydrated broccoli stalk under microwave-assisted hot air drying was found to have a shorter drying time compared to untreated samples to achieve the required moisture content.

5.3.2 Mathematical Modelling of Osmo-MWHA

Figure 5.3 show the fitted graph of the four mathematical drying models as listed in Table 5.1 (Equation 5.3 – 5.6) to the experimental data of Osmo-MWHA. Parameters of the thin layer models were estimated as presented in Table 5.2. The results show that Page model was found to be the best to describe the drying of Osmo-MWHA of broccoli slices because it provided an excellent fit with the highest R^2, and lowest χ^2 and *RMSE* at 40, 50 and 60 °C.

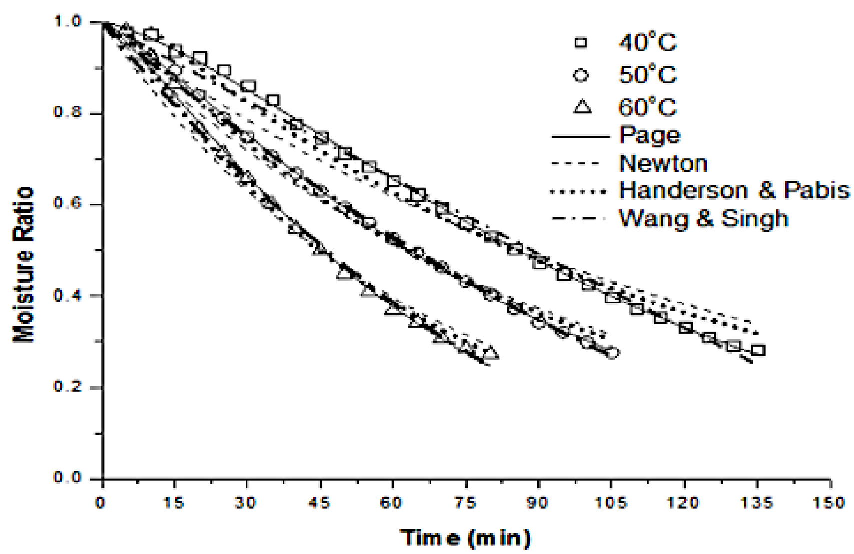


Figure 5.3: Mathematical modelling of Osmo-MWHA broccoli slices at different drying temperatures.

Table 5.2: Model parameters and performance of the thin layer model of Osmo-MWHA at different drying temperatures.

Drying temperature	Model	Parameter		χ^2	RMSE	R^2
40 °C	Page	k=1.40x10^{-3}	n=1.39	3.65x10^{-5}	6.03x10^{-3}	0.999
	Newton	k=8.02x10^{-3}		2.15x10^{-2}	4.64x10^{-2}	0.962
	Handerson & Pabis	a=1.08	k=1.51x10^{-2}	1.03x10^{-3}	3.21x10^{-2}	0.982
	Wang & Singh	a=-5.80x10^{-3}	b=1.77x10^{-6}	3.97x10^{-4}	1.99x10^{-2}	0.993
50 °C	Page	k=4.76x10^{-3}	n=1.20	2.08x10^{-5}	4.56x10^{-3}	0.999
	Newton	k=1.10x10^{-2}		6.73x10^{-4}	2.60x10^{-2}	0.987
	Handerson & Pabis	a=1.04	k=1.17x10^{-2}	3.53x10^{-4}	1.88x10^{-2}	0.993
	Wang & Singh	a=-8.99x10^{-3}	b=1.93x10^{-5}	6.13x10^{-5}	7.83x10^{-3}	0.998
60 °C	Page	k=5.85x10^{-3}	n=1.25	2.08x10^{-4}	1.44x10^{-2}	0.998
	Newton	k=1.54x10^{-2}		1.30x10^{-3}	3.60x10^{-2}	0.979
	Handerson & Pabis	a=1.07	k=1.69x10^{-2}	5.65x10^{-4}	2.38x10^{-2}	0.991
	Wang & Singh	a=-1.27x10^{-2}	b=4.07x10^{-5}	5.67x10^{-4}	2.38x10^{-2}	0.991

5.3.3 Effective Moisture Diffusivity

The values of moisture effective diffusivity (D_{eff}) for different temperatures of Osmo-MWHA broccoli slices are given in Table 5.3. It can be seen that the values were comparable within the general range of 10^{-6} to 10^{-11} m^2/s for food samples (Zarein et al., 2013). The value of D_{eff} increased with increase of drying temperature.

Table 5.3: Effective moisture diffusivity of Osmo-MWHA broccoli slices.

Drying temperature (°C)	Effective moisture diffusivity , $\boldsymbol{D_{eff}}$ (m^2/s)
40	3.63×10^{-8}
50	4.52×10^{-8}
60	6.41×10^{-8}

5.3.4 Color Changes

It was observed that drying conditions affected the final color of the dried samples as shown in Table 5.4. The results indicated that a* (green) value of fresh and dried sample under Osmo-MWHA are not significantly different compared to untreated sample. These effects are possibly due to sucrose used as osmotic agent which was found to be a good inhibitors of polyphenoxidase (PPO) (Khan, 2012). PPO is a main enzyme that involves oxidative browning in many vegetables and fruits (Yamada et al., 2009). For Osmo-MWHA, the results show that total color changes were increased with increasing drying temperature. Highest retention of color was observed under Osmo-MWHA at 40 °C compared to untreated samples.

Table 5.4: Color parameters of fresh and dried broccoli slices.

System	Drying Temp.	*L**	*a**	*b**	ΔE
Fresh		77.37 ± 0.67^{ab}	-8.75 ± 0.87^{b}	21.17 ± 1.72^{c}	-
HA	40 °C	76.23 ± 1.55^{abc}	-11.35 ± 0.30^{bcd}	34.42 ± 1.30^{ab}	11.61 ± 1.79^{b}
MWHA		79.29 ± 1.55^{a}	-9.83 ± 0.56^{bc}	30.45 ± 2.17^{b}	10.11 ± 1.36^{b}
Osmo-MWHA		78.86 ± 1.44^{abc}	-11.72 ± 0.11^{bc}	35.38 ± 2.90^{a}	9.87 ± 0.67^{b}
HA	50°C	74.72 ± 2.24^{bc}	-12.87 ± 0.76^{d}	35.23 ± 0.66^{ab}	17.81 ± 0.54^{a}
MWHA		76.45 ± 0.24^{abc}	-12.47 ± 1.51^{d}	33.08 ± 1.89^{b}	12.67 ± 2.90^{b}
Osmo-MWHA		79.50 ± 1.11^{abc}	-9.66 ± 0.66^{bc}	35.81 ± 1.64^{a}	11.59 ± 2.59^{b}
HA	60°C	75.66 ± 2.16^{abc}	-11.77 ± 1.36^{cd}	31.80 ± 0.72^{b}	10.60 ± 1.86^{b}
MWHA		72.63 ± 1.86^{bc}	-5.56 ± 0.30^{a}	31.59 ± 2.25^{b}	13.08 ± 0.93^{b}
Osmo-MWHA		72.50 ± 2.60^{abc}	-7.37 ± 1.38^{bc}	44.31 ± 0.22^{a}	13.63 ± 1.72^{b}

Column–wise values followed by the same letter are not significantly different. Test used: Duncan's multiple range test ($p < 0.05$)

5.3.5 Shape Changes

Food shape is one of the important quality criteria that need to be conserved carefully (Fernández et al., 2005). Table 5.5 shows the results of shape analysis under different drying conditions. Higher circularity was observed when osmotic dehydration method is applied as a pre-treatment at different temperatures. The improved appearance of dried broccoli slices due to osmotic dehydration pre-treatment can be clearly seen in Figure 5.4. Broccoli slices dried under the MWHA results in curling effects which are similar to the results reported by Singh et al. (2013) on potato slices. The results of this study indicated that broccoli slices pre-treated with osmotic dehydration provides a uniform moisture distribution within the broccoli slices.

Table 5.5: Shape analysis of fresh and dried broccoli slices

System	Temperature (°C)	Circularity
Fresh sample	-	0.84 ± 0.04^{a}
HA	40	0.63 ± 0.05^{bc}
MWHA		0.57 ± 0.06^{cde}
Osmo-MWHA		0.77 ± 0.06^{b}
HA	50	0.64 ± 0.06^{bc}
MWHA		0.48 ± 0.06^{de}
Osmo-MWHA		0.67 ± 0.06^{b}
HA	60	0.58 ± 0.05^{cd}
MWHA		0.52 ± 0.01^{de}
Osmo-MWHA		0.75 ± 0.05^{b}

Column–wise values followed by the same letter are not significantly different. Test used: Duncan's multiple range test ($p < 0.05$)

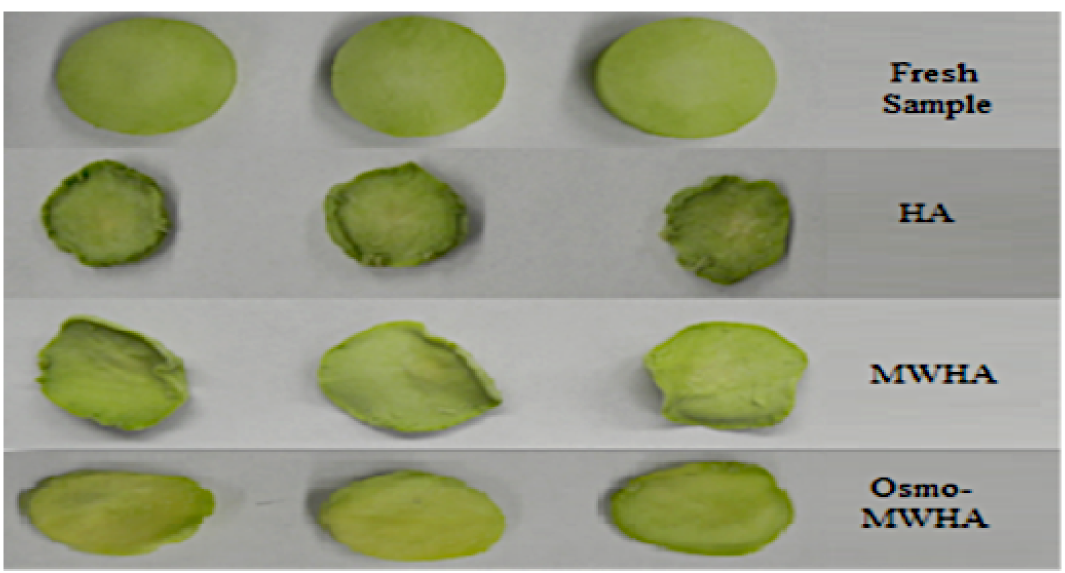


Figure 5.4: Images of fresh and dried broccoli stalk slices.

5.4 Conclusion

From the above results, it was concluded that osmotic dehydration pre-treatment prior to microwave-assisted hot air drying was a beneficial drying method to produce dehydrated products from broccoli stalk. The dried broccoli stalk slices by Osmo-MWHA not only took shorter drying time, but also produced a better quality dried products. Better shape retention, less shrinkage and less color changes were observed, especially when drying was done at lower drying temperature. Hence, promotion of broccoli stalk as a ready-to-eat product could be an alternative marketing option for the broccoli industry which in turn could lead to the minimization of losses leading to better economic return to the producers.

CONNECTING STATEMENT FOR CHAPTER 6

In **Chapter 5**, the application of osmotic dehydration prior to microwave-assisted hot air drying has been demonstrated as a means to reduce the drying time and improve the quality of dried products. However, slower mass transfer observed during osmotic dehydration process under static bath needs to be improved. Thus, in the next chapter, continuous flow osmotic dehydration equipment system was developed and the performance of the equipment was investigated. This new equipment could provide better hydrodynamic control during the osmotic dehydration process and subsequently increase the mass transfer process.

CHAPTER 6

DESIGN OF CONTINUOUS FLOW OSMOTIC DEHYDRATION EQUIPMENT AND ITS PERFORMANCE ON MASS TRANSFER EXCHANGE DURING OSMOTIC DEHYDRATION OF BROCCOLI STALK SLICES

Abstract

An equipment with continuous flow solution possibility for osmotic applications was designed and built with a sole purpose of achieving better hydrodynamic control during the osmotic dehydration pre-treatment process. The initial study was set up to calibrate the flow meter at different sucrose solutions at different concentrations and temperatures to obtain a flow velocity range between 1.5 to 3.5 mm/s. In this study, broccoli stalk slices were used to investigate the effect of the flow velocity on mass transfer kinetics and compared with static condition. Further, the optimization of this equipment system was performed to achieve higher water loss with minimal solute gain as pre-drying condition. Comparative studies between static and dynamic conditions show that flow velocity helps in faster rate of water removal with lower solute gain during the osmotic dehydration process of broccoli stalk slices. The optimum conditions was found to be at a temperature of 30 °C with a sucrose concentration of 54 °Brix for 120 min of immersion time at a flow velocity of 3.5 mm/s.

Keywords: Osmotic dehydration, Flow velocity, Broccoli stalk, Mass transfer, Optimization.

6.1 Introduction

The major challenge in food dehydration system is longer processing time required for water removal in food, which can lower the nutritional quality and increase the energy use (Orsat et al., 2007). Therefore, osmotic dehydration is considered as a very promising technique to address this issue. Osmotic dehydration is a process of water removal by contacting food products with the concentrated osmotic solution. Lewicki and Lenart (1995) reported that by osmotic dehydration, it needs only 0.1-2.4 MJ per kg of removed water compared to convective drying which needs about 5 MJ per kg of evaporated water. Furthermore, osmotic dehydration was found to be advantageous in improving the quality of food product (Azoubel et al., 2015; Tonon et al., 2007; Zhao et al., 2013; Zhao et al., 2014b).

Broccoli is one of the commonly consumed cruciferous vegetables, known for its great source of nutrients such as glucosinolates, vitamins, minerals, and phenolic components that are vital for human health (Jeffery & Araya, 2009; Latté et al., 2011). Typically, broccoli is usually marketed for its floret, leaving a considerable amount of harvest (Bekhit et al., 2013). The stalk, which habitually are discarded contains useful food with a higher nutritional profile still left in them (Aguilo-Aguayo et al., 2015; Alvarez-Jubete et al., 2013; Singh et al., 2011). Adding value to the stalk is beneficial in a number of ways, such as reducing food losses, providing more food for human consumption, and also creates a better economic return for broccoli producers. Our preliminary investigation proved that by applying osmotic dehydration on broccoli stalk slices prior to microwave-assisted hot air drying not only enhanced the drying performance but also improved the quality of the dried product compared to untreated samples (Md Salim et al., 2014).

Conventionally, osmotic dehydration process is conducted under static bath due to its simple operation. However, it appears that this technique takes longer processing time due to slow mass transfer process (Md Salim et al., 2016b). This phenomenon happened due to non-renewal solution around the samples, as the water is removed from the food products, leading to diluted contact zone around the sample (Tonon et al., 2007). To combat the issue, solution renewal during the osmotic dehydration process becomes an important factor to be considered. The elimination of external resistance through solution renewal can be achieved by applying the flow circulation around the osmotic contactor system.

Flow is a term used to exemplify the fluid motion and it can be categorized as laminar, turbulent and transient. Laminar flow is a smooth and orderly flow pattern, whereas turbulent flow has a continuously irregular pattern, rough and chaotic (Shubert & Leyba, 2013). Therefore, laminar flow condition could be a promising option to achieve better hydrodynamic control during osmotic dehydration process and subsequently, enhance the mass transfer exchange during the osmotic dehydration process. However, studies on quantification of laminar flow conditions in osmotic dehydration are still limited in the literature. Previous study conducted by Moreira and Sereno (2003) shows that effects of the laminar flow conditions during osmotic treatment exhibits satisfactory results as it reduces up to two hours of osmotic time under static conditions without affecting the solute gain. Meanwhile, Li and Ramaswamy (2006) found that there are no significant changes to water loss and solute gain when operated at a laminar flow conditions compared to conventional osmotic method. Therefore, the effects are still not conclusive in osmotic dehydration process. For this reason, osmotic dehydration equipment with continuous flow of solution was designed and developed.

Basically, the main components for liquid transport system for obtaining the continuous flow osmotic dehydration are solution tank, pipeline, pump, fittings and the contactor. The solution is transported to the osmotic contactor through the pipeline with the mechanical energy provided by the pumps (except for the situations where gravity system is feasible and are used). Fittings such as flow meter, valve or elbow are installed to control the pump to achieve the desired flow rates. The variable area type flow meter is one of the most convenient, reliable and economical components in indicating the flow rates in a fluid system. This flow meter consists of a uniformly tapered flow tube placed in a vertical position, a float, a reading scale and a control valve. The working principle of this flow meter is given in Cole Palmer (2006); when fluid enters the tube, it passes the float from its initial position at the flow tube's inlet, forcing the float up in the tube. The float rises until a sufficient annular opening is large enough to allow the entire volume of the fluid to flow past the float. At this position, the float is in equilibrium and its height is proportional to the flow rate. The reading scale on the flow meter can be either directly read or in reference scale units (Raju, 2011). Commonly, the reading scale is calibrated for pure water at specific temperatures and pressures. It should be noted that changes of fluid properties would affect the float's position. In a nutshell, control of the flow rate at different osmotic solution conditions is achieved through the calibration.

Therefore, the objectives of this study are: (1) To evaluate the flow characteristics of different sucrose solution conditions in this osmotic dehydration equipment system; (2) To perform calibration of flow meter as an indicator to control the flow at different levels of velocity at different sucrose concentration conditions; (3) To investigate the effectiveness of the flow velocity on mass transfer exchange compared to static condition; and (4) To evaluate the effect of processing factors and determine the optimum condition to achieve higher water removal with minimal solute gain during the osmotic dehydration of broccoli stalk slices under continuous flow condition.

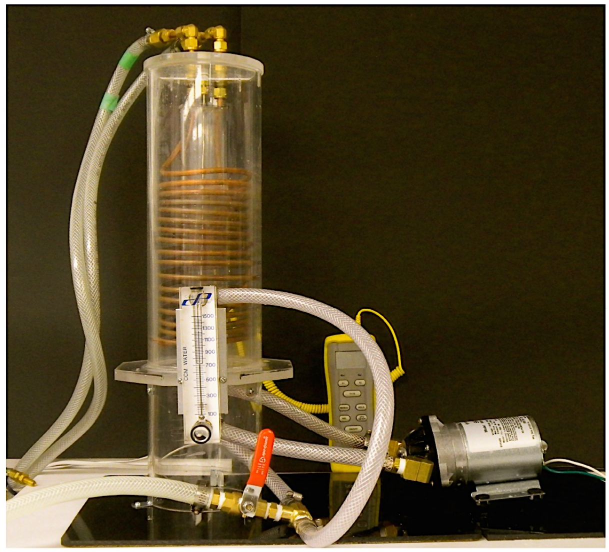
6.2 Experimental Procedures

6.2.1 Sample Preparation

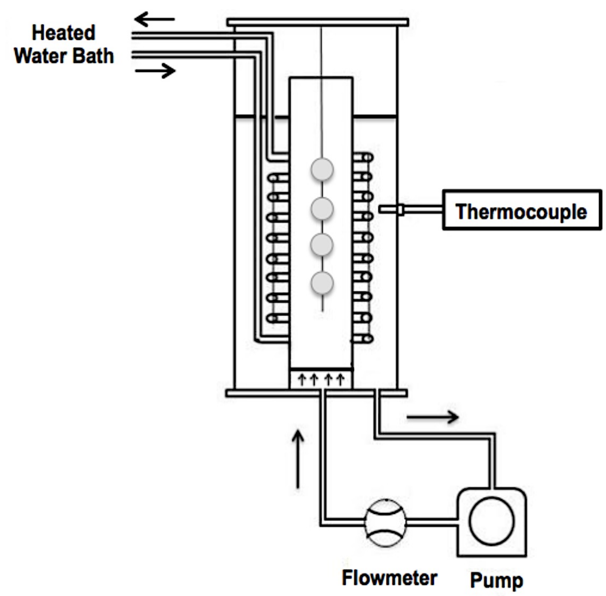
The broccoli stalks (*Brassica oleracea* L. var. *italica*) were washed, peeled and sliced to a uniform shape with a thickness of 6 mm and a diameter of 23 mm. The thickness was chosen based on the preliminary investigation. Moisture content of samples were determined by drying them in a hot air oven at 105 °C (Thermo Scientific Model 6528, Rockford, IL, USA) until a constant mass was reached (AOAC 935.29) (AOAC, 2000). Sucrose (Redpath Sugars, Montreal, QC, Canada) was used as an osmotic agent and it was purchased locally. The osmotic solutions were prepared by dissolving the sucrose in the proper amount of distilled water.

6.2.2 Design of an Osmotic Dehydration Equipment for Continuous Flow System

Continuous flow osmotic dehydration equipment and its schematic diagram are shown in Figure 6.1 (a) and (b) respectively. The main components of this closed loop system consist of two acrylic cylindrical column named as outer cylinder and inner cylinder, pump (Pentair SHURflo 8000, New Brighton, MN, USA), thermocouple, heating/cooling unit (Isotemp 1013S, Fisher Scientific Inc., Pittsburgh, PA, USA), sample holder and variable area flow meter (Cole Palmer Instrument Co. Vernon Hills, IL, USA). The outer cylinder with a height of 350 mm and a diameter of 115 mm was designed as a water bath system controlled by the heating/cooling unit. The inner cylinder with a height of 300 mm and a diameter of 45 mm was designed as a contact system where the sample holder hanging from the top is placed at the center of the cylinder. In brief, the working principle of this equipment is that the osmotic solution is poured into the outer cylinder and once the osmotic solution reached the required temperature, the solution is transported to the inner cylinder through the pipeline with the mechanical energy provided by the pump. A flow meter was installed to monitor the pump to achieve a desired flow rate. Flow straightener was installed at the entry of the flow to provide uniform flow profile.



(a)



(b)

Figure 6.1: (a) Continuous flow osmotic dehydration equipment system; (b) Schematic diagram of the system.

6.2.3 Flow Characteristics of the Sucrose Solution and the Conditions Achieved in the Continuous Flow System

The transport of osmotic solution to the contactor is directly related to the liquid properties, primarily viscosity and density. Different osmotic agents have different density and viscosity at various solution concentrations operated at different temperatures. Those properties also influence the flow characteristics within the contactor system. In this section, the sucrose solutions were prepared with concentrations of 40, 50 and 60 °Brix and the viscosities of sucrose solution were measured by using a rheometer (AR2000, TA Instruments, Texas, USA) with 40 mm parallel plate geometry. The densities of sucrose solution were determined by measuring the solution's mass at a known volume. All the experiments were replicated thrice.

The flow characteristics were determined by calculation of the Reynolds number as shows in Equation 6.1. Reynolds number was used to categorize laminar flow ($Re <$ 2100), transition flow (2100 $< Re <$ 4000) and turbulent flow ($Re >$ 4000). The selection of flow velocity at a range of 1.5 - 3.5 mm/s was used in this study.

$$Re = \frac{\rho v D}{\mu} \qquad (6.1)$$

Where Re is the Reynolds number (dimensionless number), ρ is the density of the fluid (kg/m^3), v is the velocity in the contactor system (m/s), D is the diameter of the contactor system (m), and μ is the viscosity of the fluid (Pa.s).

6.2.4 Flow Meter Calibration

The calibration of the flow meter was carried out by collecting the volume of liquid over a period of time to determine the actual flow rate at a specific osmotic solution condition (Paton, 2005). In this study, the calibration was performed using sucrose solution at the concentrations of 40, 50 and 60 °Brix. For each concentration, the experimental design using full factorial concept with three different temperatures (25, 45 and 65 °C) at five indicated flow meter reading scale ranging from 500 to 1300 cm^3/min. Three replicates were used for data collection. The experimental data was analyzed using statistical software, JMP version 11(SAS Institute, Cary, NC, USA). Predictive equations of flow meter calibration were established.

6.2.5 Flow Velocity Calculation

Model equation obtained from the previous section was used to calculate the actual flow rate of sucrose solutions at temperatures of 30, 40 and 50 °C for evaluation in further studies. The computed actual flow rate was then divided by the cross sectional area of the equipment system to determine the flow velocity (Equation 6.2). Predictive equation to provide the relationship of flow meter reading scale and the flow velocity for each sucrose concentration at different temperatures were established.

$$v = \frac{Q}{A} \tag{6.2}$$

where v is the velocity (m/s), Q is the volumetric flow rate (m^3/s) and A is the cross sectional area (m^2).

6.2.6 Comparison with Static and Continuous Flow Conditions

In this study, sucrose concentration of 40 °Brix was poured into the outer cylinder. Once the solution reached the required temperature (30, 40 or 50 °C), the solution was pumped into the inner cylinder (contactor system) at a required velocity. The solution flowed for about 10 min before the immersion of the samples; to ensure a steady-state flow condition. For each trial, flow velocity of zero (static condition) and 3.5 mm/s (flow condition) was used to evaluate the comparative mass transfer exchange during the osmotic dehydration process.

The terminology used to evaluate the mass exchange between sample and solution (for broccoli stalk slices and sucrose solution) during osmotic dehydration were calculated as follows (Hawkes & Flink, 1978) :

$$Water\ Loss\ (WL) = \frac{w_{wo} - (w_t - w_{st})}{w_{wo} + w_{so}} \text{ x } 100 \tag{6.3}$$

$$Solids\ Gain\ (SG) = \frac{w_{st} - w_{so}}{w_{wo} + w_{so}} \text{ x } 100 \tag{6.4}$$

$$Performance\ Ratio\ (PR) = \frac{WL}{SG} \tag{6.5}$$

Where W_{wo} is the mass of water in the sample before dehydration (g), W_t is the mass of sample after dehydration (g), W_{so} is the mass of the solids in the sample before dehydration (g) and W_{st} is the mass of solids in the sample after dehydration (g).

In addition to the detailed experimental work, modeling steps were carried out. The mass transfer kinetics during osmotic dehydration of broccoli stalk slices under both conditions was modeled according to the Peleg model (Peleg, 1988), Magee model (Magee et al., 1983) and penetration model (Hawkes & Flink, 1978):

Peleg model $$WL \text{ or } SG = \frac{t}{k_1 + k_2 t} \tag{6.6}$$

Magee model $$WL \text{ or } SG = A + k\sqrt{t} \tag{6.7}$$

Penetration model $$WL \text{ or } SG = k\sqrt{t} \tag{6.8}$$

Where A and k are constants and t is time (min).

The statistical parameters such as the reduced chi-square (χ^2), root mean square error ($RMSE$) and coefficient of determination (R^2) are used in selecting the best equation to describe the osmotic kinetic equation. The highest R^2 and the lowest χ^2 and *RMSE* values required were used to evaluate the goodness of fit.

$$R^2 = 1 - \frac{\sum_{i-1}^{i=n}\left(X_i^{exp} - X_i^{pred}\right)^2}{\sum_{i=n}^{i=n}\left(X_i^{exp} - \bar{X}^{exp}\right)^2} \tag{6.9}$$

$$\chi^2 = \sum_{i-1}^{i=n} \frac{(X_i^{pred} - X_i^{exp})^2}{n-m} \tag{6.10}$$

$$RMSE = \sqrt{\frac{1}{n}\sum\nolimits_{i=1}^{n}(X_i^{pred} - X_i^{exp})^2} \tag{6.11}$$

Where *n* is the number of observations, X_i^{pred} is the *i*th predicted value of WL or SG, X_i^{exp} is the *i*th experimental value of WL or SG, *m* is the number of variables and $\bar{X}^{exp}$ is the mean of the experimental value of WL or SG.

6.2.7 Effect of Processing Factors on Mass Transfer Exchange During Osmotic Dehydration Under Continuous Flow System.

The response surface methodology was used to estimate the main effects of osmotic dehydration process under continuous flow condition. The experiments were designed according to face-centered central composite design using the statistical software JMP version 11 (SAS Institute, Cary, NC, USA). The face-centered central composite design was chosen due to the required limited experimental range. Additionally, this design uses only three levels of each factor (-1, 0 and +1), which helps in reducing the number of observations and lessen the experimental cost. Three replicates were taken at the design center, so that the total number of experimental runs was n = 2^4 full factorial design + 8 axial points + 3 center points = 27. The four continuous independent variables were temperature (X_1), solution concentration (X_2), flow velocity (X_3) and processing time (X_4), which were examined at three different levels as shown in Table 6.1.

Table 6.1: Process variables and the levels of the osmotic dehydration under continuous flow conditions of broccoli stalk slices.

Independent Variables		Variation intervals		
		-1 (low)	0 (center)	+1 (high)
$\boldsymbol{X_1}$ **:**	Temperature (°C)	30	40	50
$\boldsymbol{X_2}$ **:**	Sucrose Concentration (°Brix)	40	50	60
$\boldsymbol{X_3}$ **:**	Velocity (mm/s)	1.5	2.5	3.5
$\boldsymbol{X_4}$ **:**	Time (minutes)	60	120	180

The WL (Y_1), *SG* (Y_2) and WL/SG (Y_3) obtained from experimental data are the dependent variables, also referred as responses which were subjected to an analysis of variance (ANOVA) in order to determine the significant effects of independent variables on each response. The following second-order polynomial model was used for the statistical analysis:

$$Y = b_0 + \sum_{i=1}^{k} b_i X_i + \sum_{i=1}^{k} b_{ii} X_i^2 + \sum_{i=1}^{k-1} \sum_{j=i+1}^{k} b_{ij} X_i X_j \tag{6.12}$$

Where Y is the response (i.e. *WL, SG* and *WL/SG*); b_n are the regression coefficients; X_1, X_2, X_3 and X_4 are the coded independent variables.

6.3 Results and Discussion

6.3.1 Flow Characteristics

Reynolds number is a useful non-dimensional parameter to identify how the fluid would behave under selected flow conditions. Certainly, based on Equation 6.1, viscosity and density are the important physical property that influences the Reynolds number. The experimental results on sucrose viscosity at concentration of 40, 50 and 60 °Brix are presented in Figure 6.2. Regression approach to model the correlation of temperature and the viscosity was used. The regression equation for all sucrose solution is presented in Table 6.2. From the results, it is apparent that the viscosity of sucrose solution changes with the temperature; the higher the temperature, the lower the viscosity. The density of sucrose solutions at different temperatures is listed in Table 6.3. The result shows that the density decreases as the temperature increases.

The selection of flow velocity in this study was dependent on the range of the flow pattern of sucrose solution conditions under laminar flow range. Figure 6.3 provides the schematic diagram of flow past the circular solid surface at different *Re*. From the illustrated figure, it is learnt that *Re* affects the flow pattern in laminar flow. When *Re* exceeds 60 the rear flow becomes unstable, as shown in Figure 6.3 (c). Therefore, this value was considered while selecting the flow velocity range in order to have a good contact between the solution and the food sample during mass transfer exchange process under continuous flow condition. Table 6.4 provides the summary of the selection of flow velocity used in the evaluation of the hydrodynamic control of the osmotic dehydration in the continuous flow equipment.

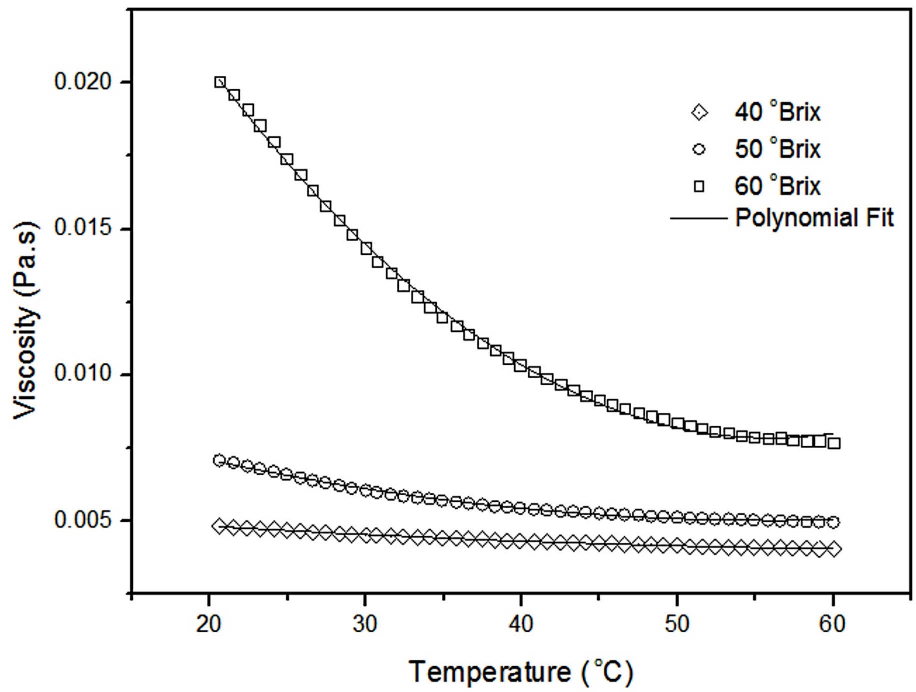


Figure 6.2: The viscosity profile of sucrose solution as a function of temperature.

Table 6.2: Regression equation of viscosity at different sucrose concentrations.

Sucrose Concentration (°Brix)	Model Equation	R^2	Equation No.
40	$y = -4.47 \times 10^{-5}\, x + 3.16 \times 10^{-7} x^2 + 5.60 \times 10^{-3}$	0.99	(6.13)
50	$y = -1.78 \times 10^{-4}\, x + 1.58 \times 10^{-6}\, x^2 + 0.01$	0.99	(6.14)
60	$y = -1.10 \times 10^{-3} x + 9.81 \times 10^{-6}\, x^2 + 0.04$	0.99	(6.15)

Table 6.3: Density of sucrose solutions at different temperatures.

Concentration (°Brix)	Density (g/ml)		
	30 °C	40 °C	50 °C
40	1.183 ± 0.003	1.177 ± 0.003	1.141 ± 0.003
50	1.228 ± 0.003	1.215 ± 0.001	1.209 ± 0.002
60	1.256 ± 0.003	1.232 ± 0.002	1.227 ± 0.003

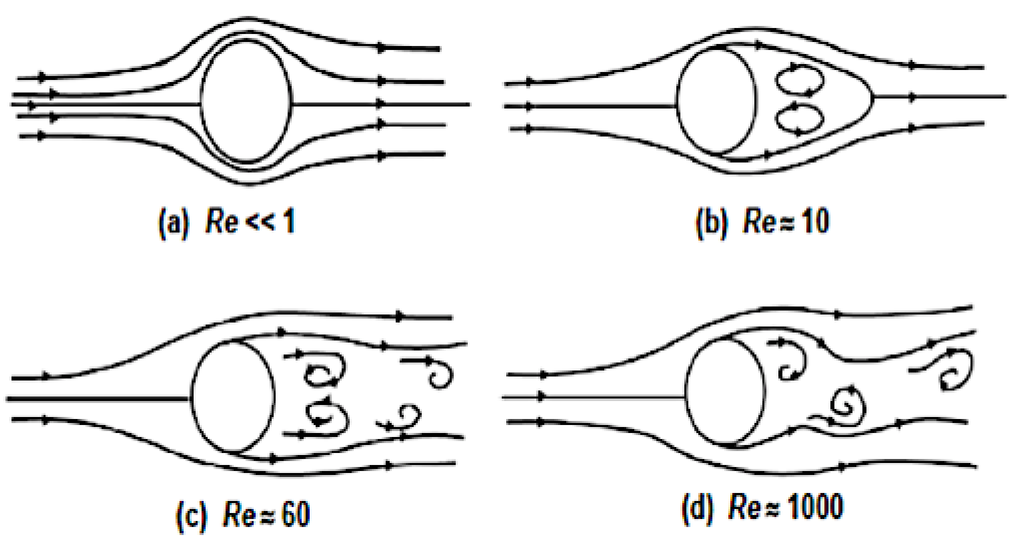


Figure 6.3: Flow pattern around circular solid surface at various range of *Re* (Source: adapted from William et al. (1999)).

Table 6.4: The selection conditions of flow velocity at $Re < 60$.

Temperature (°C)	Concentration (°Brix)	Velocity (m/s)	Re
30	40	1.50×10^{-3}	19.53
		2.50×10^{-3}	32.55
		3.50×10^{-3}	45.57
	50	1.50×10^{-3}	15.09
		2.50×10^{-3}	25.15
		3.50×10^{-3}	35.21
	60	1.50×10^{-3}	6.54
		2.50×10^{-3}	10.90
		3.50×10^{-3}	15.27
40	40	1.50×10^{-3}	20.43
		2.50×10^{-3}	34.08
		3.50×10^{-3}	47.68
	50	1.50×10^{-3}	16.77
		2.50×10^{-3}	27.95
		3.50×10^{-3}	39.13
	60	1.50×10^{-3}	9.00
		2.50×10^{-3}	15.01
		3.50×10^{-3}	21.01
50	40	1.50×10^{-3}	20.60
		2.50×10^{-3}	34.33
		3.50×10^{-3}	48.06
	50	1.50×10^{-3}	17.86
		2.50×10^{-3}	29.77
		3.50×10^{-3}	41.68
	60	1.50×10^{-3}	11.38
		2.50×10^{-3}	18.96
		3.50×10^{-3}	26.54

6.3.2 Flow Meter Calibration at Different Sucrose Concentrations

The interaction of flow meter with the flowing fluid is affected by the properties of the fluid or the velocity distribution of the fluid passing through the device. Therefore, it is desirable to determine the output related to the indicated flow rates to establish the relationship. The Least Square Methods were fitted to the experimental data for each sucrose concentration. The experimental data and the predicted flow rate for sucrose concentration of 40, 50 and 60 °Brix at different temperatures with reference to reading scale of the flow meter is presented in Table 6.5.

Table 6.5: The actual and predicted flow rate at indicated flow meter reading scale of 40, 50 and 60 °Brix at different temperatures.

Temperature (°C)	Indicated flow meter reading scale (cm^3/min)	Flow rate, Q (cm^3/min)					
		40 °Brix		50 °Brix		60 °Brix	
		Actual	Pred.	Actual	Pred.	Actual	Pred.
25	500	358	367	176	179	77	70
	700	500	511	302	303	125	125
	900	650	656	422	426	173	179
	1100	795	800	550	550	238	234
	1300	938	945	672	674	292	288
45	500	402	404	262	257	122	122
	700	557	564	403	399	207	212
	900	715	723	540	540	295	302
	1100	875	883	690	681	385	392
	1300	1038	1042	825	823	485	482
65	500	450	443	335	335	175	173
	700	612	617	482	494	303	300
	900	792	791	642	654	425	425
	1100	958	965	833	813	548	550
	1300	1143	1140	963	972	680	676

The relationship between the temperature and flow meter reading scale to the actual flow rate are described in Equation 6.16 for 40 °Brix, Equation 6.17 for 50 °Brix and Equation 6.18 for 60 °Brix. The statistical analysis for all concentrations indicate that the model is highly significant (p < 0.001) with R^2 of 0.99 and insignificant lack of fit (p = 0.43 for 40 °Brix, p = 0.1 for 50 °Brix and p = 0.08 for 60 °Brix).

$$\text{Actual volumetric flow rate, } Q_{40Brix}\left(\frac{mL}{\text{min}}\right) = -158.61 + 3.57\,X_1 + 0.80\,X_2 + (X_1 - 45)\big((X_2 - 900)\;3.48 \times 10^{-3}\big) \tag{6.16}$$

$$\text{Actual volumetric flow rate, } Q_{50Brix}\left(\frac{mL}{\text{min}}\right) = -353.1 + 5.69X_1 + 0.71\,X_2 + (X_1 - 45)\big((X_2 - 900)\;4.44 \times 10^{-3}\big) \tag{6.17}$$

$$\text{Actual volumetric flow rate, } Q_{60Brix}\left(\frac{mL}{\text{min}}\right) = -379.3 + 6.13X_1 + 0.45\,X_2 + (X_1 - 45)\big((X_2 - 900)\;8.90 \times 10^{-3}\big) \tag{6.18}$$

where X_1 is the temperature (25 °C ≤ X_1≤ 65 °C) and X_2 is the flow meter reading scale (500 cm^3/min ≤ X_2 ≤ 1300 cm^3/min).

6.3.3 Predictive Equation for Flow Meter at Different Velocity Range of Sucrose Solution Concentrations

Figure 6.4 represents the graph of flow meter reading scale versus flow velocity of sucrose solutions at temperatures of 30, 40 and 50 °C. Linear regression is the approach taken to model the correlation of the two parameters. The linear regression equation for all sucrose solution is presented in Table 6.6. Higher value of R^2 obtained indicates that the regression line perfectly fits the data.

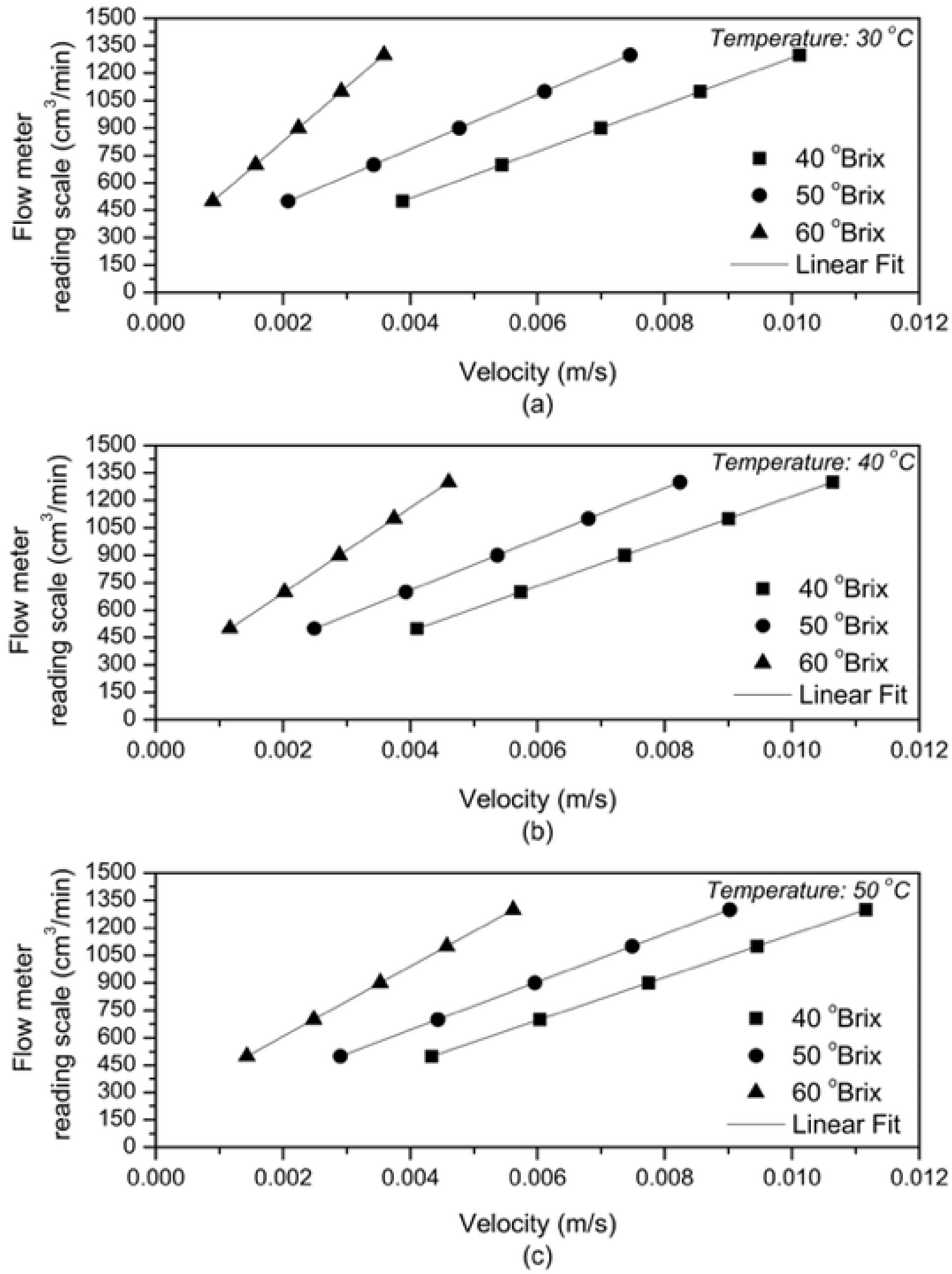


Figure 6.4: The flow meter reading scale as an indicator of flow velocity at sucrose concentration of 40, 50 and 60 °Brix at temperatures of (a) 30 °C; (b) 40 °C; and (c) 50 °C.

Table 6.6: Regression model of flow velocity for sucrose concentrations of 40, 50 and 60 °Brix at temperatures of 30, 40 and 50 °C.

Temperature (°C)	Sucrose concentration (° Brix)	Model Equation	Equation No.
30	40	y = 128143x + 3.55 (R^2 = 1)	(6.19)
	50	y = 148716x + 191.18 (R^2 = 1)	(6.20)
	60	y = 297306x + 234.19 (R^2 = 1)	(6.21)
40	40	y = 122422x - 2.2 (R^2 = 1)	(6.22)
	50	y = 139094x + 154.15 (R^2 = 1)	(6.23)
	60	y = 232726x + 229.23 (R^2 = 1)	(6.24)
50	40	y = 117190x - 7.45 (R^2 = 1)	(6.25)
	50	y = 130641x + 121.63 (R^2 = 1)	(6.26)
	60	y = 191196x + 226.04 (R^2 = 1)	(6.27)

6.3.4 Comparative Study Between Static and Continuous Conditions

The effects of static and continuous flow conditions studied at a concentration of 40 °Brix at temperatures of 30, 40 and 50 °C on mass transfer exchange during osmotic dehydration of broccoli stalk slices are shown in Figure 6.5. For both conditions, after three hours immersion time, the WL was reduced by more than 50%. Furthermore, this finding provides evidence that continuous flow improved the water removal rate during the osmotic dehydration at all temperatures due to renewal of boundary layers around the samples. Interestingly, continuous flow condition reduces the solute uptake compared to the static condition at all temperatures. The plausible explanation for these findings is that the presence of circulation of flow around the boundary layer causes disruption of the solute to diffuse into the food samples. The increase in water loss under continuous flow condition at a low temperature range between 5 °C and 25 °C has been reported by Moreira and Sereno (2003). However, the study reported that the solute gain was not affected while compared to static condition. Temperature usually has a significant effect on solution viscosity, which contributes to the different flow velocity passing through the boundary layer (Asadi, 2006).

The effectiveness of the osmotic process is calculated from obtained mean values of WL and SG and is presented in Table 6.7. It is apparent from the table that the ratio was higher at the beginning stage of the process, after which the rate of water transfer slowed down. While comparing both conditions, continuous flow results in a higher ratio of WL/SG than static condition, e.g. at the first 60 minutes for all temperatures, the ratio was about 30% higher compared to static condition.

The modeling of the mass transfer was carried out using Peleg, Magee and penetration models to fit the experimental data on WL and SG. The parameters obtained from the non-linear regression analysis for the Peleg model are presented in Table 6.8. The parameter $1/k_1$ is related to initial mass transfer rate, which lowers the k_1 thus showing the higher mass transfer rate. It can be observed that the value of k_1 was lower when continuous flow was applied for WL. Contrarily for SG, the k_1 was lower under static condition, indicating that the higher solute was diffused into the product. The k_2 values describe the values at equilibrium, which did not exhibit a trend when both conditions were compared.

Magee model parameters obtained from the non-linear regression analysis are shown in Table 6.9. For WL it can be seen that the parameters A and k are higher under continuous flow condition compared to static condition at all temperatures. For SG, higher parameter value of A and k was obtained under static condition. However, for static condition, the parameter did not demonstrate a trend when the temperature was increased. In contrast for continuous flow, both parameters increased when the temperature increased.

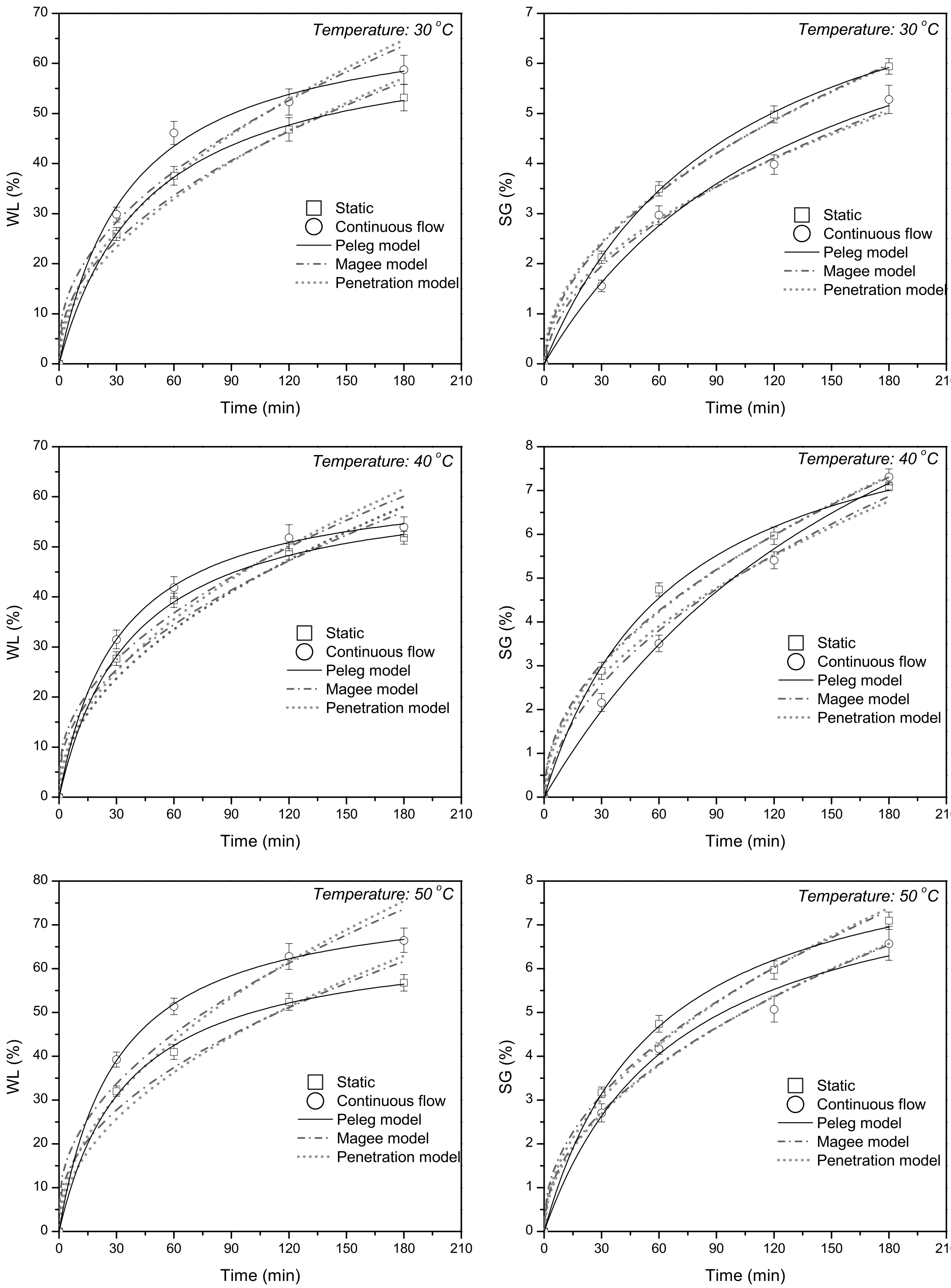


Figure 6.5: Kinetics of osmotic dehydration at a temperature of 30, 40 and 50 °C on water loss (WL) and solute gain (SG).

Table 6.7: Values of WL/SG of 40 °Brix sucrose solution at different temperatures.

Temperature (°C)	Time (min)	Performance Indicator WL/SG	
		Static	Continuous Flow
30	30	12.19	19.17
	60	10.75	15.53
	120	9.40	13.14
	180	8.95	11.12
40	30	9.60	14.59
	60	8.30	11.93
	120	8.20	9.58
	180	7.30	7.39
50	30	10.09	14.50
	60	8.64	12.31
	120	8.78	12.37
	180	8.00	10.11

Parameters obtained for WL and SG using penetration model are presented in Table 6.10. Higher k value can be seen under continuous flow conditions for WL compared to static condition. While for SG, the inverse trend was observed. For static conditions, the parameter of this model increases when the temperature increased for WL and SG. However, under continuous flow, only the SG exhibits an increase of k as the temperature increased.

For both conditions at all temperatures, the Peleg model had an excellent fit with higher value of R^2, lower value of χ^2 and $RMSE$ compared to Magee and penetration model for WL and SG. The Peleg model has been reported as an adequate model in predicting the osmotic dehydration mass transfer kinetics of various biological products such as banana (Mercali et al., 2010) and guava (Ganjloo et al., 2012).

Table 6.8: Value of Peleg model parameters for water loss and solute gain.

Temperature (°C)	Condition	Water Loss (%)					Solute Gain (%)				
		k_1	k_2	χ^2	$RMSE$	R^2	k_1	k_2	χ^2	$RMSE$	R^2
30	Static	0.706	0.015	0.400	0.632	0.999	10.773	0.109	0.001	0.035	0.999
	Continuous Flow	0.531	0.014	3.916	1.979	0.995	15.202	0.109	0.043	0.208	0.992
40	Static	0.593	0.016	0.412	0.642	0.999	6.947	0.104	0.032	0.178	0.996
	Continuous Flow	0.483	0.016	0.451	0.672	0.999	13.243	0.066	0.042	0.204	0.996
50	Static	0.525	0.015	1.177	1.085	0.998	6.278	0.109	0.026	0.161	0.997
	Continuous Flow	0.383	0.013	0.232	0.482	0.999	7.954	0.115	0.102	0.319	0.988

Table 6.9: Value of Magee model parameters for water loss and solute gain.

Temperature (°C)	Condition	Water Loss (%)					Solute Gain (%)				
		A	k	χ^2	$RMSE$	R^2	A	k	χ^2	$RMSE$	R^2
30	Static	2.575	4.007	11.442	3.383	0.980	-0.095	0.453	0.032	0.180	0.996
	Continuous Flow	4.071	4.431	33.696	5.805	0.954	-0.207	0.394	0.091	0.301	0.984
40	Static	3.696	3.972	24.053	4.904	0.959	0.107	0.536	0.104	0.323	0.990
	Continuous Flow	4.908	4.114	35.829	5.986	0.944	-0.183	0.466	0.050	0.222	0.994
50	Static	4.298	4.275	25.119	5.012	0.963	0.207	0.531	0.094	0.307	0.991
	Continuous Flow	6.207	5.024	53.947	7.345	0.944	0.090	0.481	0.073	0.270	0.991

Table 6.10: Value of penetration model parameters for water loss and solute gain.

Temperature (°C)	Condition	Water Loss (%)				Solute Gain (%)			
		k	χ^2	$RMSE$	R^2	k	χ^2	$RMSE$	R^2
30	Static	4.255	10.862	3.296	0.975	0.444	0.027	0.165	0.995
	Continuous Flow	4.824	30.973	5.565	0.944	0.374	0.083	0.288	0.980
40	Static	4.328	22.739	4.768	0.949	0.547	0.082	0.286	0.990
	Continuous Flow	4.587	35.161	5.930	0.927	0.448	0.049	0.221	0.992
50	Static	4.690	25.194	5.019	0.951	0.551	0.086	0.292	0.989
	Continuous Flow	5.623	53.716	7.329	0.925	0.490	0.058	0.240	0.991

6.3.5 Influence of Processing Factors on WL, SG and WL/SG

The experimental data on the mass transfer exchange of broccoli stalk slices under different combination of processing factors is presented in Table 6.11. The response for WL and SG are in the range of 38.04% to 75.11% and 2.97% to 8.94%, respectively. The computed ratio of WL over SG was observed in the range of 9.82 to 17.96. The regression coefficients for the second-order polynomial equations are presented in Table 6.12. Some non-significant terms ($p > 0.05$) were eliminated and the resulting equations were tested for adequancy and fitness by analysis of variance (ANOVA). Table 6.13 summarizes the results for WL, SG and WL/SG. The model as fitted shows significance ($p < 0.05$) and the lack of fit was not significant ($p > 0.05$) with satisfactory values of R^2. As illustrated in Table 6.12, the WL was mainly affected by the processing time and sucrose concentration, followed by the temperature. Meanwhile for SG, temperature and time were observed as the most prominent factor, followed by the flow velocity and sucrose concentration. The interaction effects of temperature with concentration and time are also significant on the SG. For the WL/SG, all the operating factors were significant for linear effect.

To visualize the influence of temperature, solution concentration, flow velocity and immersion time on WL, SG and WL/SG, response surface curves were generated as shown in Figures 6.6 to 6.8. These surface plots were generated considering only the significant regression coefficients according to Table 6.12. Also, these surfaces reflect the influence of two operating variables on WL, SG and WL/SG while the other factors were kept constant at their center point. As presented in Figure 6.6 (a), WL increased as the time and sucrose concentration increased. The longer duration of processing time increases the rate of WL during osmotic dehydration of broccoli stalk slices. According to Akbarian et al. (2014), the WL will continuously increase until it reaches equilbrium state between product and osmotic solution, which commonly approached 20 hours. It also can be observed that WL increases gradually with solution concentration over the entire process which is due to high osmotic driving force between the sample and the solution.

Table 6.11: Experimental conditions and their corresponding values of dependent variables.

Independent Variables				Dependant Variables		
Temperature (°C)	Concentration (°Brix)	Velocity (mm/s)	Time (min)	WL (%)	SG (%)	WL/SG
30	40	1.5	60	46.09	3.96	11.64
30	40	1.5	180	58.73	5.94	9.89
30	40	3.5	60	45.4	2.97	15.29
30	40	3.5	180	56.16	5.28	10.64
30	50	2.5	120	63.42	3.55	17.86
30	60	1.5	60	54.5	3.79	14.38
30	60	1.5	180	75.11	6.05	12.41
30	60	3.5	60	51.73	2.88	17.96
30	60	3.5	180	70.29	5.13	13.7
40	40	2.5	120	53.82	5.48	9.82
40	50	1.5	120	60.18	4.26	14.13
40	50	2.5	60	52.79	3.7	14.27
40	50	2.5	120	62.8	4.88	12.87
40	50	2.5	120	63.74	4.52	14.1
40	50	2.5	120	63.06	4.25	14.84
40	50	2.5	180	69.37	6.04	11.49
40	50	3.5	120	66.26	4.19	15.81
40	60	2.5	120	63.86	4.55	14.04
50	40	1.5	60	43.37	4.28	10.13
50	40	1.5	180	51.68	8.94	5.78
50	40	3.5	60	38.04	3.57	10.66
50	40	3.5	180	54.02	7.73	6.99
50	50	2.5	120	58.83	6.2	9.49
50	60	1.5	60	58.07	3.69	15.74
50	60	1.5	180	70.47	7.02	10.04
50	60	3.5	60	49.22	2.83	17.39
50	60	3.5	180	69.11	6.19	11.16

Table 6.12: Regression coefficients of the second-order polynomial model for water loss (WL), solute gain (SG) and the performance ratio (WL/SG).

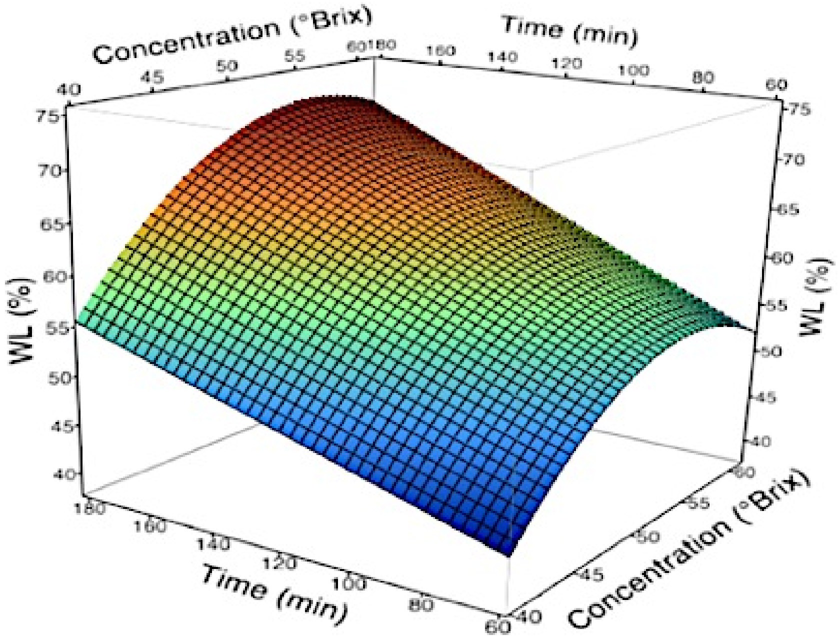
	WL (%)		SG (%)		WL/SG	
Source	Coefficient	*Prob* > F	Coefficient	*Prob* > F	Coefficient	*Prob* > F
Model	62.9858	<.0001	4.6131	<.0001	13.8443	<.0001
X_1 : Temperature	-1.59	0.0169	0.6056	<.0001	-1.4661	0.0007
X_2 : Concentration	6.3917	<.0001	-0.3344	0.0078	1.9989	<.0001
X_3 : Velocity	-0.9983	0.1074	-0.3978	0.0026	0.8589	0.0207
X_4 : Time	7.5406	<.0001	1.4806	<.0001	-1.9644	<.0001
X_1X_2	0.9069	0.1619	-0.2806	0.0269	0.6113	0.0994
X_1X_3	-0.1469	0.8133	-0.0081	0.943	-0.2975	0.4019
X_1X_4	-0.3744	0.5499	0.4194	0.0027	-0.4575	0.2062
X_2X_3	-0.7219	0.2585	0.0031	0.9781	0.0938	0.7888
X_2X_4	1.4856	0.0311	-0.1194	0.3047	-0.2338	0.5077
X_3X_4	0.7019	0.2712	-0.0094	0.9343	-0.315	0.3756
X_{11}	-1.7537	0.2704	0.2304	0.423	-0.1231	0.8877
X_{22}	-4.039	0.0208	0.3704	0.2071	-1.8681	0.0492
X_{33}	0.3413	0.8259	-0.4196	0.1567	1.1719	0.1951
X_{44}	-1.7987	0.2589	0.2254	0.4329	-0.9181	0.3034

Table 6.13: Analysis of variance (ANOVA) for water loss (WL), solute gain (SG) and the performance ratio (WL/SG).

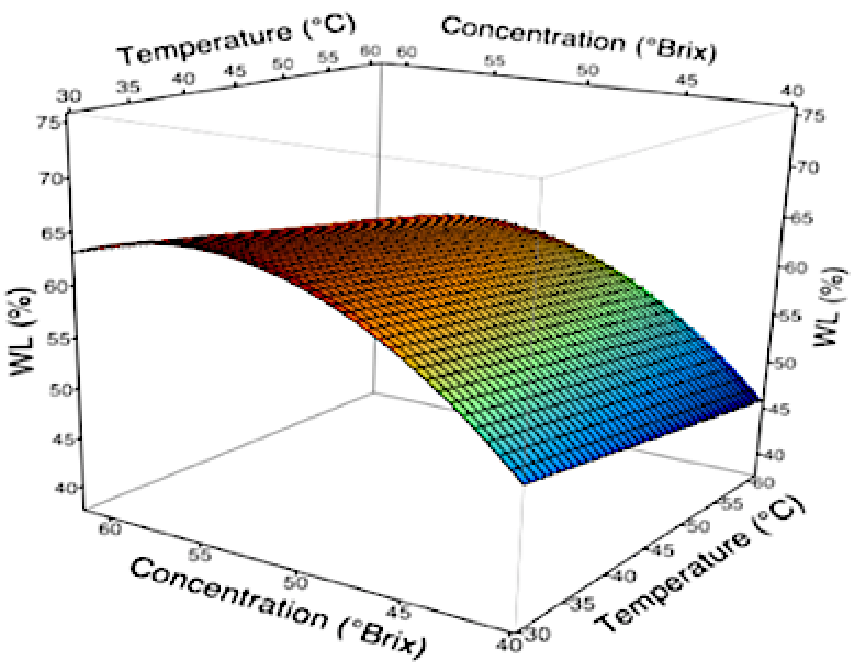
Source	WL			SG			WL/SG		
	d.f.	MS	*F*	d.f.	MS	*F*	d.f.	MS	*F*
Model	5	413.76	58.87*	6	9.17	44.64*	4	48.3	17.91*
Error	21	7.03		20	0.21		22	2.69	
Corrected Total	26			26			26		
Lack of fit	9	5.85	0.74 (n.s.)	18	0.22	2.17 (n.s.)	20	2.87	2.90 (n.s.)
Pure error	12	7.91		2	0.1		2	0.99	
Total error	21			20			22		
R^2	0.93			0.93			0.77		

d.f.= degree of freedom; MS= mean square ; n.s. = non-significant ($p > 0.05$)

* Significant at 5% level



(a)

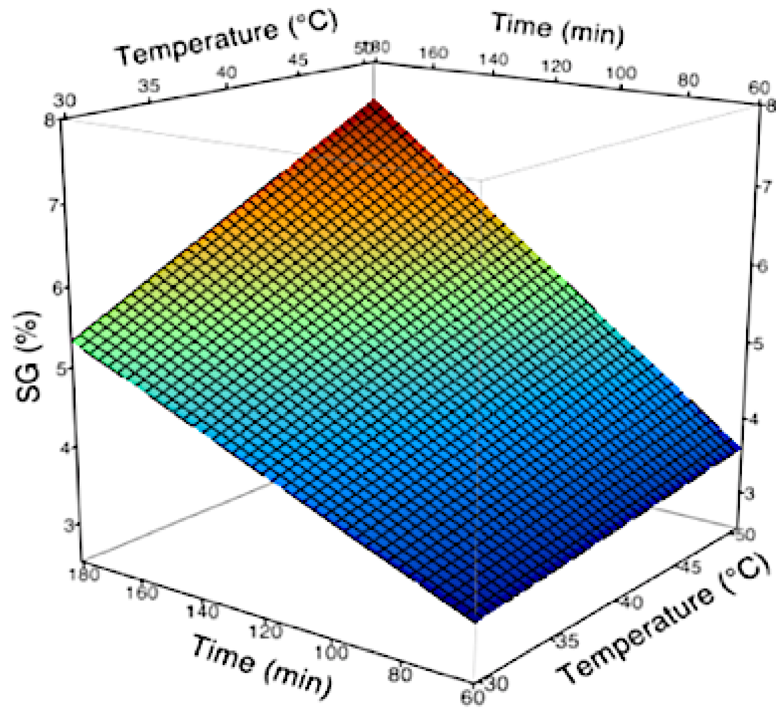


(b)

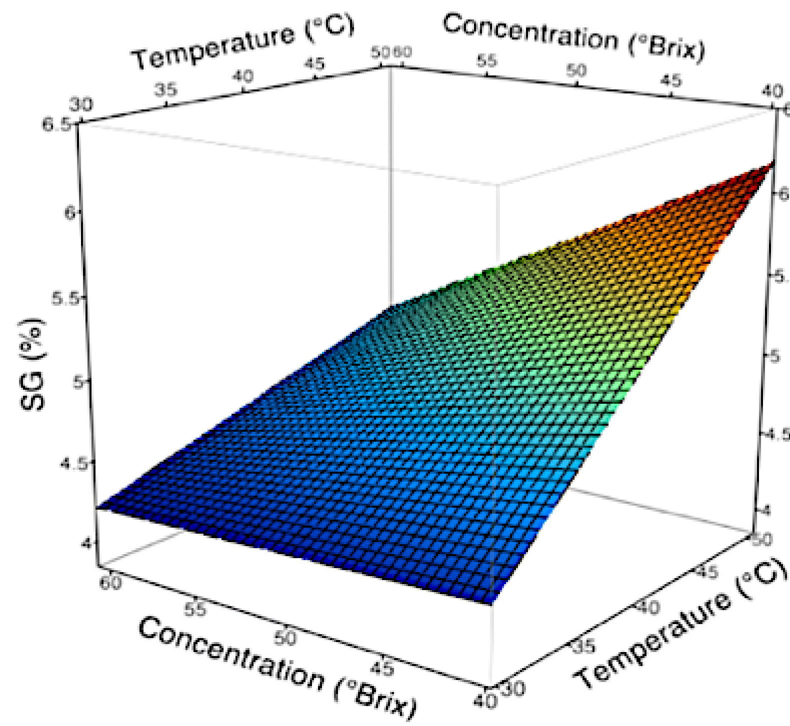
Figure 6.6: Water loss (WL) variation as a function of (a) immersion time and sucrose concentration, and (b) sucrose concentration and temperature.

As shown in Figure 6.6 (b), an increase in temperature was found to slightly lower the WL. Higher temperatures seem to increase the solubility of solids in liquids. Therefore, the osmotic solution becomes more dilute (less concentrated) and it reduces the driving force during the osmotic dehydration process. Moreover, the level of flow velocity was found to be not significantly affecting the WL. This finding was obtained possibly due to the effects of Reynolds number of different conditions of sucrose concentration as computed in Table 6.4, which has a different flow pattern despite having the same velocity. Thus, these findings hint that laminar flow at $Re < 60$ has a crucial influence on mass transfer process during osmotic dehydration conducted under dynamic flow condition.

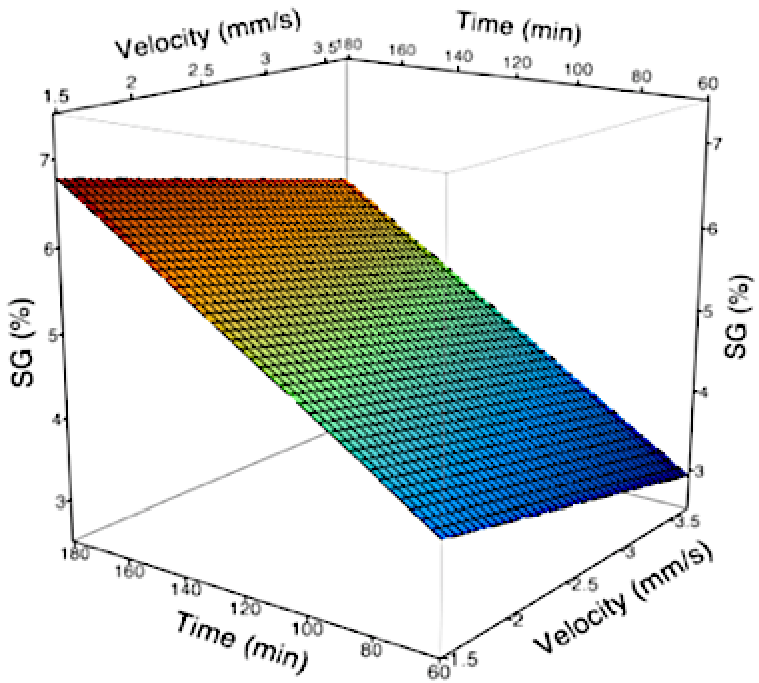
Figure 6.7 (a) depicts variations in SG with changing temperature and processing time. The rate of SG increased with higher temperature and longer duration time. Increase in temperature promotes membrane swelling effect which might increase the cell membrane permeability to sucrose molecules (Lazarides et al., 1997). Similar results were found in the literature for various vegetables and fruits (Azoubel & Da Silva, 2008; Badwaik et al., 2013; Lee & Lim, 2011). In addition, higher values of temperature in combination with longer immersion time produced sharper increases of SG. On the other hand, SG is slightly affected by the sucrose concentration as shown in Figure 6.7 (b). However, through combination of sucrose concentration and temperature, it can be observed that higher temperature with lower sucrose concentration gives higher SG. Contrarily from WL, the effects of flow velocity were found to be significant on SG. As shown in Figure 6.7 (c), the fast flow results in lower SG which probably happens due to the change in hydrodynamic characteristics of external phase that provides mass transfer resistance.


Temperature (°C)
Time (min)
SG (%)
SG (%)
Time (min)
Temperature (°C)

(a)


Temperature (°C)
Concentration (°Brix)
SG (%)
SG (%)
Concentration (°Brix)
Temperature (°C)

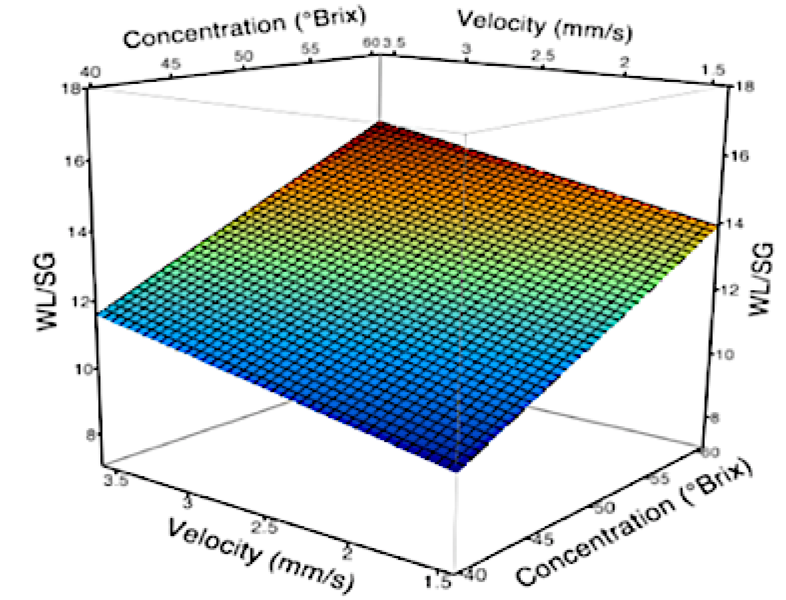
(b)



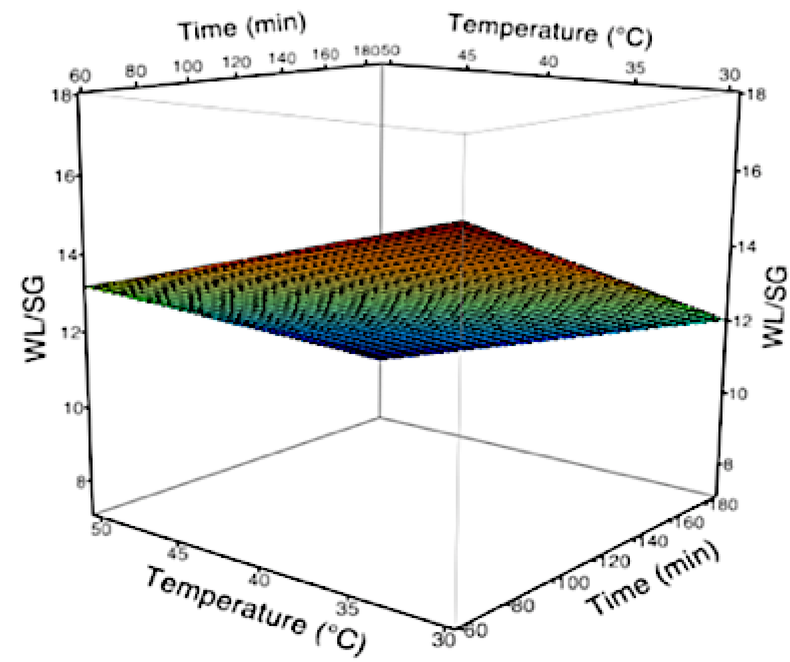
(c)

Figure 6.7: Solute gain (SG) variation as a function of (a) immersion time and temperature; (b) sucrose concentration and temperature; and (c) immersion time and flow velocity.

Higher water removal during the osmotic dehydration process provides less energy use for finish drying. However, excessive solute uptake during the osmotic process can block the surface layer of the product and this additional resistance can lower the water diffusion rate during the finish drying process (Menting et al., 1970). Thus, higher WL/SG provides the information on the effectiveness of this approach as a pre-treatment process. The effect of processing factors on parameter WL/SG are illustrated in Figure 6.8. It can be seen that WL/SG was higher with the increase of flow velocity and sucrose concentration, whereres temperature and time contributed to a lesser extent.



(a)



(b)

Figure 6.8: Performance ratio (WL/SG) variation as a function of (a) flow velocity and sucrose concentration; and (b) immersion time and temperature.

6.3.6 Optimization

A graphical multi-response optimization based on the desirability function was used to identify the optimal operating conditions of the four independent variables: (1) temperature in the range of 30 - 50 °C; (2) sucrose concentration in the range of 40 – 60 °Brix; (3) flow velocity in the range of 1.5 – 3.5 mm/s; and (4) immersion time in the range of 60 to 180 min for osmotic dehydration of broccoli stalk slices as a pre-drying stage. The main criterion for optimization was to achieve higher water removal with minimal solute gain and higher WL/SG. The prediction profiler for all the responses for selected optimum conditions is presented in Figure 6.9.

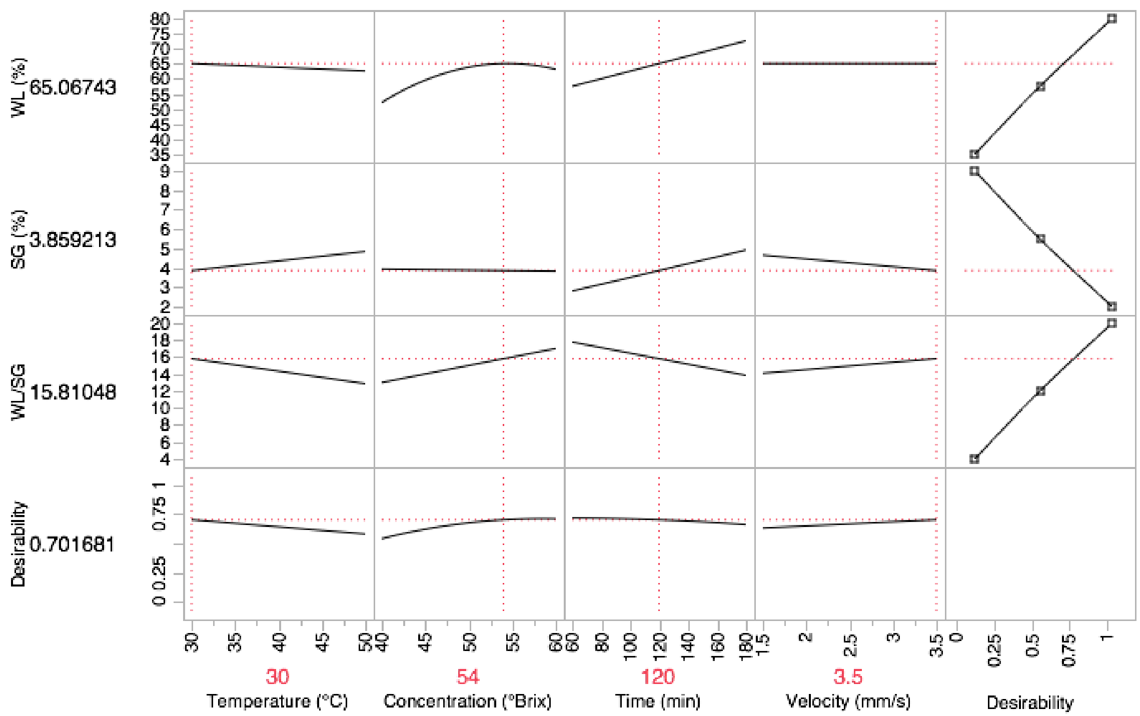


Figure 6.9: Prediction profiler for optimization of osmotic dehydration as pre-drying stage.

The results indicate that to achieve 65 g / 100 g fresh sample of WL with corresponding SG of 3.9 g /100 g fresh sample with the desirability of 0.70, the conditions required are: a temperature of 30 °C with a concentration of 54 °Brix for 120 min of immersion time at a flow velocity of 3.5 mm/s. Verification experiments obtained for WL and SG showed a reasonably close value of 65.8 ± 1.9 g / 100 g fresh sample and 3.8 ± 0.2 g / 100 g fresh sample, respectively and thus confirmed the adequacy of the predicted model.

6.4 Conclusion

In this study, the design and construction of the continuous flow osmotic dehydration system was accomplished. The flow meter used in this study has been calibrated to evaluate the relationship between the flow meter reading scale and the flow velocity of different osmotic solute conditions. The selection of flow velocity for this equipment system ranged from 1.5 to 3.5 mm/s corresponding to $Re < 50$. The use of a laminar flow during the osmotic dehydration of broccoli stalk slices demonstrates the potential improvement in the rate of water removal while minimizing the rate of solute uptake when compared to the static condition. The model proposed by Peleg was satisfactorily applied to fit the WL and SG under both conditions. Further, to provide the details on the performance of this system, the effects of temperature, sucrose concentration, level of flow velocity and immersion time on mass transfer exchange during osmotic dehydration of broccoli stalk slices was investigated using response surface methodology. Analysis of variance shows that temperature, sucrose concentration, flow velocity and immersion time significantly affected the mass transfer process, except flow velocity which did not significantly affect the WL. One of the most significant findings to emerge from this study is that *Re* is another important parameter that needs to be considered to provide better hydrodynamic control during the osmotic dehydration process. Ultimately, in this continuous flow system, water removal of 65% with only 3.9% gain of solute can be accomplished through the temperature conditions of 30 °C with concentration of 54 °Brix at 120 min of immersion time and a flow velocity of 3.5 mm/s.

CONNECTING STATEMENT FOR CHAPTER 7

The performance of newly developed continuous flow osmotic dehydration equipment system has been detailed in the **Chapter 6**. It clearly shows that temperature, sucrose concentration, flow velocity and immersion time significantly affected the mass transfer process during the dehydration process. Thereby, it caused a change to the dielectric properties of the material. The dielectric properties influence the ability of the material to absorb electromagnetic energy during finish drying. In this **Chapter 7**, the changes in dielectric properties as influenced by the osmotic dehydration were investigated.

CHAPTER 7

INFLUENCE OF OSMOTIC DEHYDRATION OPERATING VARIABLES ON DIELECTRIC PROPERTIES OF BROCCOLI STALK SLICES

Abstract

The variation in dielectric constant, ε' and the loss factor, ε'' of broccoli stalk slices after osmotic dehydration process conducted under continuous flow condition was studied using an open-ended coaxial probe technique. The results showed that both ε' and ε'' were significantly influenced by all the osmotic dehydration operating variables. The relationship of osmotic dehydration operating variables on the dielectric properties of the broccoli stalk slices could be established by a second-order polynomial model, with the coefficient of determination higher than 0.9. Compared to fresh sample, the ε' decreases and ε'' increases when broccoli stalk is subjected to the osmotic dehydration pre-treatment, resulting in better conversion of microwave energy into heat.

Keywords: Dielectric properties, Osmotic dehydration, Broccoli stalk.

7.1 Introduction

Pre-treatment prior to drying is gaining interest in food production to tackle two main challenges: (i) To enhance the quality of the final product; and (ii) To reduce the energy consumption. One of the most commonly used pre-treatment is osmotic dehydration concept. When the food material is immersed in a hypertonic solution, the osmotic pressure would begin the mass transfer exchange. The two major mass transfer mechanisms to occur are the water outflow from the product (water loss) and the solute inflow from the solution to the product (solute gain) concurrently. This phenomenon can reduce the mass of the sample by 50% at relatively low expenditure of energy, as there is no phase change of the liquid.

Oftentimes, the pre-treatment based on osmotic dehydration is coupled with microwave drying for production of dried food products. The feasibility of microwave energy to provide the rapid energy dissipation throughout the material becomes an advantage to overcome the limitation of other slow drying processes and improves the quality of final product (Zhang et al., 2006). The success of this combined method is the faster drying time leading to enhanced quality retention of dried products which has been reported on various vegetables and fruits such as strawberry (Venkatachalapathy & Raghavan, 1999); mushroom (Torringa et al., 2001); mangosteen (Therdthai & Visalrakkij, 2012); and papaya (Nimmanpipug et al., 2013).

Dielectric properties are key factors to understand the interaction of microwaves with food materials. Dielectric properties of material are described by the complex permittivity in the following relationship:

$$\varepsilon^* = \varepsilon' - j\varepsilon'' \quad (7.1)$$

The real part, ε' is the dielectric constant that reflects the ability of material to store energy and it is directly related to the strength of polarization, while the imaginary part, ε'' is the dielectric loss factor that measures the energy absorbed from the applied field.

Many techniques have been introduced for measuring dielectric properties of material in microwave frequencies such as by transmission lines, cavity perturbation and open-ended coaxial probe (Venkatesh & Raghavan, 2005). The open-ended coaxial probe method has been found to be the most widely used technique for dielectric properties measurement for application in liquid food and semi-solid materials, including biological materials (Ahmed et al., 2007a; Ahmed et al., 2007b; Bansal et al., 2015; Dev et al., 2008; Jafari et al., 2015). This technique uses a coaxial probe with the open end in contact with the sample. During the measurement, a signal is sent from the source of electromagnetic waves (e.g. vector analyzer or impedance analyzer) of defined frequency and transmitted to the probe. Then it passes into the material-under-test, where it displaces the ions and charge carriers in the material and reflected back to the probe. The magnitude and phase change of the reflected wave are used to compute the dielectric properties of the material (Jian & Yifen, 2008). Dielectric properties measurement using an open-ended coaxial probe is becoming more convenient, easy to use and quick due to its commercial availability; also it has a large bandwidth, and requires little sample preparation (Marc Regier & Helmar Schubert, 2005). Furthermore, the accuracy of the measured dielectric properties is adequate for most dielectric heating research (Tang & Lau, 2002).

Dielectric properties of materials depend on frequency, temperature, moisture content and chemical composition. A change in the osmotic conditions during osmotic dehydration will cause a change in the dielectric properties of the food material and its ability to absorb electromagnetic energy. According to work done by Al-Harahsheh et al. (2009) tomato pomace subjected to osmotic pre-treatment before microwave drying resulted in modification of the dielectric properties of the product with decrease in ε' and increase in ε'' which eventually enhances the drying rate. Changrue et al. (2008b) identified that sucrose and salt were the dominant variables affecting the ε'' during osmotic dehydration of carrots. Recently, Koskiniemi et al. (2013) reported that citric acid has a minimal contributing effect on dielectric properties compared to salt during acidified process of vegetables.

Although the dielectric properties of food materials have been widely reported (Venkatesh & Raghavan, 2004; Wang et al., 2011), information on broccoli stalk has not previously been documented. Adding value to the broccoli stalks has a good potential to be explored for enhancing the food security and helps in achieving better economic return for broccoli industry. Earlier study suggested that combination of osmotic dehydration prior to microwave drying could improve the final quality of broccoli stalk slices. Therefore, the objectives of this study were: (1) To investigate the effects of osmotic dehydration processing conditions on the dielectric properties of broccoli stalk at 2.45 GHz; (2) To develop a predictive equation in order to establish the relationship between the osmotic operating variables and the dielectric properties of the osmotically dehydrated broccoli stalk; and (3) To evaluate the prospects of osmotic dehydration of broccoli stalk prior to microwave energy as finish drying based on the dissipation factor and penetration depth.

7.2 Materials and Methods

7.2.1 Materials

The broccoli (*Brassica oleracea* L. var. *italica*) was purchased from a local market and the stalk were separated from the florets. The stalk were then washed, peeled and sliced to a uniform shape with a thickness of 0.6 cm and a diameter of 2.3 cm. Moisture content of samples were determined by drying in a hot air oven at 105 °C (Thermo Scientific Model 6528, Rockford, IL, USA) until a constant mass was reached (AOAC 935.29) (AOAC, 2000). Sucrose (Redpath Sugars, Montreal, QC, Canada), used as an osmotic agent was purchased locally. The osmotic solutions were prepared by dissolving the sucrose in the proper amount of distilled water.

7.2.2 Osmotic Dehydration Procedure

The osmotic dehydration of broccoli stalks was conducted in the continuous flow dehydration system as shown in Figure 7.1. The main components of this closed loop system consist of two acrylic cylindrical column named as outer cylinder and inner cylinder, pump (Pentair SHURflo 8000, New Brighton, MN, USA), thermocouple, heating/cooling unit (Isotemp 1013S, Fisher Scientific Inc., Pittsburgh, PA, USA), sample holder and variable area flow meter (Cole Palmer Instrument Co. Vernon Hills, IL, USA). The information of this equipment system is detailed in Chapter 6. Sucrose solution of a known concentration (40, 50 and 60 °Brix) was poured into the outer cylinder. Once the solution reached the required temperature (30, 40 and 50 °C), it was pumped into the inner cylinder (contactor system) at a required velocity (1.5, 2.5 and 3.5 mm/s). Then, the samples were immersed inside the inner cylinder using a sample holder and kept for the required time period (60-180 minutes). Samples were then removed from the osmotic solution and rinsed immediately in flowing water, drained on a tissue paper to remove the surface moisture.

7.2.3 Dielectric Properties Measurement

The dielectric properties of osmotically dehydrated broccoli stalk slices were measured using an open-ended coaxial probe coupled with a computer controlled software automated network analyzer (Agilent Technologies, 8722ES, Palo Alto, CA, USA). Before the measurements, the testing probe was calibrated using the equipment's standard configuration: air, short block and water. To avoid interferences, careful considerations were taken while immersing the probe into the water to make sure that no air bubbles exist on the surface of the sensor. After calibration, the probe was then kept in contact with the samples during the measurement. The mean values of three replicates of broccoli samples measured at 2.45 GHz are reported in this study.

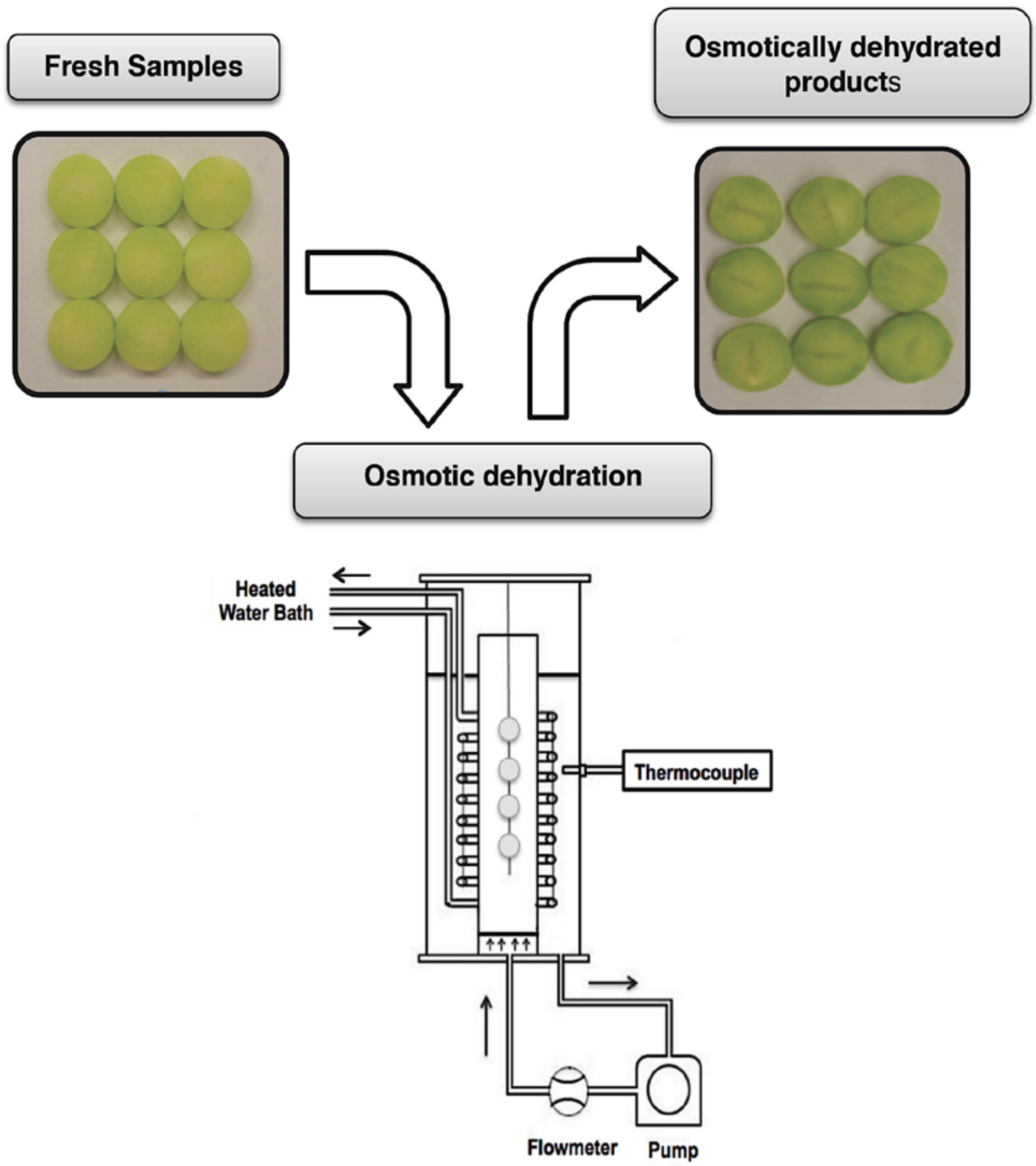


Figure 7.1: Flow diagram of osmotic dehydration of broccoli stalk slices under continuous flow osmotic dehydration system.

7.2.4 Determination of Dissipation Factor and Penetration Depth

After obtaining the dielectric properties, the dissipation factor and the penetration depth of microwaves at a frequency of 2.45 GHz into the osmotically dehydrated broccoli stalk slices can be calculated. Dissipation factor, also called as the loss tangent, is an indicator of material's ability to generate heat was calculated by the following equation:

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} \tag{7.2}$$

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Another important key factor that is associated with dielectric properties of food materials is the penetration depth. It is defined as the depth, where the dissipated power is reduced to 37% of the power entering the material surface. The d_p is expressed as follows (Ashim et al., 2014):

$$d_p = \frac{\lambda_0}{2\pi\sqrt{2\varepsilon'}}\left(\sqrt{1+\left(\frac{\varepsilon''}{\varepsilon'}\right)^2}-1\right)^{-\frac{1}{2}} \tag{7.3}$$

Where, λ_0 is wavelength in free space (for 2.45 GHz, λ_0 = 12.2 cm).

7.2.5 Experimental Design

The response surface methodology (JMP version 11 SAS Institute, Cary, NC, USA) was used to estimate the main effects of process variables on dielectric properties of osmotically dehydrated broccoli stalk slices. Temperature (X_1), solution concentration (X_2), flow velocity (X_3) and processing time (X_4) were selected as continuous independent variables based on the preliminary investigations. The face-centered central composite design was chosen due to limited experimental range. Additionally, this design uses only three levels of each factor (-1, 0 and +1), which helps in reducing the number of observations and lessened the experimental cost.

7.2.6 Development of Predictive Equation

The predictive equation was developed for each response by using stepwise regression (Draper et al., 1966). Analysis of variance (ANOVA) was performed to check the adequacy and accuracy of the fitted model and the coefficient of determination (R^2) was used to indicate how the model fits the variability of the results. The terms of second-order polynomial model consist of linear, quadratic and interaction terms as indicated in the following equation:

$$Y = b_0 + \sum_{i=1}^{k} b_i X_i + \sum_{i=1}^{k} b_{ii} X_i^2 + \sum_{i=1}^{k-1} \sum_{j=i+1}^{k} b_{ij} X_i X_j \tag{7.4}$$

where Y is the response (i.e. ε' and ε"); b_o, b_i, b_{ii}, b_{ij} are the regression coefficients for intercept, linear, quadratic and interaction terms; ; X_1, X_2, X_3 and X_4 are temperature (°C), solution concentration (°Brix), flow velocity (mm/s) and processing time (min), respectively.

7.3 Results and Discussion

7.3.1 Effect of Osmotic Dehydration Processing Variables on Dielectric Properties of Broccoli Stalk Slices

Table 7.1 presents the experimental data on ε' and ε" of different runs of osmotic dehydration conditions on broccoli stalk slices. The results obtained are in the range of 43.81 to 64.11 for ε' and 20.21 to 25.52 for ε" with a relative standard error less than 2%. The regression coefficients for the second-order polynomial equations are presented in Table 7.2. The results show that ε' was mainly influenced by the sucrose concentration and processing time, followed by temperature. Whereas, the linear effect of velocity was not significant (P > 5%) to ε', but its interaction effect with temperature was significant at the 5% level. Meanwhile, sucrose concentration showed linear, quadratic, and interaction effects on ε", indicating its larger influence on ε". Although the linear effect of processing time was not significant (P > 5%), but its interaction effects with temperature and sucrose concentration were significant at the 5% level.

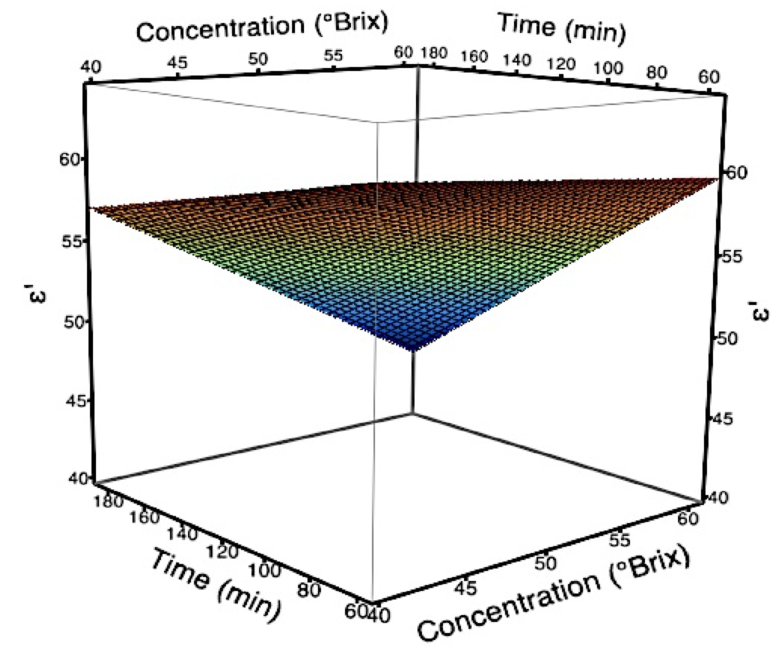
Table 7.1: Experimental conditions with values of response variables.

Run	Independent variables				Dependent variables	
	Temp. (°C)	Conc. (°Brix)	Velocity (mm/s)	Time (min)	ε'	ε''
1	30 (-1)	40 (-1)	1.5 (-1)	60 (-1)	61.48 ± 0.32	20.30 ± 0.37
2	30 (-1)	40 (-1)	1.5 (-1)	180 (+1)	58.30 ± 0.45	22.37 ± 0.21
3	30 (-1)	40 (-1)	3.5 (+1)	60 (-1)	62.81 ± 0.63	21.78 ± 0.03
4	30 (-1)	40 (-1)	3.5 (+1)	180 (+1)	57.90 ± 0.33	24.76 ± 0.24
5	30 (-1)	50 (0)	2.5 (0)	120 (0)	56.71 ± 0.35	24.94 ± 0.41
6	30 (-1)	60 (+1)	1.5 (-1)	60 (-1)	61.27 ± 1.12	24.48 ± 0.13
7	30 (-1)	60 (+1)	1.5 (-1)	180 (+1)	43.81 ± 0.56	24.55 ± 0.16
8	30 (-1)	60 (+1)	3.5 (+1)	60 (-1)	62.12 ± 0.76	24.71 ± 0.37
9	30 (-1)	60 (+1)	3.5 (+1)	180 (+1)	48.20 ± 0.78	25.38 ± 0.36
10	40 (0)	40 (-1)	2.5 (0)	120 (0)	57.19 ± 0.34	21.92 ± 0.08
11	40 (0)	50 (0)	1.5 (-1)	120 (0)	57.76 ± 0.65	23.89 ± 0.38
12	40 (0)	50 (0)	2.5 (0)	60 (-1)	60.04 ± 0.74	24.08 ± 0.18
13	40 (0)	50 (0)	2.5 (0)	120 (0)	54.60 ± 0.76	23.87 ± 0.13
14	40 (0)	50 (0)	2.5 (0)	120 (0)	55.44 ± 0.29	23.78 ± 0.28
15	40 (0)	50 (0)	2.5 (0)	120 (0)	55.91 ± 0.19	24.85 ± 0.02
16	40 (0)	50 (0)	2.5 (0)	180 (+1)	51.09 ± 0.42	24.50 ± 0.29
17	40 (0)	50 (0)	3.5 (-1)	120 (0)	54.52 ± 0.07	25.52 ± 0.46
18	40 (0)	60 (-1)	2.5 (0)	120 (0)	52.96 ± 0.05	23.76 ± 0.08
19	50 (+1)	40 (-1)	1.5 (-1)	60 (-1)	64.11 ± 0.79	20.21 ± 0.75
20	50 (+1)	40 (-1)	1.5 (-1)	180 (+1)	57.47 ± 0.28	21.06 ± 0.64
21	50 (+1)	40 (-1)	3.5 (+1)	60 (-1)	58.79 ± 1.13	23.78 ± 0.42
22	50 (+1)	40 (-1)	3.5 (+1)	180 (+1)	56.55 ± 0.49	22.65 ± 0.12
23	50 (+1)	50 (0)	2.5 (0)	120 (0)	51.55 ± 0.47	23.36 ± 0.27
24	50 (+1)	60 (+1)	1.5 (-1)	60 (-1)	57.73 ± 0.22	24.17 ± 0.32
25	50 (+1)	60 (+1)	1.5 (-1)	180 (+1)	46.16 ± 0.22	21.34 ± 0.27
26	50 (+1)	60 (+1)	3.5 (+1)	60 (-1)	55.67 ± 0.89	24.47 ± 0.20
27	50 (+1)	60 (+1)	3.5 (+1)	180 (+1)	44.99 ± 0.34	20.24 ± 0.38

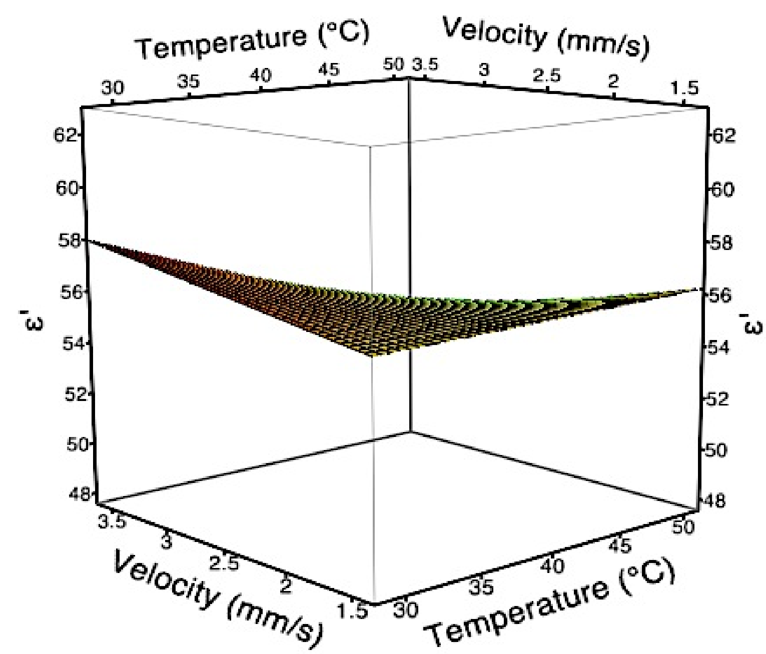
Table 7.2: Regression coefficients of the second-order polynomial model for dielectric constant, ε' and dielectric loss, ε".

	ε'		ε''	
Source	Coefficient	Prob > F	Coefficient	Prob > F
Model	55.095	<.0001	24.284	<.0001
Temperature (X_1)	-1.088	0.0082	-0.667	0.0003
Concentration (X_2)	-3.427	<.0001	0.794	<.0001
Velocity (X_3)	-0.364	0.3114	0.607	0.0005
Time (X_4)	-4.420	<.0001	-0.062	0.6491
Temperature*Concentration	-0.455	0.2361	-0.462	0.0058
Temperature*Velocity	-0.977	0.0201	-0.037	0.7923
Temperature*Time	0.522	0.1782	-0.820	<.0001
Concentration*Velocity	0.459	0.2327	-0.548	0.0018
Concentration*Time	-2.291	<.0001	-0.694	0.0003
Velocity*Time	0.444	0.247	-0.116	0.4179
Temperature*Temperature	-0.854	0.3667	-0.193	0.5846
Concentration*Concentration	0.088	0.9246	-1.503	0.0009
Velocity*Velocity	1.154	0.2291	0.360	0.3165
Time*Time	0.588	0.5304	-0.051	0.8839

The response surface plot illustrated in Figure 7.2 (a) shows variation in ε' with a function of sucrose concentration and immersion time. The ε' decreased rapidly with increase in sucrose concentration and processing time. Higher sucrose concentration possesses higher osmotic pressure. Thus, higher osmotic driving force between the sample and solution at longer processing time accelerate the water removal during the osmotic dehydration process (Md Salim et al., 2016b). Consequently, reduction in moisture content after the osmotic dehydration process implied the decrease in ε' of osmotically dehydrated broccoli stalk slices. The results are following the trends reported by Changrue et al. (2008b) on carrots.



(a)



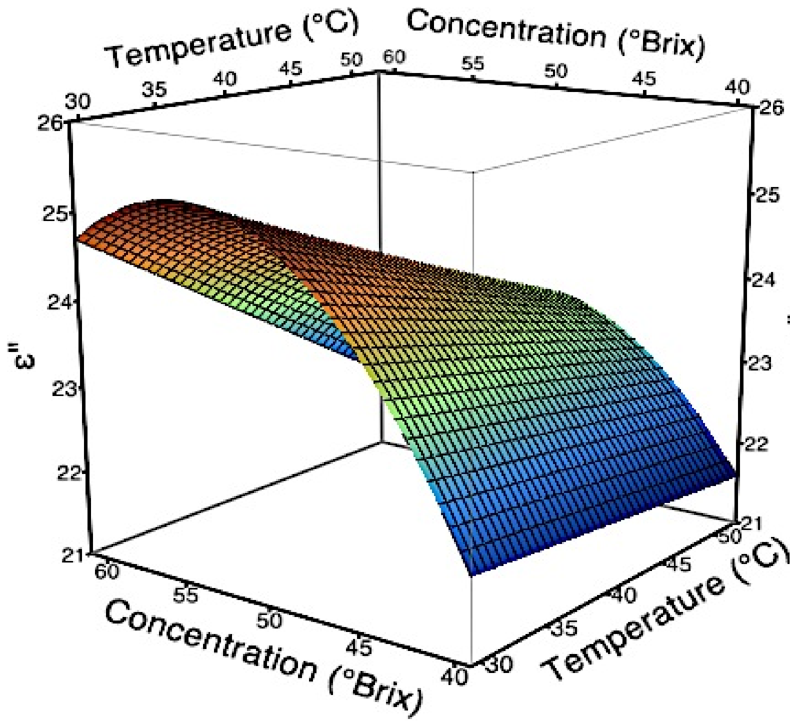
(b)

Figure 7.2: Dielectric constant, ε' variation as a function of (a) sucrose concentration and processing time; and (b) flow velocity and temperature.

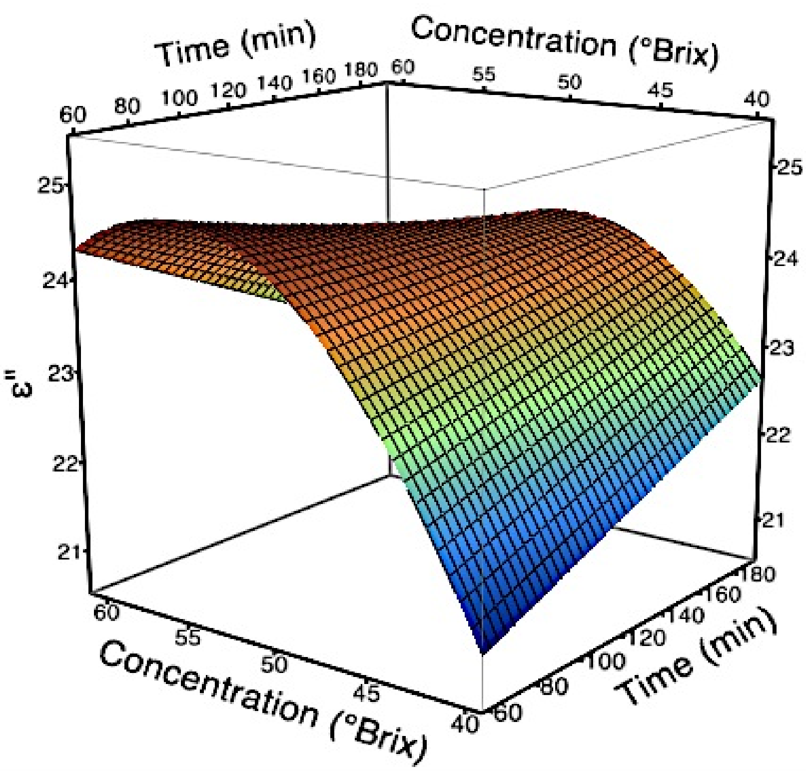
Furthermore, Figure 7.2 (b) demonstrated the influence of temperature and flow velocity on ε' and the result shows that high temperature along with the influence of higher velocity results in lower ε'. This is probably due to the renewal boundary layer through agitation facilitates the water removal process (Moreira & Sereno, 2003). Moreover, higher temperature might increase the cell membrane permeability to sucrose molecules, which is alluded to the membrane swelling effect. Thus, sucrose binds with the water molecules inside the sample and thus reducing the amount of free water, which reflects the reduction of ε'. Hence, this finding supports the previous research that sucrose is a good additive to lower the ε' of food samples (Luan et al., 2015; Sakai et al., 2005).

Figure 7.3 (a) presents the response surface plot of variations in ε'' as function of sucrose concentration and temperature. The results reveal that ε'' first increases markedly with the increase in sucrose concentration and then decreases slightly towards the higher concentration of 60 °C. On the other hand, the effect of temperature on ε'' demonstrates that lower temperature seem to have higher ε''. According to Liao et al. (2003), the quadratic trends of ε'' on sugar solutions was observed due to the effect of temperature. Thus, the possible explanation of these findings might be that the solubility of sucrose increased at higher temperature, and thus, the osmotic solution becomes more dilute (less concentrated) and it reduces the driving force during the osmotic dehydration process. Subsequently, the mass transfer process becomes slower during the osmotic dehydration which signifies the decreases of ε'' at higher concentration when the temperature is also higher.

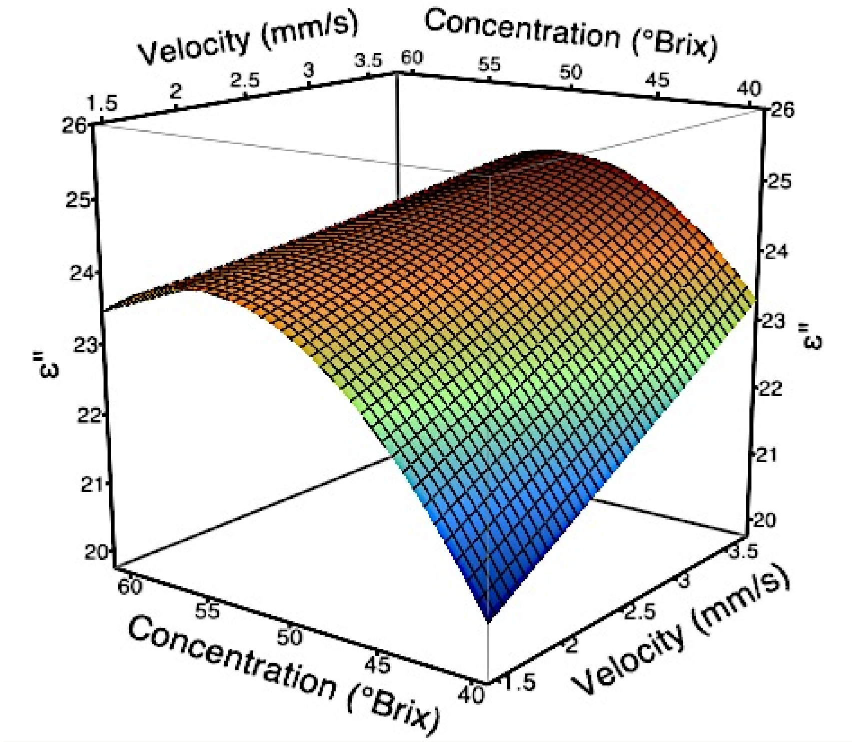
Figure 7.3 (b) presents the response surface plot of variations in ε'' as a function of sucrose concentration with processing time. The migration of sucrose molecules from the osmotic solution into the broccoli samples and the water removal from the samples to solution is a time-dependent process. Therefore, the longer the processing time, higher is the ε''. This dynamic mass transfer exchange affects the ε'' as reported by previous works on various vegetables (Koskiniemi et al., 2013).


Temperature (°C)
Concentration (°Brix)
ε''
ε''
Concentration (°Brix)
Temperature (°C)

(a)


Time (min)
Concentration (°Brix)
ε''
ε''
Concentration (°Brix)
Time (min)

(b)



(c)

Figure 7.3: Dielectric loss, ε'' variation as a function of (a) sucrose concentration and temperature; (b) sucrose concentration and processing time; and (c) and sucrose concentration and flow velocity.

Figure 7.3 (c) provides the intercorrelations of sucrose concentration and flow velocity to the variations in ε'' of osmotically dehydrated broccoli stalk slices. It can be seen that osmotic dehydration conducted at a higher flow velocity provides higher ε'' compared to lower flow velocity. This result may be explained by the fact that higher flow velocity tends to provide changes in hydrodynamic characteristics of external phase thus providing mass transfer resistance; causes disruption of the solute to diffuse into the food samples. Therefore, with the aid of higher flow agitation around the sample results in lower gained sucrose and hence, increases the ε'' of osmotically dehydrated broccoli stalk slices. The finding is in agreements with findings of Liao et al. (2003) which showed that ε'' decreased as the volume of sugar increased.

7.3.2 Predictive Equations for Dielectric Properties of Osmotically Dehydrated Broccoli Stalk Slices

In order to develop a predictive equation to elucidate ε' and ε'' as a functions of osmotic dehydration processing factors, some non-significant terms ($p > 0.05$) from Table 7.2 were eliminated and the resulting equations were tested for adequancy and fitness by analysis of variance (ANOVA) using JMP software. Table 7.3 summarises the results for ε' and ε''. The mathematical expression for ε' and ε'' are shown in Equation 7.5 and Equation 7.6, respectively. The model was found to be significant ($p < 0.05$) and the lack of fit was not significant ($p > 0.05$) with satisfactory values of R^2. The plots of predicted versus of experimental data for ε' and ε'' are shown in Figure 7.4 and Figure 7.5, respectively.

$$\varepsilon' = 55.746 - 1.088\boldsymbol{X_1} - 3.427\boldsymbol{X}_2 - 0.364\boldsymbol{X}_3 - 0.442\boldsymbol{X}_4 - 0.977\boldsymbol{X_1}\boldsymbol{X}_3 - 2.291X_2X_4 \tag{7.5}$$

$$\varepsilon'' = 24.309 - 0.667\boldsymbol{X_1} + 0.794\boldsymbol{X}_2 - 0.607\boldsymbol{X}_3 + 0.062\boldsymbol{X}_4 - 0.4619\boldsymbol{X_1}\boldsymbol{X}_2 - 0.820\boldsymbol{X_1}\boldsymbol{X}_4 - 0.548\boldsymbol{X_2}\boldsymbol{X}_3 - 0.684\boldsymbol{X}_2\boldsymbol{X}_4 - 1.426\boldsymbol{X_2}{}^2 \tag{7.6}$$

where;

$X_1 = \dfrac{(Temperature - 40)}{10}$; 30 °C ≤ $Temperature$ ≤ 50 °C

$X_2 = \dfrac{(Concentration - 50)}{10}$; 40 °Brix ≤ $Concentration$ ≤ 60 °Brix

$X_3 = Velocity - 2.5$; 1.5 mm/s ≤ $Velocity$ ≤ 3.5 mm/s

$X_4 = \dfrac{(Time - 120)}{60}$; 60 min ≤ $Time$ ≤ 180 min

Table 7.3: Analysis of variance (ANOVA) for dielectric constant, ε' and dielectric loss, ε''.

	ε'				ε''			
Source	d.f.	SS	MS	F	d.f.	SS	MS	F
Model	6	686.117	114.353	47.241*	9	64.941	7.216	28.891*
Error	20	48.413	2.421		17	4.246	0.250	
C. Total	26	734.530			26	69.187		
Lack of fit	18	47.531	2.641	5.990 (n.s.)	15	3.546	0.236	0.675 (n.s.)
Pure error	2	0.882	0.441		2	0.700	0.350	
Total error	20	48.413			17	4.246		
R^2	0.93				0.94			

d.f. = degree of freedom; MS = mean square ; n.s. = non-significant ($p > 0.05$)

* Significant at 5% level.

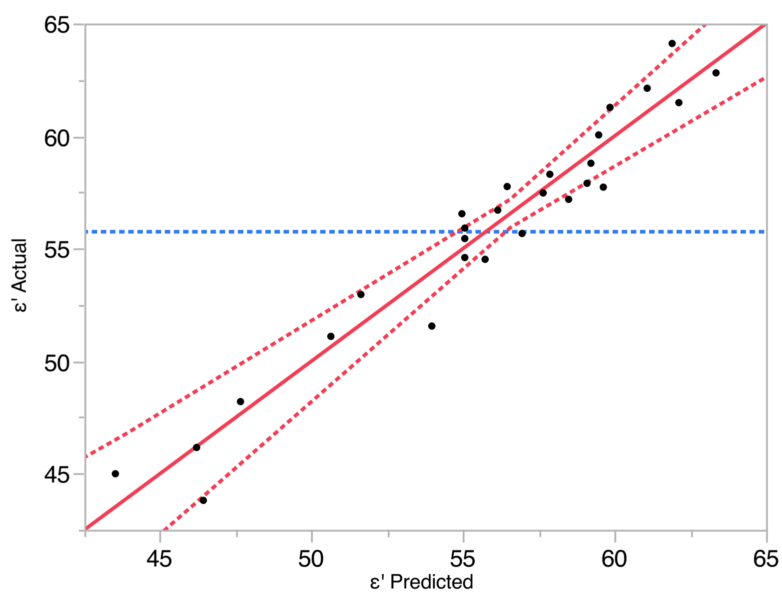


Figure 7.4: Predicted versus experimental values for dielectric constant, ε'.

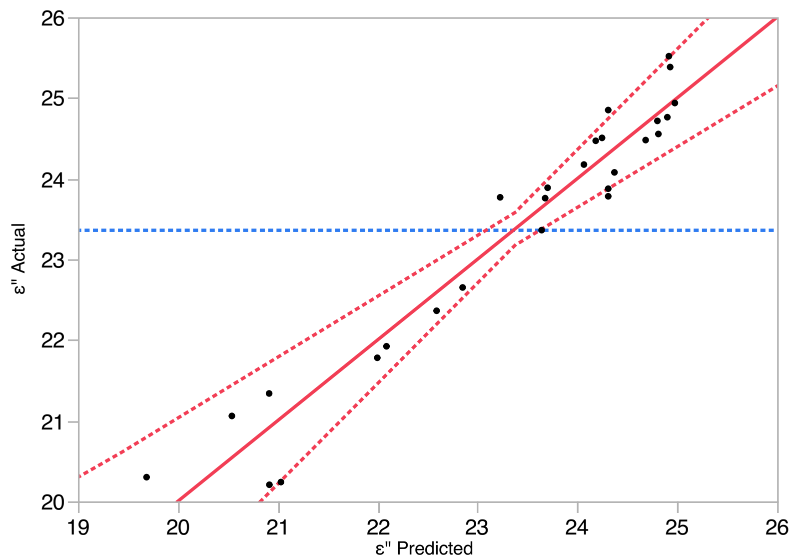


Figure 7.5: Predicted versus experimental values for dielectric loss, ε''.

7.3.3 Dielectric Properties, Dissipation Factor and Penetration Depth for Optimum Conditions of Osmotic Dehydration

The fresh broccoli stalk at moisture content of 94% (w.b.) possess a ε' and ε'' value of 71.30 and 20.10, respectively. In earlier investigation on mass transfer exchange during osmotic dehydration of broccoli stalk slices (Md Salim et al., 2016a), it was found that the conditions required to achieve higher water loss, 65 g / 100 g fresh sample with minimal solute gain, 3.9 g /100 g fresh sample are: a temperature of 30 °C with a sucrose concentration of 54 °Brix at a flow velocity of 3.5 mm/s for 120 min of immersion time. To classify the dielectric properties at the obtained conditions, prediction profiler function illustrated in Figure 7.6 was used in the JMP statistical software.

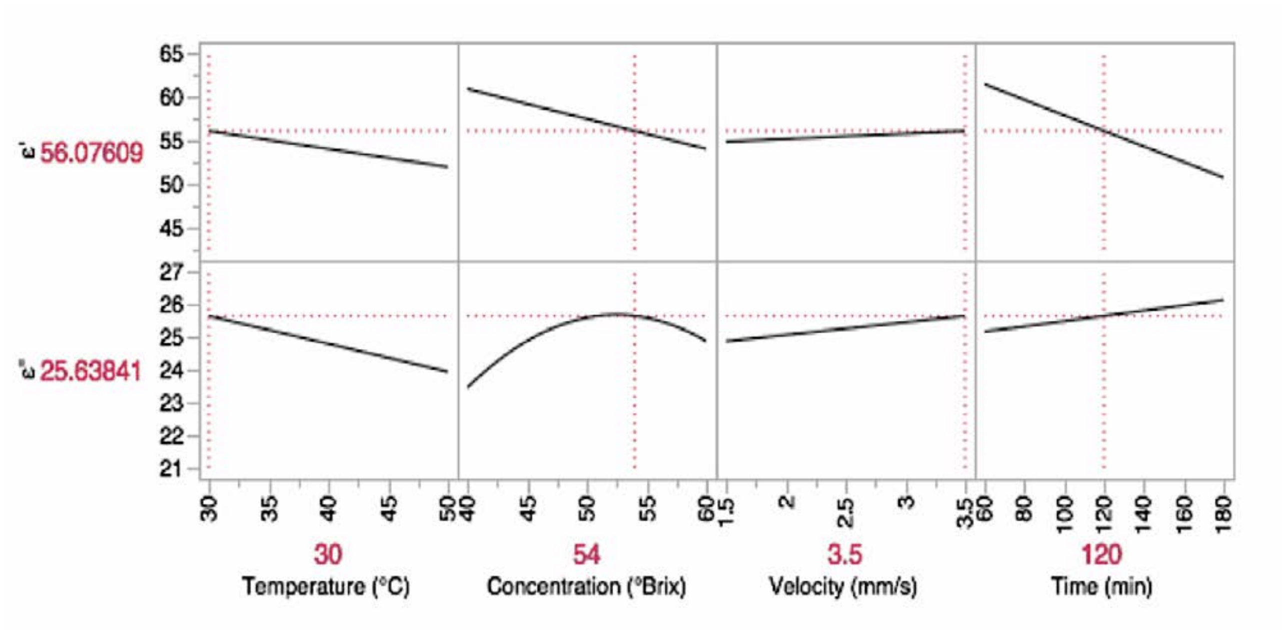


Figure 7.6: Prediction profiler for dielectric constant, ε' and dielectric loss, ε".

The results show that the value of ε' and ε" were found to be 56.08 and 25.64, respectively. The decrease in ε' indicates that microwaves are less reflected and coupled more when falling on the osmo-pretreated broccoli stalk compared to the fresh samples. While, higher ε" of osmo-pretreated broccoli stalk was observed in comparison with fresh samples implies that higher heating rate or higher absorption of microwaves can be achieved for further microwave drying.

Other parameters that help in understanding the dielectric properties are dissipation factor ($\tan\delta$) and penetration depth (d_p). In the current study, the computed $\tan\delta$ of osmotically dehydrated broccoli stalk slices was found to be 0.46, which is increased by 62% compared to fresh sample. Higher $\tan\delta$ describes that the energy is more quickly absorbed and dissipated as microwaves pass into the broccoli stalk slices. The increase in $\tan\delta$ of osmo-pretreated sample in comparison with fresh sample coincided with those reported by Therdthai and Visalrakkij (2012). Meanwhile, d_p for fresh and osmotically dehydrated broccoli stalk slices was calculated as 0.82 and 0.58 cm, respectively. The decrease in d_p of the osmo-pretreated sample describes that microwave energy can be totally absorbed within the sample which advantageous for further microwave drying. With these results, it is interesting to note that microwave heating can be very efficient for osmo-pretreated broccoli stalk slices.

7.4 Conclusion

Dielectric properties are essential parameters in predicting the material's performance in microwave environment. Basically, the properties of broccoli stalk slices are modified by moisture loss and the solute uptake during osmotic dehydration. The results of this study provide information that both, ε' and ε'' of broccoli stalk were dependent on the osmotic dehydration processing factors. Thus, the predictive equation were developed to obtain the ε' and ε'' for osmotically dehydrated broccoli stalk slices as functions of all the processing factors. These equations could be used to stipulate the desired heating pattern in microwave drying. In addition, the decrease of ε' and increase in ε'' explained the correlation of dielectric properties with higher water removal rate with minimal solute uptake during osmotic dehydration. Furthermore, higher $\tan\delta$ and lower d_p obtained after pre-treatment showed that microwave drying at 2.45 GHz could be applied for further drying of broccoli stalk slices efficiently.

CONNECTING STATEMENT FOR CHAPTER 8

Knowledge on dielectric properties provided in **Chapter 7** is required for better understanding on the microwave interaction with the sample for further drying. During osmotic dehydration and drying processes, the physical, bioactive compounds and nutritional quality might be degraded due to presence of water, oxygen, or heat. Therefore, in this following chapter, the physical, bioactive components and nutritional changes as affected by the processing during the development of osmo-dried broccoli stalk slices were determined.

CHAPTER 8

OSMO-DRIED BROCCOLI STALK SLICES: EFFECTS OF PROCESSING ON VITAMIN C, TOTAL CHLOROPHYLL, TOTAL PHENOLIC CONTENT, COLOR AND TEXTURE

Abstract

In this study, the broccoli stalk was converted into dried product by using the osmotic dehydration as pre-treatment followed by microwave-assisted hot air drying as finish drying. The influences of these processing steps on the product quality such as vitamin C, total chlorophyll, total phenolic content, color and texture of broccoli stalk slices were investigated. It has been demonstrated that when compared to fresh sample, osmotic dehydration pre-treatment resulted in a significant decrease ($p < 0.05$) in vitamin C content, chlorophyll content, and total phenolic content. In addition, the osmotically dehydrated product has minimal color changes and softer texture. While considering drying temperature as factor, better quality retention was observed when osmotically dehydrated broccoli stalk slices were dried at a drying temperature of 40 °C.

Keywords: Broccoli Stalk, Microwave-assisted drying, Osmotic dehydration, Drying kinetics, Product quality.

8.1 Introduction

In the last few years, market for food products has changed in the way consumers consume food. Consumers are now better informed and more concerned about nutrition and health implications on their chosen food products resulting in higher requirements for product quality. This trend is leading to an increased demand for nutritious food products from food industry. To some extent, consumers are seeking convenient food products due to their hectic lifestyle, which makes snack foods becoming a meal replacement. In respond to consumer demands, the food industry has begun offering a wide variety of healthy snack products such as dried vegetable chips. Instead of chips, dried vegetable can be used in a variety of ways; they are ideal for soup, seasoning mixes and they also can be used as an ingredient in instant foods.

Broccoli is a nutritious vegetable which has higher demand at the market place. In Canada alone, the production of broccoli is increased by 15.6% during the period of 2011 to 2014 (Statistics Canada, 2015). Broccoli is an excellent source of health promoting phytochemicals (Ares et al., 2013). It has conclusively been shown that consumption of broccoli can help reduce the risk for the development of certain forms of cancer (Finley, 2003; Latté et al., 2011). In current scenario, broccoli is consumed for its florets, leaving so many harvest residues such as the stalk and leaves (Bekhit et al., 2013).Therefore, the introduction of value added broccoli stalk as ready-to-eat products is an excellent idea not only for providing variety of convenience healthy foods to consumers but also to help in the better economic return for the producers in broccoli industry.

In recent years, osmotic dehydration has received considerable attention as pre-treatment step prior to the thermal process due to its energy saving advantage. In terms of quality, osmotic dehydration also offers an alternative way to improve the quality of dried products and potentially minimizing degradation of bioactive compounds from the thermal effects of drying process. It has been demonstrated by Torreggiani et al. (1995) that pigment retention of strawberry halves was higher when subjected to osmotic dehydration compared to untreated sample. Chauhan et al. (2011) reported that cellular structure of apple slices was retained in sucrose-treated samples. While, study done by Tonon et al. (2007) on osmotic dehydration of tomato shows that the carotenoid content were retained even after six hour of processing. Recently, Zhao et al. (2013) found that chili undergone osmotic dehydration process prior to drying resulted in higher retention of ascorbic acid and color.

For finish drying, there are many different drying techniques available nowadays as there is no one technique of drying that is applicable for all food products. Each food material has their own unique properties and hence the requirements for effective drying are varied. Microwave drying is one of the potential methods that have been implemented in food processing as it offers a fast volumetric heating and increases the drying rate. From economic point of view, stand-alone microwave drying is very costly. Hence, the combination of microwaves with other drying techniques is recommended (Chou & Chua, 2001; Orsat et al., 2007). One promising option is by using microwave in conjunction with hot air drying.

Microwave-assisted hot air drying is an emerging technique which combines advantages of volumetric heating and surface heating fastening the moisture removal during the drying process (Raghavan & Orsat, 2008). A study conducted by İzli et al. (2013) shows that microwave assisted hot air drying retain the phenolic content and antioxidant activity of goldenberry. During drying, air temperature has the decisive influence on product quality. Study conducted by Tello-Ireland et al. (2011) reported that alga dried at temperature of 50 °C showed increased change in color resulting from the presence of higher concentration of photosynthesis pigments (phycoerythin and phycocyanin) compared to the alga dried at 40 °C and 60 °C. While Oliveira et al. (2015) reported that in drying of kale, total phenolics, and vitamin C decreased with the increase of drying temperature. To our knowledge, no study has been performed to date on the impact of osmotic dehydration and microwave-assisted hot air drying on bioactive components of broccoli stalks.

Therefore, the aim of the present work is to investigate the effect of osmotic dehydration and microwave-assisted hot air drying at different air drying temperature on some of the quality parameters (vitamin C, chlorophyll content, total phenolic content, color and texture properties) during the development of osmo-dried broccoli stalk slices.

8.2 Materials and Methods

8.2.1 Chemicals and Reagents

Commercial sucrose (Redpath Sugars, Montreal, QC, Canada) was used as an osmotic agent. Trichloroacetic acid, Folin-Ciocalteu reagent, gallic acid, sodium carbonate (Na_2CO_3) were purchased from Sigma Aldrich Co. (St. Louis, MO, USA), ethanol, acetone and methanol were purchased from Fisher Scientific (Fair Lawn, NJ, USA).

8.2.2 Processing Overview of Dried Broccoli Stalk Slices

The processing step of dried broccoli stalk slices are summarized in Figure 8.1. The broccoli (*Brassica oleracea* L. var. *italica*) was purchased from a local market in Sainte Anne de Bellevue, QC, Canada. The stalks were separated from the florets and washed. Then, the stalks were peeled and sliced to a uniform shape with a thickness of 6 mm and a diameter of 23 mm. The moisture content of the broccoli slices was determined by drying to constant mass at 105 °C in a hot air oven (AOAC 935.29) (AOAC, 2000) and was found to be 94% (w.b.). The broccoli slices were subjected to osmotic dehydration as a pre-treatment process before being dried under microwave-assisted hot air dryer at different air drying temperatures until it reached moisture content of 10% (w.b.).

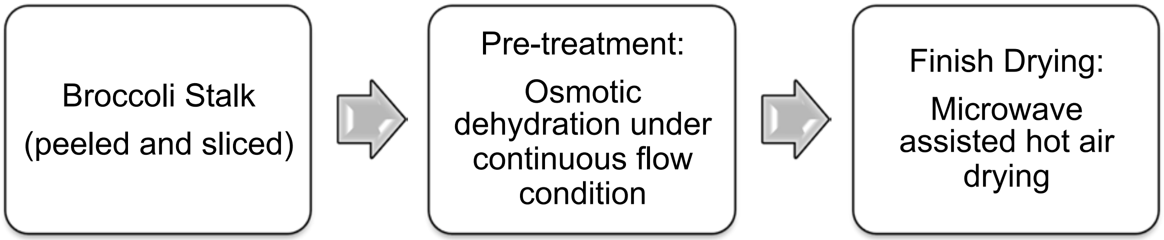


Figure 8.1: Flow diagram of processing of dried broccoli stalk snacks.

8.2.3 Osmotic Dehydration

The experiment on osmotic dehydration was conducted using newly developed laboratory scale continuous flow osmotic dehydration equipment illustrated in Figure 8.2. The outer cylinder of the system was filled with the sucrose solution at a concentration of 54 °Brix and was maintained at a temperature of 30 °C by using a heating/cooling unit (Isotemp 1013S, Fisher Scientific Inc.,Pittsburgh PA,USA). Then, the solution was pump to the inner cylinder at a velocity of 3.5 mm/s. After that, the samples were immersed inside the inner cylinder using the sample holder and kept within 120 minutes. Samples were then removed from the osmotic solution and rinsed immediately in flowing water, drained on a tissue paper to remove surface moisture. This given condition was the optimized processing condition evaluated from the previous experiment conducted with the aim of maximizing the water loss while minimizing the solute gain.

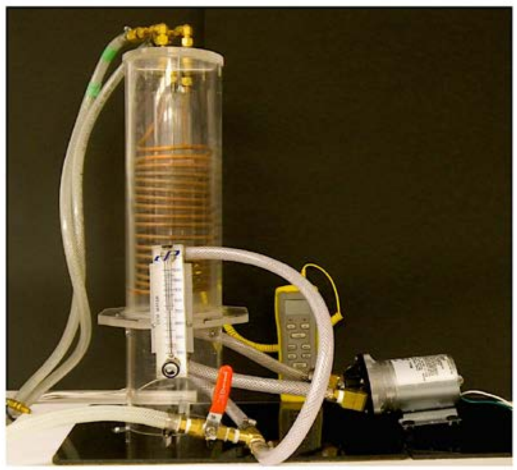
The water loss, solute gain and performance ratio of the osmotic dehydration were determined as follows (Hawkes & Flink, 1978) :

$$Water\ Loss\ (WL) = \frac{w_{wo} - (w_t - w_{st})}{w_{wo} + w_{so}} \text{ x } 100 \qquad (8.1)$$

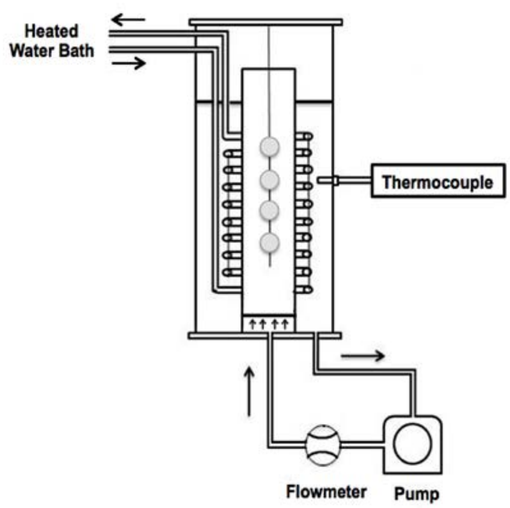
$$Solids\ Gain\ (SG) = \frac{w_{st} - w_{so}}{w_{wo} + w_{so}} \text{ x } 100 \qquad (8.2)$$

$$Performance\ Ratio\ = \frac{WL}{SG} \tag{8.3}$$

Where W_{wo} is the mass of water in the sample before dehydration (g), W_t is the mass of sample after dehydration (g), W_{so} is the mass of the solids in the sample before dehydration (g) and W_{st} is the mass of solids in the sample after dehydration (g).



(a)



(b)

Figure 8.2: (a) Continuous flow osmotic dehydration system; (b) Schematic diagram of osmotic dehydration system.

8.2.4 Microwave-assisted Hot Air Drying

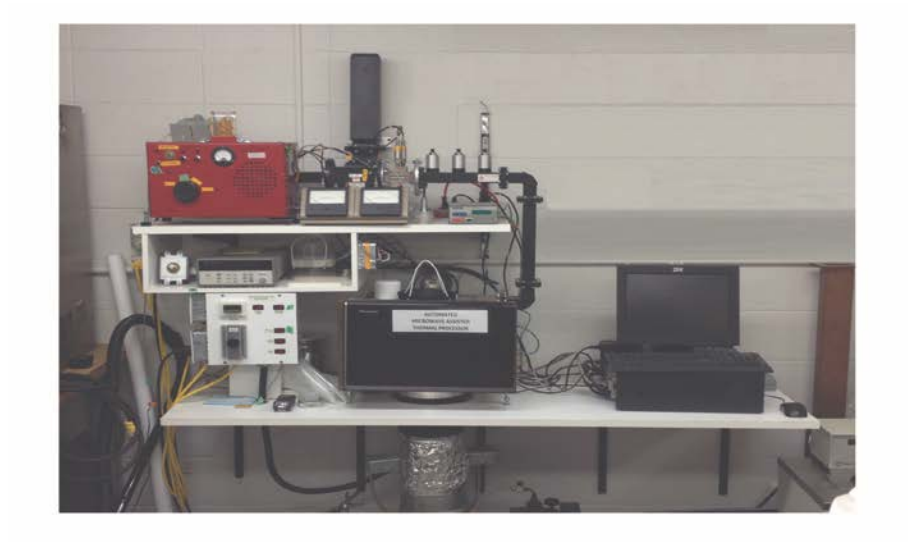
The final drying was carried out in an automated microwave-assisted laboratory scale dryer (Figure 8.3). The technical information on this custom built dryer is given by Dev et al. (2011). For each trial, about 50 g of osmotically dehydrated broccoli stalk slices were prepared. All the slices were put as a thin-layer in a sample holder and placed inside the microwave cavity. The power of the dryer was set to 100 Watts along with the convective drying air temperatures of 40, 50 and 60 °C and at the superficial air velocity of 1.4 m/s. The mass of the sample was recorded during drying process by the computer via data acquisition system (Hewlett-Packard, USA). The change of moisture in the osmotically dehydrated samples during drying was expressed as the moisture ratio:

$$\text{Moisture Ratio (MR)} = \frac{M_t - M_e}{M_o - M_e} \tag{8.4}$$

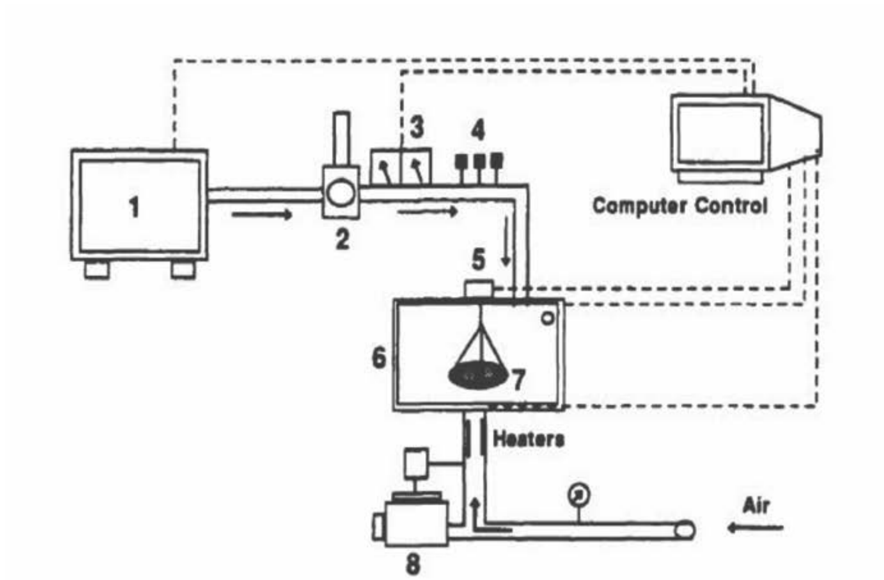
Where M_t is the moisture content at time t_i, M_o is the initial moisture content and M_e is the equilibrium moisture content. However, during the drying process, the M_e are relatively small to be quantified. So, in the current study, the M_e of broccoli stalk slices was considered as zero. The Page model (Equation 8.5) was used to model the drying kinetics. This model has been widely used to describe drying kinetics of various agricultural products (Dev et al., 2011; Therdthai & Visalrakkij, 2012).

$$\text{MR} = \exp(-kt^n) \tag{8.5}$$

Where, k is the drying rate constant; n is the regression coefficient; and t is the time (min).



(a)



(b)

Figure 8.3: (a) Automated microwave-assisted laboratory dryer; (b) Schematic diagram of microwave-assisted laboratory dryer (1: microwave generator, 2: circulator, 3: power meters, 4: tuning screws, 5: strain gauge, 6: microwave cavity, 7: sample holder and 8: blower).

The goodness of fit was determined using the coefficient of determination (R^2), chi-square (χ^2) and root mean square error ($RMSE$) as follows:

$$R^2 = 1 - \frac{\sum_{i-1}^{i=n}\left(MR_i^{exp} - MR_i^{pred}\right)^2}{\sum_{i=n}^{i=n}\left(MR_i^{exp} - \overline{MR}^{exp}\right)^2} \tag{8.6}$$

$$\chi^2 = \sum_{i-1}^{i=n} \frac{(MR_i^{pred} - MR_i^{exp})^2}{n-m} \tag{8.7}$$

$$RMSE = \sqrt{\frac{1}{n}\sum_{i=1}^{n}(MR_i^{pred} - MR_i^{exp})^2} \tag{8.8}$$

Where n is the number of observations, MR_i^{pred} is the *i*th predicted dimensionless MR, MR_i^{exp} is the *i*th experimental dimensionless MR, m is the number of variables and $\overline{MR}^{exp}$ is the mean of the experimental dimensionless MR.

8.2.5 Quality Parameters

8.2.5.1 Vitamin C

Vitamin C was determined by colorimetric method described by Jagota and Dani (1982). 0.25 g of fresh, pre-treated and dried samples were extracted in distilled water at 3000 rpm and then filtered. The filtered solution was diluted with distilled water up to 100 ml. 0.2 ml of this mixture was mixed with 0.2 ml of 10% trichloroacetic acid and was left in an ice bath for 5 min. About 0.2 ml of Folin-Ciocalteu reagent (previously diluted tenfold with distilled water) were added and the absorbance was measured using spectrophotometer (Ultrospec 2100pro, Amersham Biosciences, New Jersey, USA) at 760 nm. The vitamin C content was expressed as mg/g dry weight.

8.2.5.2 Chlorophyll Content

The total chlorophyll content in the fresh, pre-treated and dried broccoli stalk was determined using the procedure described by Topcu et al. (2015). A 0.25 g of sample was homogenized in 80% v/v acetone. The supernatant was collected and diluted to 25 ml with 80% v/v acetone. The absorbance readings were measured at 663 nm and 647 nm using spectrophotometer (Ultrospec 2100pro, Amersham Biosciences, New Jersey, USA). The results were used to calculate total chlorophyll content according to Lichtenthaler (1987) :

$$Total\ chlorophyll = 7.15A_{663} + 18.71A_{647} \tag{8.9}$$

where A is the absorbance.

8.2.5.3 Total Phenolic Content

The amount of total phenolic was determined using Folin-Ciocalteu reagent as described by Singleton and Rossi (1965) using the procedure described by Bakar et al. (2009). About 1 g of sample was extracted in 80% aqueous methanol (4.5 ml) on an orbital shaker set at 180 rpm for two hours. The mixture was centrifuged at 3000 rpm for 15 min and filtered through a Whatman No.1 filter paper. Each extract (0.3 ml) was mixed with 1.28 ml of Folin-Ciocalteu reagent (previously diluted tenfold with distilled water) and 0.8 ml of 7.5% sodium carbonate (NA_2CO_3). After three min, 1.6 ml of distilled water was added and the solution was incubated for 30 minutes at 40°C. The absorbance was analyzed at 760 nm using spectrophotometer (Ultrospec 2100pro, Amersham Biosciences, New Jersey, USA). The results were expressed as milligram of gallic acid equivalents (GAE) / g of dry weight.

8.2.5.4 Color

The color of fresh sample and osmoticallly dehydrated broccoli stalk before and after drying was measured using a tristimulus colorimeter (CR-310, Minolta Co. Ltd., and Japan). The measured parameters are L* (lightness), a* (green–red coordinate) and b* (blue–yellow coordinate). The total color changes were calculated using the Hunter-Scotfield equation (Equation 8.10); where $\Delta a^* = a^* - a^*_0$; $\Delta b^* = b^* - b^*_0$; and $\Delta L^* = L^* - L^*_0$, subscript 0 represents the fresh sample.

$$\Delta E = \sqrt{(\Delta a)^2 + (\Delta b)^2 + (\Delta L)^2} \tag{8.10}$$

8.2.5.5 Texture Analysis

The texture of fresh, pre-treated and dried samples was measured using Instron materials testing device (Instron 4502, Boston, USA) controlled by computer software (Instron series IX, version 8.25). The probe used was cylindrical with a puncture diameter of 3.7 mm and passed through a sample at a constant speed of 10 mm/min. The measured parameters for the texture analysis are the mean value of the maximum force and energy required to puncture the samples (Singh et al., 2013).

8.2.5.6 Statistical Analysis

All experiments were conducted in triplicate and the means were analyzed using analysis of variance (ANOVA). Duncan's multiple range test was used to identify significant difference ($p < 0.05$) among samples for each attribute.

8.3. Results and Discussion

8.3.1 Osmotic Dehydration

Aside from water removal, solute gaining will also occur concurrently during the mass transfer process. However, gaining excessive solute would hinder the mass transfer process during further drying. Thus, efficiency of this dehydration process is dependent on the performance ratio. Table 8.1 presents the results of water loss, solute gain and the performance ratio of broccoli stalk slices after osmotic dehydration process. From this table, it is clear that the average performance ratio of the samples dehydrated by osmotic system under continuous flow was higher compared to previous study reported by Jain et al. (2011), Alam et al. (2010) and Md Salim et al. (2016b) under static conditions. The moisture content of the broccoli stalk slices after osmotic dehydration was found to be 70% (w.b.). In earlier investigation (Md Salim et al., 2014), the drying time required to achieve moisture content of 70% in untreated broccoli stalk slices dried under hot air drying was about four hours when dried at an air temperature of 50 °C. Thus, the effect of reduced time during osmotic dehydration process showed that exchange of mass occurred at a faster rate within the initial 120 min, providing the advantage of processing time reduction.

Table 8.1: Mass transfer exchange of osmotically dehydrated broccoli stalk.

Sample	WL (%)	SG (%)	Performance Ratio
Broccoli stalk slices	65.8 ± 1.9	3.8 ± 0.2	17.3 ± 1.3

8.3.2 Drying Characteristics of Osmotically Dehydrated Broccoli Stalk Slices under Microwave assisted Hot Air Drying

Figure 8.4 shows the moisture content versus drying time for microwave-assisted hot air drying of osmotically dehydrated broccoli stalk slices as influenced by drying air temperature of 40, 50 and 60 °C. In this figure, there is a clear trend that drying temperature influences the drying time to reach the desired moisture content. Drying times at 40 °C was 210 min, longer than that of 50 and 60 °C. Thus, as the temperature increased, lesser drying time was resulted. This finding is consistent with the findings of past studies (Dev et al., 2011; Singh et al., 2013; Workneh et al., 2011).

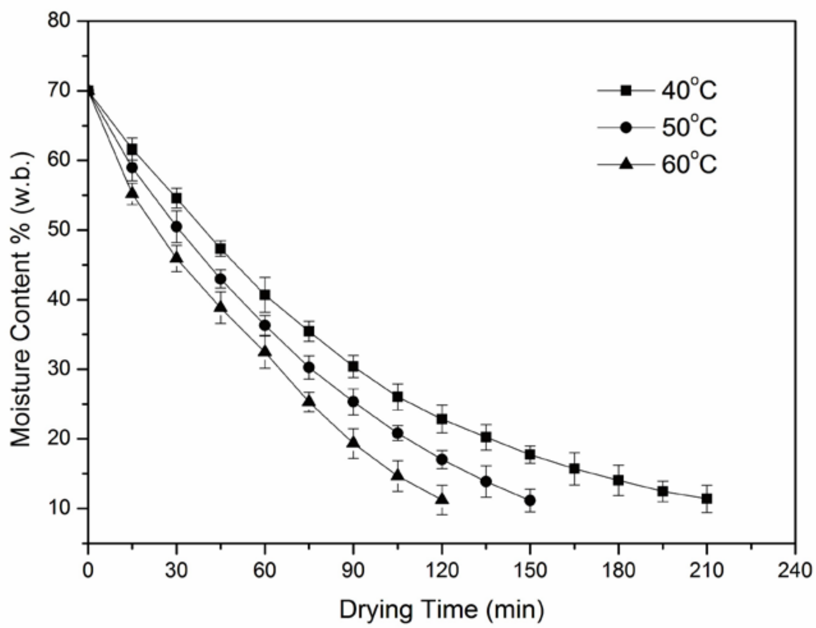


Figure 8.4: Characteristic drying curves of osmotically dehydrated broccoli stalk slices at temperatures of 40, 50 and 60 °C.

Page model used to stimulate the drying kinetics at different drying temperatures is presented in Figure 8.5. The parameters *k* and *n* and the regression analysis are shown in Table 8.2 for a given drying condition. The model gave an excellent fit for all the experimental data with R^2 of 0.99, χ^2 and *RMSE* lower than 4.39 x 10^{-4} and 2.10 x 10^{-2}, respectively. As can be seen from the table, the value of drying constant *k* parameter increased with the increase in drying temperature. This result signifies that higher temperature provides faster drying of the broccoli stalk slices.

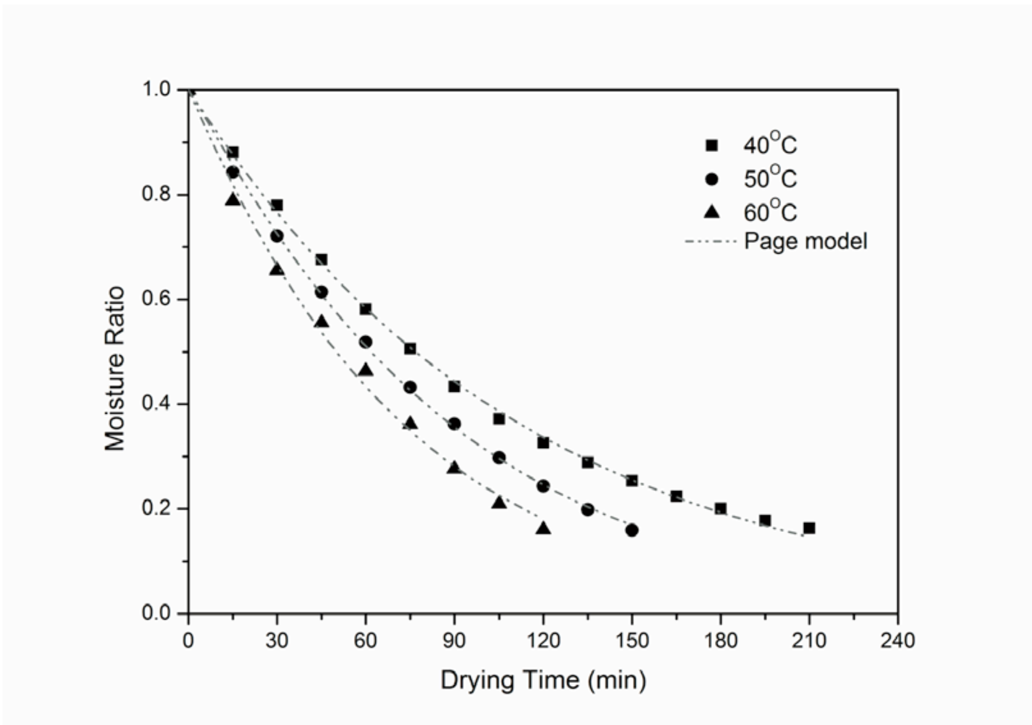


Figure 8.5: Comparison of the experimental and predicted moisture ratio obtained using the Page model for osmotically dehydrated broccoli stalk slices at temperatures of 40, 50 and 60 °C.

Table 8.2: Effect of drying temperature on Page model regression parameters.

Drying temperature	Parameters				
	k	*n*	χ^2	*RMSE*	R^2
40 °C	8.44 x 10^{-3}	1.02	8.68 x 10^{-5}	9.32 x 10^{-3}	0.99
50 °C	8.64 x 10^{-3}	1.06	5.75 x 10^{-5}	7.58 x 10^{-3}	0.99
60 °C	1.21 x 10^{-2}	1.03	4.39 x 10^{-4}	2.10 x 10^{-2}	0.99

8.3.3 Quality Evaluation

8.3.3.1 Vitamin C

The vitamin C content of fresh broccoli stalks, osmotically dehydrated broccoli stalks and that of osmo-pretreated broccoli stalks dried by the three different drying temperatures under microwave-assisted hot air drying are shown in Figure 8.6. The initial content of the fresh broccoli stalk was 4 mg/g dry weight, which is equivalent to 24 mg/100 g fresh weight. This value is within the range reported by Singh et al. (2011) on broccoli stalk (25 ± 2.79 mg/100g fresh weight).

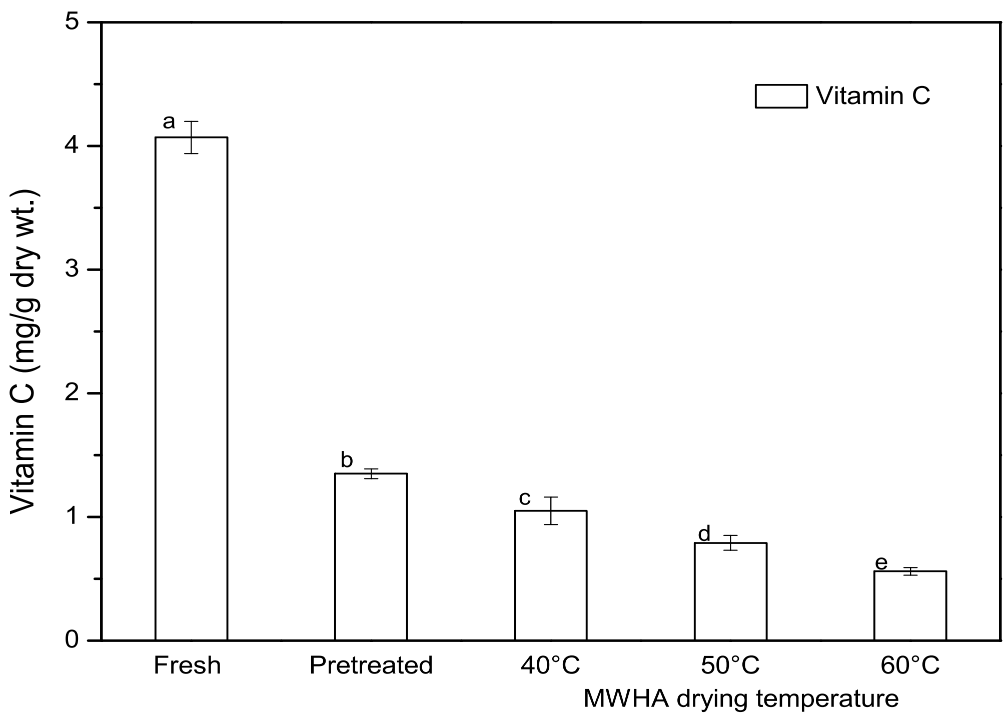


Figure 8.6: Vitamin C value of fresh, osmotically dehydrated and osmo-dried broccoli stalk slices at different microwave-assisted hot air (MWHA) drying temperatures. (Values with the same letter are not significantly different).

The effect of processing of osmo-dried products resulted in less vitamin C compared to the fresh sample. It can be seen that the vitamin C content dropped 67% after two hours immersion time during osmotic dehydration process. The losses of this water-soluble vitamin could be explained due to diffusion in the osmotic solution and chemical degradation during the process (Welti-Chanes et al., 2000). Heng et al. (1990) reported the losses of vitamin C from papaya during osmotic dehydration up to 80% after two hours processing time. Further, the results show that vitamin C was negatively correlated with drying temperatures under microwave-assisted hot air drying. At higher temperatures, vitamin C content significantly decreased which could be due to oxidation and thermal degradation (Hawlader et al., 2006). This trend has been reported by Xiao et al. (2010) and Kaya et al. (2010). Thus, drying of osmotically dehydrated broccoli stalk slices at drying temperature of 40 °C provide higher retention of vitamin C.

8.3.3.2 Total Chlorophyll Content

The amount of total chlorophyll assessed in the fresh sample was 0.96 mg/g dry weight, which is attributed to 34% less than the reported value by Sharma et al. (2011) for the broccoli florets (1.45 mg/g dry weight). Figure 8.7 shows the total chlorophyll content as affected by the processing for the development of osmo-dried broccoli stalk slices. From the figure, it is observed that osmotic dehydration pre-treatment prior to drying process contribute to the maximum loss in the total chlorophyll content with 55%. This highest loss is believed to be due to the losses of the magnesium atom of chlorophyll by the organic acids leaching from the cell into the osmotic solution during the immersion time (Vieira, 1996). Further, these findings also found that the drying temperature had a detrimental effect on the retention of chlorophyll content. This trend is in line with the previous study reported by Guan et al. (2005) and Ahmed et al. (2001) that chlorophyll are susceptible to thermal treatment attributable to conversion of chlorophyll to pheophytin. Thus, drying temperature of 40 °C gave the highest retention of chlorophyll content for osmo-dried broccoli stalk products.

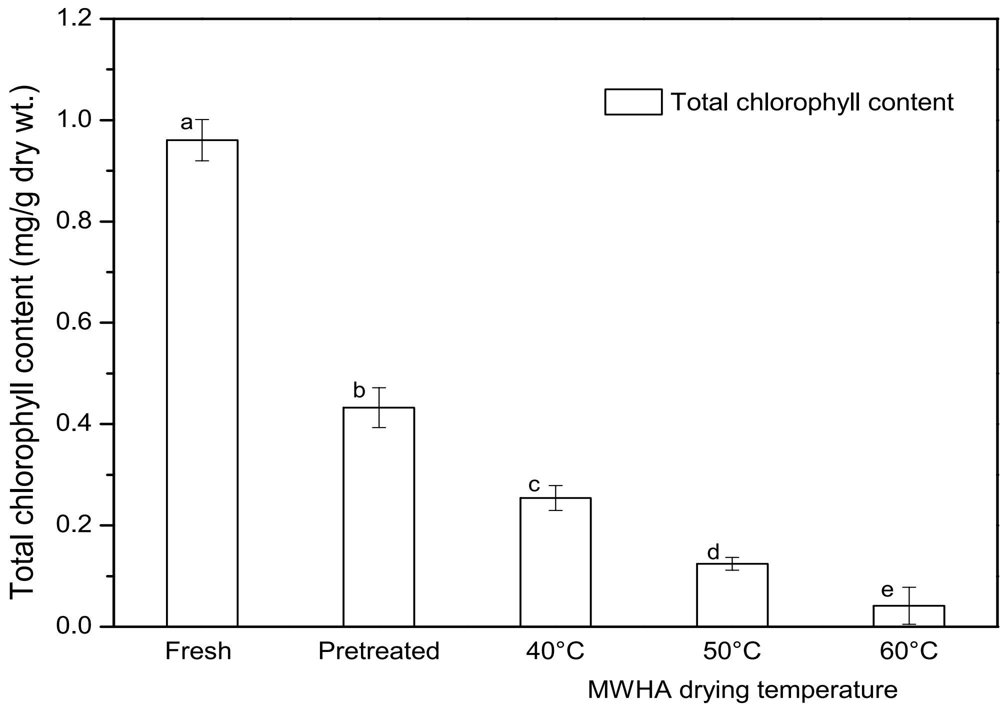


Figure 8.7: Total chlorophyll content of fresh, osmotically dehydrated and osmo-dried broccoli stalk slices at different microwave-assisted hot air (MWHA) drying temperatures. (Values with the same letter are not significantly different).

8.3.3.3 Total Phenolic Content

The total phenolic in the fresh broccoli stalks, osmo-pretreated and dried samples are presented in Figure 8.8. The results indicated a decreasing tendency between the total phenolic and the step of processing. The initial total phenolic content of broccoli stalk was 1.8 mg GAE/g dry weight. This value decreased by 44% after osmotic dehydration pre-treatment process. The losses of phenolic content during osmotic dehydration might be due to migration to osmotic solution. This event has been reported by Kucner et al. (2013) on blueberries where the phenolic content in fruits and syrups before and after osmotic dehydration process was balanced. It was also observed that the drying process led to a notable reduction of phenolic content in the range of 66.5% to 75.4% that contained in fresh sample. In fact, osmotically dehydrated broccoli stalk dried at temperature of 60 °C showed the lowest retention of phenolic content compared to other drying temperatures. Previous study reported that drying at lower temperature associated with longer drying time, resulting in lower retention of phenolic content (López et al., 2010). However, in this study, we found that longer drying time did not necessarily contributed to lower phenolic content. Instead, the temperature rise plays a key role in the retention of phenolic content in the osmo-dried broccoli stalk. The degradation of phenolic content during drying might be due to the binding of polyphenols with other compounds such as proteins or the alterations in the chemical structure of polyphenols that cannot be extracted and determined by available methods (Martín-Cabrejas et al., 2009; Oliveira et al., 2015).

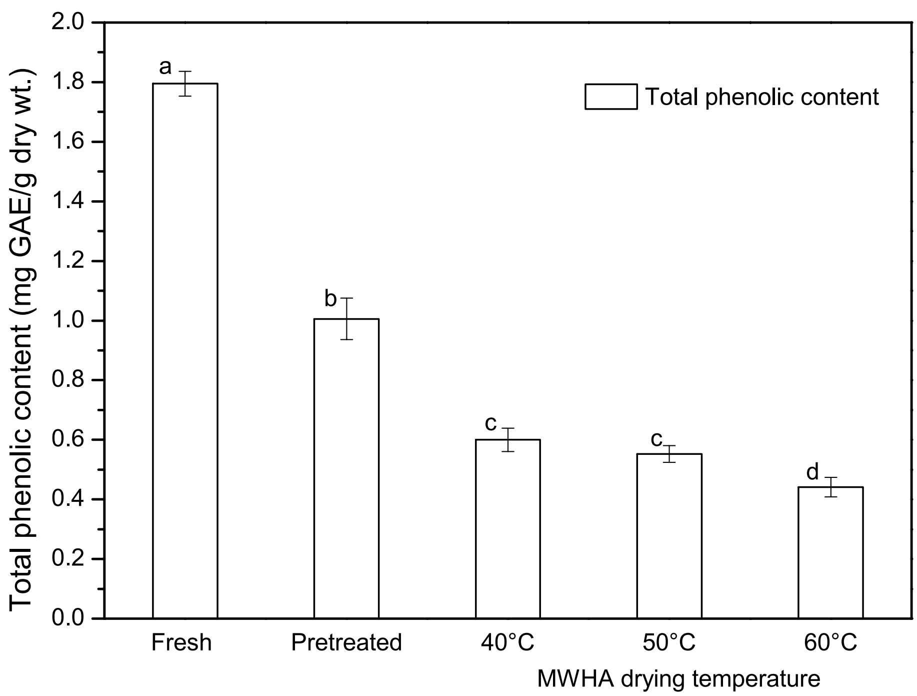


Figure 8.8: Total phenolic content of fresh, osmotically dehydrated and osmo-dried broccoli stalk slices at different microwave-assisted hot air (MWHA) drying temperatures. (Values with the same letter are not significantly different).

8.3.3.4 Color

The average lightness (L^*), greenness ($-a^*$) and yellowness ($+b^*$) color properties obtained for fresh, osmo-pretreated and dried broccoli stalk slices under drying temperatures of 40, 50 and 60 °C are presented in Table 8.3. It can be observed that the value of L^* and a^* were decreased while value of b^* was increased after osmotic dehydration process. These results indicate that immersion of samples in osmotic solution for 120 min results in the reduction of the lightness, increased the degree of greenness and increased slightly in yellowness. For the dehydrated samples, the L^* presented the highest at lower temperature. However, the value of a^* for all dehydrated samples showed that the green color was not significantly ($p > 0.05$) affected by drying temperatures. On the contrary, the b^* values showed an increasing tendency when drying temperature increased. The increased values of yellowness could be due to the result of generation of brown products due to nonenzymatic browning reactions occurring during drying.

When a sample with sucrose contents are involved in heat-process, a number of chemical reactions occur, one of which is well-known Millard reaction. Millard reaction is mainly responsible for browning development in osmo-pretreated broccoli stalk slices during drying. The Millard reaction involves the reaction of an amine (usually a protein or amino acid) and an aldehyde (usually a reducing sugar) and is highly temperature-dependent (Chua et al., 2000). During drying process, the sucrose can be hydrolyzed into reducing sugars (fructose and glucose) that can later be involved in Millard reaction (Phisut et al., 2013). Since total color difference, ΔE is a function of the three L^*, a^* and b^* coordinates, the results showed that the changes were associated with processing. Rising drying temperature resulted in a significant ($p < 0.05$) increase in total color changes. These results reveal that temperature plays a crucial role on color parameters when drying the osmotically dehydrated broccoli stalk slices; higher temperature results in sample discoloration. Similar results were reported by Vega-Gálvez et al. (2009) and Oliveira et al. (2015). The photograph image of the color changes for each step of processing is presented in Figure 8.9.

Table 8.3: Color parameters of fresh, osmotically dehydrated and dried broccoli stalk slices.

System	L*	a*	b*	ΔE
Fresh sample	71.10 ± 2.79 [ab]	-10.43 ± 0.2 [a]	24.65 ± 0.31 [e]	-
Osmo-pretreated	67.24 ± 1.41 [c]	-12.15 ± 0.23 [b]	29.71 ± 0.64 [d]	6.64 ± 1.3 [a]
Osmo-MWHA 40°C	73.16 ± 0.73 [a]	-12.66 ± 0.01 [b]	34.25 ± 2.30 [c]	10.13 ± 2.02 [b]
Osmo-MWHA 50°C	69.28 ± 1.02 [bc]	-13.02 ± 0.74 [b]	38.63 ± 1.2 [b]	14.37 ± 1.14 [c]
Osmo-MWHA 60°C	68.06 ± 1.87 [bc]	13.26 ± 1.83 [b]	41.62 ± 1.39 [a]	17.58 ± 1.77 [d]

(Column–wise values followed by the same letter are not significantly different).

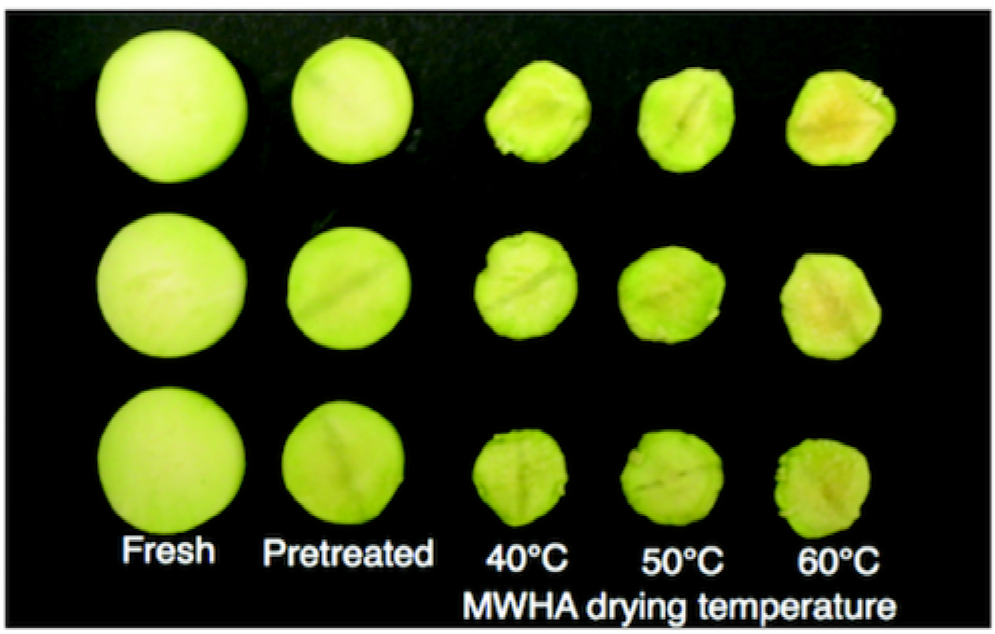


Figure 8.9: Photograph of fresh, osmotically dehydrated and osmo-dried broccoli stalk slices at different microwave-assisted hot air (MWHA) drying temperatures.

8.3.3.5 Texture Analysis

Table 8.4 shows the textural parameters for fresh broccoli stalk, osmo-pretreated and dried samples. The maximum force applied to puncture the sample is commonly associated with the texture firmness (Tello-Ireland et al., 2011; Vega-Gálvez et al., 2011). It can be observed that, lower force and energy were required to puncture the osmotically dehydrated broccoli stalk compared to the fresh samples. These findings could be related to cell membrane disruption that occurred during the dehydration process indicating loss of firmness. From the table, it can be seen that the dried products have a different sensory characteristic from the fresh sample. There are no significant difference ($p > 0.05$) observed on textural property between 40 °C and 50 °C. However, higher force and energy are required to crush the sample dried at 60 °C. This could be due to the use of higher temperature, which provoked changes that made a more rigid structure (Krokida et al., 2000).

Table 8.4: Force and energy required to puncture the fresh, osmotically dehydrated and osmo-dried broccoli stalk slices

System	Force (N)	Energy (J)
Fresh sample	21.163 ^{d} ± 0.931	0.062 ^{a} ± 0.004
Osmo-pretreated	13.617 ^{c} ± 1.139	0.022 ^{d} ± 0.001
Osmo-MWHA 40°C	30.890 ^{b} ± 2.018	0.041 ^{c} ± 0.007
Osmo-MWHA 50°C	31.630 ^{b} ± 1.381	0.041 ^{c} ± 0.003
Osmo-MWHA 60°C	34.430 ^{d} ± 0.691	0.051 ^{b} ± 0.004

(Column–wise values followed by the same letter are not significantly different).

8.4. Conclusion

The application of osmotic dehydration and microwave-assisted hot air drying on drying kinetics and product quality of broccoli stalk slices was investigated in this study. It is observed that during osmotic dehydration, nutrient losses were higher expected to leaching into the osmotic solution. Thereby, adding value to osmotic syrup is essential to prevent losses of valuable natural substances after the osmotic dehydration process. The drying kinetics showed that drying time was faster when drying temperature increased. However, when the drying temperature increased from 40 °C to 60 °C, physical and nutritional quality were degraded more extensively at higher temperature. Thereby, in the development of osmo-dried broccoli stalk product, higher drying temperatures should not be adopted for preserving vitamin C, chlorophyll content, phenolic compound, color and texture.

CONNECTING STATEMENT FOR CHAPTER 9

The quality evaluation on osmo-dried products as affected by the processing has been determined in **Chapter 8**. The results obtained indicate the possibility that through utilization of broccoli stalk gives an opportunity to provide more access to food to the consumer. Therefore, in this **Chapter 9**, technology and economical aspects and the environmental benefits were analyzed discussed for implementation of combination drying at a commercial scale.

CHAPTER 9

APPLICATION AND THE TECHNO-ECONOMICAL ASPECTS OF INTEGRATED MICROWAVE DRYING SYSTEMS FOR DEVELOPMENT OF DEHYDRATED FOOD PRODUCTS

Abstract

The demand to increase food production to feed the growing number of world population is becoming a primary concern for everyone. At the same time, one-third of the food produced is lost or wasted globally. The food wastes and the by-products of the food production and processing operations still have nutritional ingredients in them suitable for human consumption. To encounter this issue, drying methods can be carried out to add value to food waste, prolong the product's shelf life and thus provide more food products. Integrated microwave drying systems have the potential to produce dehydrated products from the food waste. In this paper, integrated osmotic dehydration with microwave-assisted hot air drying method is described for the development of dried broccoli stalk product. The techno-economical aspects in terms of raw material availability, process design, energy input, capital inputs, operation costs and environmental benefits are also discussed. This integrated microwave drying concept will be of use to process other biological materials that have the potential at the market place.

Keywords: Food security, Food waste, Integrated drying, Microwave-assisted, Techno-economic aspects.

9.1 Introduction

The world population is projected to rise to 11.2 billion by the end of this century (United Nations, 2015). In order to feed this growing population, the current global food production also needs to be increased. However, an estimated one third of all the food produced in the world now ends up as waste in landfill sites (FAO, 2011). Decomposition of this waste leads to emission of greenhouse gases and thus contributes to climate change. In this scenario, wastage of food has to be recognized as a global problem that has a huge impact on economy, environment and society. Therefore, concerted efforts to prevent wastage along the entire food supply chain are required to provide more food for human consumption and also reducing the greenhouse gas emissions (WRAP, 2015).

Food supply chain is divided into five process steps such as production, post-harvest handling and storage, processing, distribution and consumption. According to FAO (2013), 54% of food wastage occurred at production and post-harvest handling and storage stages. Processing, distribution and consumption stages contribute to the remaining 46% of the total wastage. Poor harvesting techniques and improper handling of the crop produce during harvesting, especially in developing countries, also contributed to the food wastage (Godfray et al., 2010; Mendelsohn, 2014; Nonhebel & Kastner, 2011). In terms of food commodities, 44% of the total wasted food are fruits and vegetables (Gustavsson et al., 2011). Commonly, the production and processing of fruits and vegetables also generates crop remains and by-products that are usually discarded. However, they still contain sufficient nutritional ingredients that can be used for producing value added products for human consumption. After harvesting, the crop produce and residues continue to be physiologically active and start to deteriorate over time. They continue to lose water and it affects their appearance, texture, flavor and nutritive value. Hence, post-harvest technologies become an important key for extending the shelf life of crop produce, adding value to the waste resources and provide more food for human consumption.

Drying is an important processing method for water removal that reduces the water activity and extends the shelf life of food products. Drying operation facilitates the preservation and handling of foods and also reduces the transportation and storage costs through the reduction in bulk density of the food material (Orsat et al., 2007). However, the challenges associated with drying process, especially for thermal sensitive products, include reducing the moisture to a certain level where the products are shelf-stable, and produce good quality products in terms of physical properties, nutrient contents and also sensory values. There are various improved drying technologies available for food processing at the current juncture, and hybrid drying technologies that combine two or more of the drying methods are one of the most promising options to meet the demands for increased food availability.

9.2 Integrated Microwave Drying System with Osmotic Dehydration as Pre-treatment

Conventional heating techniques work remarkably at removing moisture from the surface of materials and the heat transfer inside the material deceases with increase in distance from the surface. In recent years, electro-heating using microwave energy which has become one of the most efficient drying methods as the heat is volumetrically distributed within the food material. In addition to stand-alone microwave drying methods, it also can be combined with conventional drying methods such as convection and conduction to gain even better improvement to reduce the drying time and enhance the product quality (Orsat et al., 2007). A considerable amount of research studies has been published on microwave hybrid drying systems for various food materials (Askari et al., 2013; Borquez et al., 2015; Duan et al., 2015; Kumar et al., 2014). In general, microwave assisted drying provides great synergy that can meet the four major requirements in drying of foods: (1) Speed of operation; (2) Energy efficiency; (3) Cost of operation; and (4) Quality of the dried products (Gunasekaran, 1999). Likewise, the osmotic dehydration method has received an increased attention as a potential pre-treatment for preservation of food material. Figure 9.1 present an overview on number of articles published between 2010 and 2015 on osmotic dehydration.

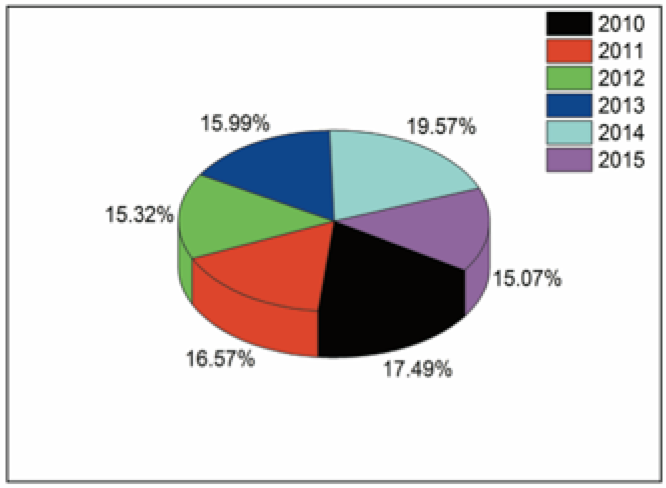


Figure 9.1: Overview of articles on osmotic dehydration from 2010 to 2015 (Source: Web of Science (2016)).

Osmotic dehydration is the partial removal of water by immersing food material in a hypertonic solution. During immersion, the food material is exposed to a gradient of chemical potential between the hypertonic solution and the inside of food that results in outflow of water from the food material and inflow of solute from the hypertonic solution to the food. The advantages of this dehydration technique as a pre-treatment prior to drying process are (Md Salim et al., 2016b; Raghavan & Orsat, 2008; Tadeusz, 2008): (1) Mass of fresh sample can be reduced by more than 50 %; (2) Energy savings from the moderate temperature used, usually from 30 to 40 °C and water is removed without a phase change; (3) Shorten the drying time for finish drying; and (4) Improve product quality attributes such as color, texture, aroma and nutritional value.

Application of osmotic dehydration as a pre-treatment prior to microwave-assisted drying has been extensively studied in producing high quality dried products (Nimmanpipug et al., 2013; Therdthai et al., 2011; Zhao et al., 2013). Torringa et al.

(2001) investigated the combined osmotic dehydration with microwave-hot air drying on mushroom and found that this hybrid drying method lowered the drying time and shrinkage of the dried product. Similar responses were reported by Changrue and Orsat (2009) on carrots. In an earlier investigation on drying of broccoli stalk slices in our laboratory, it was found that the constant drying rate period is no longer present when the osmotically dehydrated samples were dried under microwave-assisted hot air drying. While, in terms of product quality, the dried broccoli slices produced using the integrated osmotic dehydration with microwave-assisted hot air drying method was found to be better than the dried broccoli slices produced using either of microwave-assisted hot air or hot air drying methods (Md Salim et al., 2014). Thus, it is imperative to evaluate the technical and economic aspects of this hybrid drying method as discussed in the next section.

9.3 Techno-economical Aspects of the Combined Osmotic Dehydration and Microwave-assisted Hot Air Drying of Broccoli Stalk Slices

The following sections present a case study on how to add value to broccoli stalk slices, which is a harvest residue, as a dried product. Factors like raw material availability, process design, energy input, capital inputs, operation costs and environmental benefits that determine the sustainability of producing edible dried product from broccoli stalks are discussed.

9.3.1 Raw Material Availability

Broccoli is a cool season cultivated crop, with the optimum growing temperature range between 15 to 22 °C (Zvalo & Respondek, 2007). In Canada, the harvest season for broccoli begins in June and ends in October (Walker, 2005). The total production of broccoli in Canada in the year of 2015 was 41,456 metric tonnes, and this number has been steadily increasing since 2012 (Statistics Canada, 2015). About 47.6% of this production is from the province of Quebec. Commercially, broccoli is marketed for its florets, while leaving a considerable amount of harvested remains in the supply chain. The composition of harvested remains is illustrated in Figure 9.2. Thus, the estimated availability of broccoli stalks for processing, at an average farm size of 280 acres, in Quebec, Canada (Statistics Canada, 2012) is about 2.23 tonnes per day. This quantity of broccoli stalks is assumed as processed in two batches of eight hours each operations per day of the drying facility.

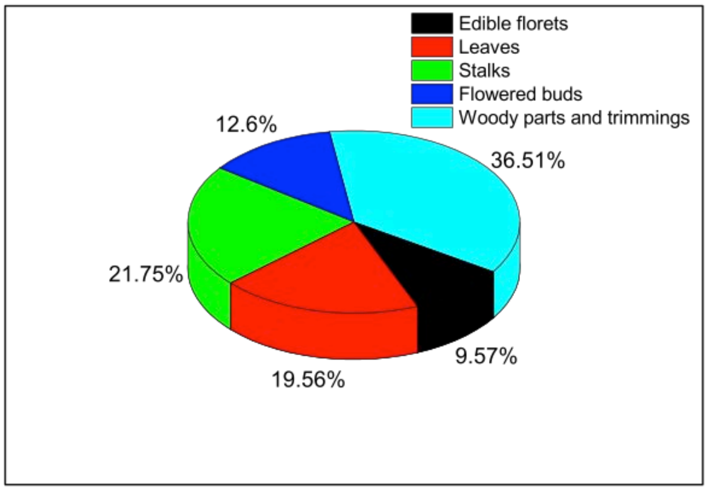


Figure 9.2: Composition of broccoli harvest remains (Source: Bekhit et al. (2013)).

9.3.2 Process Design

The processing steps for the production of osmo-dried broccoli stalk slices is illustrated in Figure 9.3. The broccoli stalks are cleaned first and then the cleaned stalks are peeled and sliced. The broccoli stalk slices contain about 94% moisture content (w.b.). Sucrose solution at a concentration of 54 °Brix was prepared and heated up from 25 °C to 30 °C. Once the desired temperature is reached, the broccoli stalk slices are immersed in the solution for two hours. For finish drying, the osmotically dehydrated broccoli stalk slices are dried using microwave-assisted hot air at a drying temperature of 40 °C, until it reached 10% moisture content (w.b.), after about 3.5 hours. The dried broccoli stalk slices are packed in printed polythene bags and cardboard boxes.

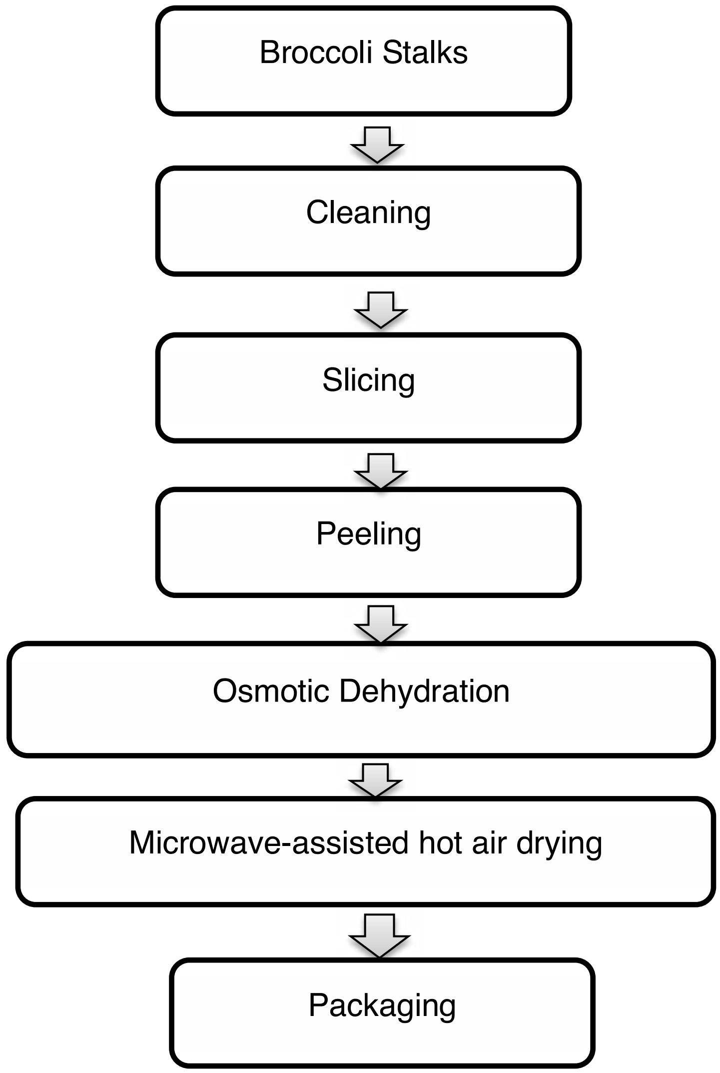


Figure 9.3 Flow diagram of processing of osmo-dried broccoli stalks.

The data on process design and yields are based on the laboratory-scale work conducted at the Department of Bioresource Engineering, McGill University. For one tonne of fresh broccoli stalks, the production of dried broccoli stalk slices is estimated as 97.5 kg. The mass loss at each stage of processing of a tonne broccoli stalk is given in Table 9.1.

Table 9.1: Estimated mass losses at each stage in the processing of a tonne of broccoli stalk.

Process	Mass loss description	Mass loss (kg)
Washing	Removal of impurities	50
Peeling	Removal of peel	95
Slicing	Removal of fine particles	85.5
Osmotic dehydration	Transfer of water into sugar solution	461.7
Drying	Water evaporation to reach 10 % final moisture content	205.2
Packaging	Loss during packaging	5.1

During osmotic dehydration, water is transferred from broccoli stalk slices to the sucrose solution and sucrose is transferred into the broccoli stalk slices. The volume (in litre) of 54 °Brix sucrose solution required for osmotic dehydration per kg of broccoli stalks being processed is at a ratio of 20:1. For every tonne of broccoli stalks, about 11 tonnes of sucrose and 9 tonnes of water are required to be in the solution. After each batch of osmotic dehydration process, the sucrose solution can be recycled at least 10 times (García-Martínez et al., 2002; Wray & Ramaswamy, 2016). During osmotic dehydration, nutrients are also leaching into the sucrose solution and therefore, the recycled sucrose solution can be used elsewhere considering the nutrients in it (Aachary & Prapulla, 2009; Morales et al., 2005; Shi & Xue, 2009).

The microwave-assisted hot air drying evaporates moisture from the osmotically dehydrated broccoli slices. This drying approach can minimize the retreating wet front, maintaining the evaporating surface and perhaps permitting entrainment-evaporation (Raghavan & Orsat, 2008). The rate of drying decreases with decrease in moisture content. For every tonne of osmotically dehydrated broccoli slices, this stage evaporate about 0.67 tonnes of water that can be condensed to use for the preparation of sucrose solution as well as to recover the heat energy in it.

9.3.3 Energy Input

The important factor in the production of dried broccoli stalk, which greatly determines the product cost, is the total energy input. In this study, the process is assumed at steady state. The energy required for heating sucrose solution as well as the energy to heat the broccoli slices could be estimated using Equation 9.1.

$$Q = mC_p\Delta T \tag{9.1}$$

Where, m is the mass (kg) of sugar solution and the broccoli slices; C_p is the specific heat (kJ/kg °C) of sugar solution and the broccoli stalk slices; and ΔT is the temperature (°C) difference from initial temperature to the processing temperature (30°C).

Considering the room temperature as 25 °C and C_p value of 54 °Brix sucrose solution as 2.95 kJ/kg °C and that of broccoli stalks as 3.85 kJ/kg °C, the minimum heat energy required for osmotic dehydration at 30 °C is about 0.65 MJ/kg-water removal from broccoli stalk slices. In addition to this, energy should be given in order to compensate for the energy loss of the process. However, the energy required in the osmotic dehydration of broccoli slices will be minimal when the atmospheric temperature is higher than 30 °C.

For the finish drying process, the energy to heat the osmotically dehydrated broccoli stalk slices can be calculated using Equation 9.1, considering the C_p value of osmotically dehydrated broccoli stalk slices as 3.56 kJ/kg °C. The energy required to evaporate water from the osmotically dehydrated broccoli slices during microwave-assisted hot air dying can be calculated using Equation 9.2.

$$Q = m\lambda \tag{9.2}$$

Where, m is the mass of water to be removed (kg) and λ is the latent heat of water evaporation, 2.46 x10^3 kJ/kg (Perrier & Tuzet, 2003).

Therefore, the minimum energy input required for microwave-assisted hot drying of osmotically dehydrated broccoli stalk slices, to a final 10% moisture content level, is about 2.54 MJ/kg-evaporated water. Additional energy must be provided, taking into account the dryer efficiency and heat losses. The total heat energy required to dry broccoli stalks in the combined osmotic dehydration and microwave-assisted hot air drying process is about 69% less than that of the stand-alone microwave hot air drying of broccoli stalks without any osmotic dehydration. Drying is the most energy intensive process in the production of osmo-dried broccoli stalk slices. Additional energy, at the rate of about 5% of the total energy required for drying, will be used for operating the facilities for handling of the materials and packaging of the dried product (Masresha, 2015).

9.3.4 Capital Inputs

Capital inputs refer to the cost of land, buildings, equipment and the working capital required to start the processing of fresh broccoli stalks to dried product (Lusch, 2011). Monetary inputs for land and buildings are required in establishing the processing facility. The area of land required is dependent on how much raw material is available at a given time and also on the processing capacity of the facility. Space is required for the delivery and storage of raw materials, consumables, and products; processing, packaging, administration works, etc. The cost of vegetable washer, peeler and slicer machines will vary depending on the machine specifications such as the input capacity and the infeed system type. Overall, the cost of automatic conveyer belt systems that will provide higher processing capacity will be higher than that of manually controlled conveyer systems. For osmotic dehydration process, direct heating method can be applied, since it is highly efficient due to direct absorption of heat by the sucrose solution from the heater. The cost of the microwave-assisted drying system includes that of microwave generator, wave guide tube, applicator, control system and conveyer (Schiffmann, 2006). In this study, the costs of equipment are obtained from different manufacturers/suppliers and the estimated fixed capital cost is divided into direct and indirect cost as shown in Table 9.2. The estimation percentage adapted from Masresha (2015).

Table 9.2: Capital costs estimation for the broccoli stalks processing facility in Quebec, Canada.

Component	Estimated Cost (CAD)
Direct cost (DC)	
Purchased equipment cost (PEC)	60,465
-Washer & Peeler (Capacity: 125 kg/h)	
- Slicer (Capacity: 110 kg/h)	
- Osmotic dehydration unit (Capacity: 100 kg/h)	
- Dryer (Capacity: 40 kg/h)	
- Packaging unit (Capacity: 15 kg/h)	
Insulation (39% PEC)	23,581
Instrumentation & control (13% PEC)	7,860
Piping (31% PEC)	18,744
Electrical equipment and materials (10% PEC)	6,046
Building (29% PEC)	17,535
Yard Improvement (10% PEC)	6,046
Land (6% PEC)	3,628
Service facilities (55% PEC)	33,256
Total DC	177,162
Indirect cost (IC)	
Design and Engineering (25% DC)	44,291
Contractor fees (18% DC)	31,889
Contingency (10% DC)	17,716
Total IC	93,896
Fixed capital cost (FCI) = DC+IC	271,058

Additionally, the working capital is estimated at 15% of initial capital inputs incurred (Masresha, 2015). Thus the total capital investment is found to be about CAD 311,717.

9.3.5 Operating Costs

The operating costs can be divided into variable operating costs and fixed operating costs. Variable operating costs can be described as the cost of consumable items like sucrose, printed polythene bags, cardboard boxes, water, and electricity. Water is required for washing and cleaning of the raw material and the facility. While, electricity is required from sample preparation to packaging stages. Packaging materials like polythene bags are required for the storage and delivery of the processed broccoli stalk slices for consumption. The cost of consumable items is directly dependent on quantity of broccoli stalks processed and the market conditions. The price of electricity and water are dependent on the local tariff.

Meanwhile, fixed operating costs will not vary much with the production rate as compared to variable costs. Fixed operating costs include, for example, the materials that are required for the maintenance and repairs of the processing machinery and the facility, wages for operating and maintenance people, laboratory charges, depreciation, taxes, and insurance costs. Labour cost required in the processing of broccoli stalks is dependent on the automation of the facility and also on the local labour availability and wage conditions (Dudbridge, 2011). The minimum wage in Quebec in 2016 is CAD 10.75 per hour. Laboratory cost is needed for process monitoring and quality control. Cost for supervision can be estimated as 20% of cost for operating labour. Whereas, the plant overhead charges are estimated as 50% of the labour cost.

There are administrative costs associated with the maintenance and operation of the facility and it will be a part of general expenses that determine the product cost. Finally, there will be distribution and selling costs, at about 2% of the total operating cost, associated with the final product price. The estimation of total operating costs is shown in Table 9.3.

Table 9.3 Summary of annual operating costs for the production of osmo-dried broccoli stalk slices in Quebec, Canada.

Component	Estimated Cost (CAD)
Variable costs	
Raw materials	253,935
Utilities	7,650
Packaging material	67,314
Sub-total A	328,899
Fixed costs	
Maintenance (6% FCI)	16,263
Operating labour	103,200
Laboratory cost (20% Operating labour)	20,640
Supervision (20% Operating labour)	20,640
Plant overheads (50% Operating labour)	51,600
Depreciation (10% FCI)	27,106
Local taxes (2% FCI)	5,421
Insurance (1% FCI)	2,711
Sub-total B	247,581
Direct production cost (A+B)	576,480
General expense	
Administrative	21,016
Distribution and selling	12,194
Sub-total C	33,209
Annual operating cost = A+B+C	609,689

The profitability assessment of a production process are performed based on the return on investment (ROI) and payback period (PBP) calculations. The ROI is calculated by dividing the expected profit by total capital investment incurred and expressed as percentage (Ray & Johnston, 1989). The PBP is the number of years it may take to recover the initial cost incurred at the start of a production operation. The payback period can be calculated as shown in Equation 9.3 (Masresha, 2015).

$$PBP = \frac{\text{Fixed capital investment}}{\text{Net profit} + \text{Depreciation}} \tag{9.3}$$

The sale price, including a minimum profit of 20%, of the dried broccoli stalk slices produced in a facility that process about 333.8 tonnes of broccoli stalks to 30.8 tonnes of dried products, is found to be CAD 23.8/kg. Thus, after tax, the net annual earning is calculated as CAD 106,756. Thereby, the ROI is computed as 34.3% and the PBP is found to be about 2.03 years.

9.4 Environmental Benefits

The global wastage of food is estimated as 1.3 billion tonnes per year. The decomposition of this waste is associated with the release of about 3.3 billion tonnes of CO_2 equivalent greenhouse gases per year globally (FAO, 2013). This emission contributes to global warming and the associated catastrophes and economical damages (Carlsson-Kanyama, 1998; Godfray et al., 2010). Diverting broccoli stalks, that otherwise would have ended up directly in the landfill sites, into nutritious food source, will reduce the GHG emissions and thus save environment and benefits economy. However, the economic benefits available through the reduction in GHG emissions, is practically difficult to estimate (Godfray et al., 2010) and possibly one of the practical ways is by considering the carbon credit associated with the projects that reduces GHG emissions. Since the conversion of broccoli stalks into food products can reduce the emission of

GHG gases, it can be used to claim and trade carbon credit to gain additional economic benefits (Bosch et al., 2008).

9.5 Conclusion

In view of improving the food security and harnessing the climate change, innovative use of food resources should be developed and practiced. Broccoli stalk is an underutilized but nutrient rich material that can be processed as food material using advanced processing methods like the combined osmotic dehydration and microwave-assisted hot air drying. The integration of these individual processes results in about 69% reduction in energy input required in the preparation of dried broccoli stalk slices, when compared to the stand-alone microwave-assisted hot air drying. The ROI of 34.3%, 2 years of PBP and environmental benefits point to the economic feasibility of this process. Thus, the production of edible products from waste material like broccoli stalks has apparent economic and environmental benefits.

CHAPTER 10

GENERAL SUMMARY, CONTRIBUTION TO KNOWLEDGE, AND RECOMMENDATION FOR FUTURE RESEARCH

10.1 General Summary

Utilization of harvest remains that still contains nutritional profile for food consumption like broccoli stalks can contribute to waste minimization and subsequently protect the environment. Microwave hybrid represents a potential and practicable drying method for development of dried product. In this study, a combination of microwave-assisted hot air drying with osmotic dehydration has been evaluated. The step-by-step approach was implemented to improve the heat and mass transfer process with better quality product during the drying of broccoli stalk slices. The main conclusions from each chapter are recapitulates as follows:

- In **Chapter 1** and **Chapter 2**, the motivation to adding value of harvest remains for food production especially in broccoli industry has been highlighted. Various microwave hybrid drying can be implemented for the development of dried products and combination of osmotic dehydration and microwave-assisted drying is emphasized in this study.
- **Chapter 3** study provided important information on the beneficial implementation of microwave energy with hot air drying in terms of saving drying time and energy required when compared to hot air drying alone for the development of the dried broccoli stalk slices. However, a pre-treatment before the drying process is

required to enhance the quality of the dried product under microwave-assisted drying.

- Among the pre-treatment process available nowadays, osmotic dehydration was chosen due to its simplicity and energy saving advantages. In **Chapter 4**, the effects of processing factors on mass transfer exchange during osmotic dehydration of broccoli stalk have been studied under static conditions. The optimum conditions for osmotic dehydration to achieve 61.5% water loss and 6.7% solute gain was found to be at sucrose concentration of 56 w/v % operating at temperature of 42 °C for four hours immersion time. This processing condition was used as a pre-drying step under microwave-assisted hot air drying.
- The performance of the hybrid drying (osmotic dehydration and microwave-assisted hot air drying) as described in **Chapter 5** is excellent in terms of improving the physical quality of the dried broccoli stalk slices. The challenges in this process was the longer processing time required during the osmotic dehydration. Therefore, an improvement in osmotic dehydration process for fastening the mass transfer is advantageous.
- **Chapter 6** described the development of a continuous flow osmotic dehydration equipment to achieve better hydrodynamic control during the pre-treatment step. In this study, it was conclusively shown that the mass transfer was enhanced with the continuous flow applied around the sample during the osmotic dehydration process. The optimum conditions of this process to achieve 65% water loss and 3.9% solute gain was found to be at temperature of 30 °C with concentration of 54 °Brix at 120 min of immersion time and a flow velocity of 3.5 mm/s. This processing condition was used as a pre-drying step for the microwave-assisted hot air drying.
- Another parameter that is important to take into consideration prior to microwave drying was the dielectric properties of the material. In **Chapter 7**, the effects of osmotic dehydration processing factors on the dielectric properties of broccoli stalk at frequency 2.45 GHz was evaluated. It was found that ε' and ε'' were significantly influenced by all the osmotic dehydration operating variables resulting

in the decrease of ε' and increase in ε'' of broccoli stalk slices. Hence, better energy conversion into heat during microwave drying of broccoli stalk slices can be achieved.

- In **Chapter 8**, some of the physical, bioactive compounds and nutritional qualities as affected by osmotic dehydration and microwave drying at different temperatures were evaluated. A significant amount of nutrient losses was found possibly due to leaching into the sucrose solution. Drying temperature of 40 °C under microwave-assisted hot air drying was suggested as a preferable drying condition for development of osmo-dried broccoli stalk.
- Based on the promising drying performance and product evaluation obtained from previous chapter, scaling up of the process technology from laboratory scale to industrial production was discussed in **Chapter 9**. The integration of the individual processes results in 69% reduction in energy input required, compared to the stand-alone microwave-assisted hot air drying. ROI of 34.3% and 2 years PBP reported in this study show that this process is economically feasible.

10.2 Contributions to Knowledge

Contributions of this research lie in enriching scientific knowledge on osmotic dehydration process and microwave hybrid drying concept for development of ready-to-eat products. The specific contributions are as follows:

- The optimum conditions for osmotic dehydration under static bath and continuous flow conditions were evaluated, which is useful to provide information to achieve better energy conservation for further drying in the development of dried broccoli stalk slices.
- The creation of laminar flow condition during osmotic dehydration was found to provide better mass transfer when compared to static condition.

- The study showed that Reynolds number (*Re)* is another important parameter that needs to be considered to provide better hydrodynamic control during the osmotic dehydration process.
- The effects of osmotic dehydration operating variables on the dielectric properties of broccoli stalk were evaluated.
- The effects of osmotic dehydration in microwave-assisted hot air drying on the drying kinetics, moisture diffusivity and product quality of broccoli stalk were determined.
- The results demonstrated for osmo-dried broccoli stalk product might be useful for developing other products.

10.3 Recommendations for Future Research

This study has assessed the potential development of osmo-dried broccoli stalk product through the application of osmotic dehydration and microwave-assisted hot air drying. In relation to that, a few recommendations for future research are highlighted as follows:

1. In economic point of view, the possibility for reusing the osmotic solution after the osmotic dehydration process can contribute to the successive waste management. Therefore a few studies in this context can be pursed:
    - The potential of recycling the osmotic solutions on the performance of mass transfer exchange can be studied. In addition, by performing the analyses of the microbiological activity, pH and water activity of the osmotic solution can helps to control the microbial spoilage.
    - Leaching of natural substances into the osmotic solution during the osmotic process can be a good source as a value added sweetener for others products. Therefore, the study on composition of the osmotic solution can be advantageous for food industry.
2. Microwave reflectrometry can be used as an alternative method to monitor the osmotic solution changes during the osmotic dehydration process. This technique can help in automatic monitoring and control.

3. Reynolds number (*Re*) was found to be an important parameter that needs to be considered to provide better hydrodynamic control during the osmotic dehydration process. Thus, following studies will be an advantage to improve the knowledge on the mass transfer exchange under continuous flow condition:
    - Simulation and modelling on the effects of flow conditions at different *Re* on the boundary layer of the sample will help to predict the performance of the mass transfer exchange occurred during the osmotic dehydration.
    - *Re* number is dependent on the density and viscosity of the solution. Thus, study on the influence of *Re* for different osmotic agents under continuous flow conditions can be performed to provide comparative study on the influences of these properties on the mass transfer process.
4. The microwave power density used in this study was 2 W/g with 1.4 m/s superficial air velocity. Optimization of both parameters could be of value for developing an efficient drying process.
5. Comparative studies under different combination of microwave with other drying techniques such as vacuum drying, spouted bed or fluidized bed can help in producing high quality dried broccoli stalk products.
6. Computational modelling and simulation can be performed to provide better understanding of the controlling mechanisms for heat and mass transfer during the drying process.

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