M. Tech.
(Research)
Thesis

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Infrared and Microwave Assisted Hot Air
Drying of Lemon (*Citrus limon* L.)

NIT
2017

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Dissertation submitted in partial fulfilment
of the requirements of the degree of

Master of Technology (Research)
in
Food Process Engineering

by

Deepika S

(Roll Number: 614FT3002)

based on research carried out
under the supervision of

Dr. P. P. Sutar

August, 2016

Department of Food Process Engineering
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May 01, 2017

Certificate of Examination

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This is to certify that the work presented in this dissertation entitled *Infrared and Microwave Assisted Hot Air Drying of Lemon (Citrus limon L.)* by *Deepika S*, Roll Number *614FT3002*, is a record of original research carried out by her under my supervision and guidance in partial fulfilment of the requirements of the degree of *Master of Technology (Research)* in *Food Process Engineering*. Neither this dissertation nor any part of it has been submitted for any degree or diploma to any institute or university in India or abroad.

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Dedicated to my

beloved Parents

Declaration of Originality

I, *Deepika S,* Roll Number *614FT3002* hereby declare that this dissertation entitled *Infrared and Microwave Assisted Hot Air Drying of Lemon (Citrus limon L.)* represents my original work carried out as a post graduate student of NIT Rourkela and, to the best of my knowledge, it contains no material previously published or written by another person, nor any material presented for the award of any other degree or diploma of NIT Rourkela or any other institution. Any contribution made to this research by others, with whom I have worked at NIT Rourkela or elsewhere, is explicitly acknowledged in the dissertation. Works of other authors cited in this dissertation have been duly acknowledged under the sections "Reference" or "Bibliography". I have also submitted my original research records to the scrutiny committee for evaluation of my dissertation.

I am fully aware that in case of any non-compliance detected in future, the Senate of NIT Rourkela may withdraw the degree awarded to me on the basis of the present dissertation.

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Acknowledgement

Any work in general and that of research in particular does not just involve the efforts put in by the people being credited for, it takes in the toil of the numerous many who work behind the screen to make it happen. Here, I made an attempt to express my gratitude to all such people who helped me in their own ways to make this project successful.

First of all, I would like to thank **my parents** for their moral support, patience and sacrifices throughout my life. Words are insufficient to express my deep love and affection towards them.

I express my gratitude and indebtness to my supervisor, **Dr. P. P. Sutar**, Assistant Professor, Department of Food Process Engineering for his valuable guidance, support and supervision throughout the project period. He has been an excellent mentor throughout the project and I am indeed lucky to receive his knowledge and advice.

I am grateful to my MSC Chairman and members, Prof. R. K. Singh, Prof. R. C. Pradhan, Prof. S. Mishra, Prof. Aditya Kumar for their guidance and support throughout the project period. I would like to acknowledge the technicians, Mr. Rajesh Kumar Khuntia and Mr. Toshamani Meher for their contribution during the research period.

I am are very thankful to my beloved seniors especially Er. Shivanand Shirkole, Er. Gitanjali Behera, Er. Om Suryawanshi, Er. Sudarshanna and Er. Adinath Kate for their affection and profound support at every step in my project. They really made things easier for me with their insights and expertise. This acknowledgement would be incomplete without giving credit to my lovable friends Srimagal, Sivasankari, Leonard, Shashikumar, Irshaan, Senthilguru and Tamanna, for their constant support and enthusiasm.

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Abstract

The Lemon (*Citrus limon* L.) has many important natural chemical components, including citric acid, ascorbic acid, minerals and flavonoids. Storage of lemons is difficult due to enzymatic activity and production of fermentative metabolites. Pectinesterase and peroxidase are the active enzymes present in the peel and juice sacs of lemon, respectively. In the present study different pretreatments like osmotic pre-concentration, chemical dipping (ascorbic acid and calcium chloride), ultra-sonication and blanching (water and steam) were given to lemon to inactivate pectinesterase and peroxidase enzymes. Osmotic dehydration was found to be a good pretreatment to attain higher moisture loss and solid gain making lemon more suitable for further drying. The optimal condition of osmotic dehydration was found to be 20% salt concentration and 30 °C osmotic solution for 180 min to attain high moisture loss, less solid loss and required salt uptake within allowable limits. Also, steam blanching was found to be most suitable pretreatment among other pretreatments to inactivate enzymes. Therefore, osmotically pretreated lemons followed by one minute steam blanching were used for infrared hot air drying. Infrared hot air drying of osmotically pretreated and steam blanched lemon slices was effective in partially drying (reduce moisture content less than 30% w.b.) during one hour without entering in drastically falling rate period. Microwave hot air finish drying was found to be energy and drying time saving with minimum deterioration of the quality of product. Also, microwaves helped in maintaining the higher drying rates in final stage where removal of moisture was difficult. The hybridization of these two drying methods overcame the limitations and combined advantages of infrared and microwaves at appropriate moisture levels of the product.

Keywords: Peroxidase, pectinesterase, enzyme inactivation, infrared, microwave

Contents

List of Figures

List of Tables

List of Abbreviations

ANOVA	Analysis of variance
AA	Ascorbic acid
cP	Centipoise
CV	Coefficient of variation
D	Diffusivity
d.b.	Dry basis
DHC	Dry matter holding capacity
DM	Dry matter content
E	Colour
FDA	Food and Drug Administration
H	Enthalpy
IM	Initial moisture
IR	Infrared
k	Rate constant
K_1	Peleg rate constant
K_2	Peleg capacity constant
mc	Moisture content
ML	Moisture loss
MR	Moisture ratio
n	Page model constant
p	Probability

PD	Power density
R^2	Coefficient of determination
RMSE	Root mean square error
S	Percentage of dry matter content
S_1	Azuara constant related to moisture diffusion out of the product (min^{-1})
S_2	Azuara constant related to salt diffusion into the product (min^{-1})
S_3	Azuara constant related to lemon’s solid diffusion from the product (min^{-1})
SEC	Specific energy consumption
SG	Salt gain
SL	Solid Loss
W	Weight of the sample
w.b.	Wet basis
X	Variable for ML,SG and SL

List of Symbols and Subscripts

Symbols

η	Viscosity
θ	Time
Δ	Difference
α	Code variable in experimental design
ε'	Dielectrical constant (real part)
ε''	Dielectrical constant (imaginary part)

Subscripts

i	initial
f	final
∞	equilibrium
os	occurring due to osmosis

Chapter 1

Introduction

The lemon (*Citrus limon* L.) is highly acidic in nature mainly because of the presence of 5-6 percent citric acid. The major morphology of a typical lemon are flavedo, albedo, endocarp, rag and pulp. Globally, India stands 1st in the production of lemon. In 2013, India produced 2.52 million tonnes of lemons that contributed around 14.35 percent of total global production [1]. Lemon is a rich source of antioxidants mainly due to ascorbic acid, polyphenols and carotenoids. Limonene is major aroma compound responsible for its flavor and bitter taste [2]. The total phenolic content in lemons is significantly higher than that in oranges and grapefruit. Lemons are indispensable element of many popular drinks. Lemonade, lemon tea, lemon pastries, lemon flavored cakes, biscuits and puddings are few products of lemon. Dried lemon slices are widely used for typical flavor. In many countries dried lemon slices are used during cooking of bacon and steak. Storage of lemons is practically difficult since cold storage can increase its shelf life but causes chilling injury due to its increasing peroxidase activity at low temperature [3]. Under aerobic conditions, lemons produce fermentative metabolites (ethanol, acetaldehyde and ethyl acetate) and these volatiles can affect the sensory attributes if they are present in high amounts [4]. Hence, there is a great need for preservation of the lemon slices. There are various enzymes responsible for the deterioration of lemon fruit. Generally in citrus fruits the common enzymes present are proteolytic enzyme like pectinesterase present in juice sacs, phosphatase present in peel and juice, and peroxidase in peel and seed coat. The most active enzyme in these are pectinesterase and peroxidase. These enzymes can be inactivated by thermal treatment, ultrasonication, chemical treatment or combination of methods. The high water activity of lemon makes it highly prone to deterioration [5]. Water activity is the most important measure for assessing the stability of the lemon [6, 7]. Low water activity reduces the growth of micro-organism, oxidative and enzymatic reactions thus providing product stability [8-10]. Osmotic dehydration is considered as one of the best methods for reducing the water activity of fruits and vegetable. It is a partial water removal process to obtain product at intermediate moisture and potential methodology to attenuate nutrient degradation during processing of food materials [11-14]. During the process of osmotic dehydration the product is immersed in an aqueous hypertonic solution [15, 16]. Due to the osmotic pressure gradient a driving force starts up and initiates a simultaneous counter-current mass transfer [17, 18]. In the mass transfer process, water diffuses out and solute infuses inside the permeable parenchymatic tissue [19, 20]. Since the semi permeable membrane is not selective, adversely another counter flow also occurs where the fruit's natural solutes percolates along with water into the osmotic solution [21, 22]. Literature shows that the transfer of natural solutes have been considered negligible during mass balance calculation of fruit and vegetables [23, 24]. But, leaching of product's constituents becomes important in case of products like lemon slices with low dry matter holding capacity. It releases directly juice sacs into the osmotic solution. Major factors responsible for the mass transfer during osmotic dehydration of fruit and vegetables are temperature, concentration of the osmotic agent, thickness and geometry of the food material, sample to solution ratio, pressure and rate of agitation [25-30]. Osmotic pre-treatment also serves in enzyme inactivation to some extent and reduces browning during finish drying. Also lemon can be dried by other mechanical methods to increase its shelf life, ease its storage and transport [6, 31]. In conventional hot air drying, higher temperatures or prolonged drying period causes severe damage to product flavor, colour and nutrients, reducing rehydration capacity of the dried product [32]. Freeze drying is proved to maintain quality of dried lemon to greater level but its cost is extremely high due to sublimation and vacuum generation.

In recent years the application of microwave is becoming popular due to its unique heating mechanism that serves as an energy efficient heating method. Microwaves are very short electromagnetic radiation with wavelengths from 1 mm to 1 m that travel at the speed of light. Its frequency range varies from 300 MHz to 300 GHz [33]. Application of microwave in various fields include polymer and ceramics industries [34, 35], medicine [36, 37] and food processing. Although the use of microwave oven is widely spread, the application of the technology in food industries is relatively a new progress. Microwave drying has emerged as an alternative to conventional drying technique since electromagnetic energy is supplied directly to the food material through volumetric heating. This provides rapid temperature rise throughout the material with reduced thermal gradients. Microwave heating can lead to overheating in food material and formation of cold and hot spots if applied improperly [38, 39]. Similarly, infrared drying which is another alternative to conventional drying produces rapid surface heating rates resulting into reduced drying time. For foods in which infrared energy penetrates significantly, addition of infrared actually increases the surface moisture build-up. When energy is absorbed mostly on the surface, infrared can reduce surface moisture rapidly [40, 41].

Hybrid drying is one of the best alternative to overcome drawbacks of different drying methods and increase efficiency of the process [42]. Literature review shows that the detailed study on hybrid drying of lemon using combined microwave and infrared heating is not available. Hence, the present study was aimed with a hypothesis that, enzyme inactivation in lemon can be done by osmotic, ultrasound, thermal and chemical pretreatment, and drying can be done by combination of microwave- infrared and hot air for better results especially to maintain the product color and retain its ascorbic acid content. The outcome of the study may provide a new hybrid process of drying for obtaining the quality dehydrated lemon slices. Therefore, the present investigation was undertaken with the following objectives:

1. To study the effect of pretreatment on lemon slices prior to drying.
2. To study the microwave and infrared hot air drying characteristics of pretreated lemon slices.
3. To study the sorption behavior of dried lemon slices.

Chapter 2

Review of Literature

This chapter deals with review of relevant literature pertaining to various parameters involved in present investigation on pretreatment of lemon using osmotic dehydration and other methods followed by microwave-infrared drying of pretreated lemon slices.

2.1 Osmotic Dehydration Pretreatment

2.1.1 Osmotic dehydration process

Osmotic dehydration is a processing method, used to produce products with intermediate moisture content. In osmotic dehydration process, the products are placed in hypertonic solutions, such as, granular salt, granular sugar, brines, syrups or ternary (sugar and salt) solutions. The moisture movement from the product to the osmotic solution is governed by the osmotic pressure gradient. Apart from the moisture movement the solute from the hypertonic solution moves into the product (Fig. 2.1).

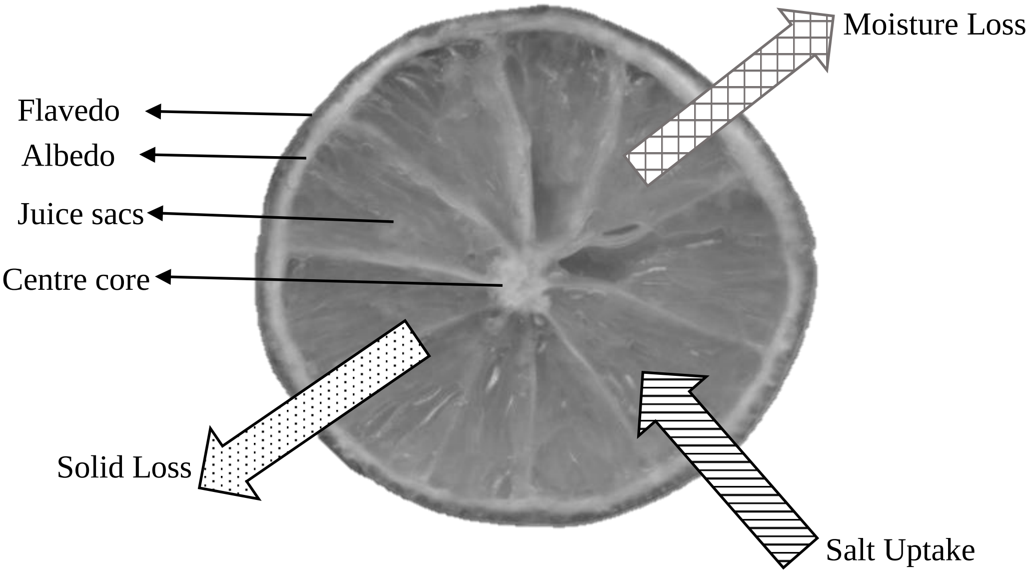


Figure 2.1 Cross sectional view of lemon slice indicating mass transfer during osmotic dehydration

In order to reduce energy consumption and heat damage, the practice of osmotic dehydration to fruits, has received attention in recent years as a technique for production of shelf-stable foods and intermediate moisture foods, or as a pretreatment prior to drying [43].

Selection of the osmotic agent should be pondered since the ionic behaviour or molecular weight influences the kinetics of moisture loss and solid gain. The most common features of osmotic agent are, low molecular weight, acceptable smell and edible taste, stability with other food components [44]. The most common osmotic agents are sugar and salt. The presence of the osmotic agent on the surface of the product hinders movement of oxygen inside and helps in reducing enzymatic browning [45, 46]. While the existence of excess solute in the product gives an undesirable taste [47]. The impregnation of the solute during osmotic dehydration changes the properties of the product after treatment [48, 49]. The most commonly recognised changes are shrinkage of cells, plasmolysis, loss of shape, loss in turgor pressure and disruption of cells due to the osmotic stress [50-52]. If osmotic dehydration is performed for a long period, equilibrium conditions occurs between the food product and the aqueous solution. At that condition the water loss, solute gain and solid matter loss becomes negligible [26].

During the process of osmotic dehydration, the mass transfer rate increases with increase in solution volume and hence the solid gain increases rapidly. In the case of apricot, researchers have reported that the best concentration and temperature that had a high solid gain to moisture loss ratio and an acceptable taste were 5% and 40°C, respectively [53]. Increment of osmotic solution concentration results in an increased osmotic pressure gradient and, hence leads to higher water loss and solid gain [50]. The moisture loss and solid gain increases with increase in osmosis time [54].

2.1.2 Factors affecting osmotic dehydration process

The osmotic dehydration kinetics is affected by the size and shape of the samples due to different specific surface area or thickness to surface ratio. Also, different forms of samples are selected on the basis of end-use of product after further processing [55-58]. Besides the cost of the solute, the organoleptic properties of the final product is the most important consideration. Salt (NaCl) is found to be an excellent osmotic agent for vegetables as it changes cell permeability, the use for dehydration of fruits is limited due to the salty taste of final product [59-61].

Temperature and time of the hypertonic solution plays a vital role during the dehydration process. Higher temperature results in the rapid water loss by plasticizing of cell membranes, increased water diffusion within the product and faster mass transfer characteristics due to the decrease in viscosity of the osmotic solution. The temperature effect on the kinetic rate of moisture loss without affecting solid gain, is more pronounced between 30 to 60ºC for fruits and vegetables [62-64].

The sample to solution ratio is an important parameter during osmotic dehydration. Several researchers have been using the sample to solution ratio ranging from 1:1 to 1:5. Higher sample to solution ratio ranging from 1:10 to 1: 60 can be used in order to prevent the dilution of the osmotic medium due to gain of moisture from the product and loss of solid to the product, which subsequently leads to decrease in the osmotic driving force during the osmotic dehydration [55, 65-68]. As the time of treatment increases, the weight loss increases while the rate of reduction decreases [60]. The rate of moisture loss and solid gain is faster within the first hour of osmosis followed by lower rates for the remaining duration [69-71] and researchers found that about 25% loss of initial moisture occurs during the first hour of treatment and 40% after three hours of treatment.

2.1.3 Mass transfer kinetics during osmotic dehydration

Various empirical and semi- empirical models were developed by researchers to predict the mass transfer kinetics during osmotic dehydration process. Peleg [72] developed a two-parameter sorption equation based on Fick’s law of diffusion. It was used to determine the sorption process in various food materials. The Peleg constants gave the rate of mass transfer and the capacity of the product to attain maximum sorption. Further, Azuara [69] proposed a two-parameter equation based on Peleg model for determining equilibrium moisture loss and solid gain. Besides, the advantages of the empirical and semi-empirical models the limitation to be considered is that the phenomenological mechanisms are not taken into account [73, 74]. In Peleg and Azuara model, the third parameter i.e. solid loss is assumed to be negligible which is the major limitation. For osmotic dehydration of lemon slices, a two parameter equation to predict the kinetics of osmotic dehydration and final equilibrium point can be used. But modelling moisture loss and solid gain of lemons dipped in salt solution by considering all other significant mass transfer phenomena (solid and juice sacs losses) is not reported in literature.

2.2 Chemical and Thermal Pretreatments

Enzymes are prime reason for deterioration of the food materials. Many researchers have reported studies on enzyme inactivation using various pretreatments. Pre-treatment is required to reduce post-harvest losses and retard degradation of the produce. Pre-treatment can inactivate enzymes but in many cases food products are dried without pre-treatments.

Typical drying temperature is not sufficient to inactivate the enzymes which leads to the presence of residual enzymes. The presence of residual enzyme in processed product may cause quality damage like color, texture and nutritional losses [75]. Blanching is the common heat treatment to inactivate the active enzymes and remove the entrapped air. Despite, it has detrimental effect due to undesirable quality changes by degrading organoleptic and nutritional properties [76, 77].

2.2.1 Chemical pre-treatments

Use of chemical additives can decrease the heat resistance of the enzymes. Researchers found that calcium ions have great effect on pectinesterase activity. Calcium ions protects the cell wall and prevents the destruction of cell compartments thereby avoiding the mixing of substrates and enzymes [78]. Lower concentration of calcium between 5 and 25 mM can activate enzyme while higher concentration have an inhibitory effect [79]. The addition of NaCl and KCl accelerates the activity of pectinmethylesterase. Thermal treatment between 65 and 90 °C for 5 min completely inactivates pectinesterase [80]. Even the thermostable pectinmethylesterase can be inactivated by heat treatment at 90 °C for 1 min [81]. Besides the enzyme inactivation purpose of calcium chloride it is also used as a firming agent and for controlling physiological disorders and reducing the incidence of fungal pathogens [82, 83]. It also reduces the mechanical damage occurring during post-harvest practises by maintaining the cell wall structure in fruit [84] . The calcium ions forms calcium pectate by interacting with the pectic substances in inner part of the cell wall [85]. According to FDA, maximum level of 0.2 and 0.4% can be used as a firming agent for fruits and vegetables respectively. The addition of ascorbic acid can inactivate peroxidase enzymes and prevent enzymatic discoloration. But the effect of ascorbic acid is insufficient since once the added ascorbic acid is completely oxidised the o-quinones accumulates and leads to colour degradation [86, 87]. Hence the combination with other treatments can give pronounced effect on enzyme inactivation.

2.2.2 Thermal pre-treatments

Blanching

Blanching is a heat treatment or a partial cooking process, usually carried out in water or steam, prior to dehydration. It is intended to inactivate the enzymes responsible for oxidation, enzymatic browning and the retard undesirable reactions that adversely affect the quality of the product. The effectiveness of blanching can be determined by the degree of inactivation of stable enzymes. Peroxidase, the heat stable enzyme, is generally used as an index for blanching. The other benefits of blanching are removal of entrapped air from the tissues, reduced drying time, retention of vitamins and softening of texture [88]. Despite of the advantages prolonged blanching leads to degradation of quality [89]. Researchers also reported that Low Temperature Long Time (LTLT) blanching can maintain the quality when compared to High Temperature Short Time (HTST) blanching [90]. The loss of ascorbic acid is high in water blanching than steam blanching. Pronounced leaching of constituents was reported during high temperature water blanching of fruits and vegetables [91].

2.3 Ultrasound Pretreatment

Researchers have found ultrasound as an alternative to other traditional heat treatment method to inactivate enzymes. Ultrasound produces cavitation bubbles and generates temporary spots of extremely high temperature and pressure when imploded thereby leading to enzyme inactivation [92]. Ultrasound can be used alone or in combination with other treatments to reduce the activity of enzymes and increase the shelf life [93, 94]. Ultrasound treatment can inactivate 60% of active pectinmethyesterase. Ultrasound treatment in tomato products can improve the rheological properties due to inactivation of pectinmethylesterase and reducing particle size [95, 96]. The treatment time and amplitude level of ultrasound are the factors affecting quality loss and ascorbic acid degradation [97]. The combined treatment of ultrasound with ascorbic acid can reduce the residual activity of peroxidase even after storage period which is not possible in individual treatment [98].

Van den Broeck et al. [99]studied the influence of additives and pH on thermal and combined pressure-temperature inactivation of orange pectinesterase. They found that inactivation kinetics are dependent on pH, enzyme concentration, Ca^{2+} ions and sucrose. Increase in calcium concentration increases the pressure stability, whereas it decreases the temperature stability. Lamikanra and Watson [100] studied the effects of ascorbic acid on inactivation of peroxidase of fresh-cut cantaloupe melon and found that ascorbic acid reduces peroxidase activity. Increased inactivation of pectinmethylesterase in ultra-sonicated juice was reported for a temperature range of 50° –72 °C compared to thermal treatment. Increase in inactivation was mainly dependent on cavitational intensity which was reported to be dependent on temperature [101]. Jang and Moon [98]studied the effects of ascorbic acid and ultrasound on activity of peroxidase and polyphenol oxidase of apple during storage and their investigation proved simultaneous effect on inactivation of the enzymes .

2.4 Conventional Drying of Lemon and Ascorbic Acid Rich Fruits

Drying is a conventional method of food preservation which inhibits microbial activation. Lemon is generally dried by open sun and using different mechanical dryers with temperature ranging more than 50 °C. During lemon drying in a closed type solar drier, loaded at 650 – 700 g batch^{-1} with the thickness of 4-6 mm, where the temperature for drying was maintained at 60 ºC and 36 to 52 ºC for hot air and solar drying, respectively. The solar dried lemon was more acceptable in all aspects especially with respect to color [6]. López et al. [102] studied the effect of air temperature on vitamin C, drying kinetics, total phenolic content, antioxidant capacity, color due to non-enzymatic browning and firmness during drying (50º - 60 ºC) of blueberries and reported that vitamin C losses at all the drying temperatures. Although total phenol content decreased as air temperature increased, the dehydration at higher temperature retained higher antioxidants. Dahiya and Dhawan [103] studied the effect of different drying methods on the quality of dehydrated aonla. They found that the osmotic-air drying was the best method for retaining nutrients like ascorbic acid and sugars. Also, the minimal browning of product can be obtained using osmotically-air dried product.

2.5 Microwave Hot Air Drying

Microwaves create alternating electric and magnetic field acting perpendicular to each other and works as the driving force for thermal applications. The conversion of microwave energy into heat in the food is because of the dielectric nature of the exposed material [104]. Important research works on microwave drying of lemons and similar products are reviewed in foregoing paragraphs.

Many researchers have proved the combined drying of microwave with conventional hot air drying as one of the good techniques to maintain the quality of final product and minimize the drying time. In conventional hot air drying, extended exposure to higher drying temperature leads to degradation of quality attributes, such as color, flavor, and nutrients [105]. Severe shrinkage also reduces rehydration capacity and bulk density. Even microwave have few limitations like overheating. So there is need of proper combinations of microwaves with the hot air for making it energy efficient process.

During microwave application in hot air drying the microwave power level is one of the most important parameters that affects the drying process and quality. In this type of drying systems the air and microwave combination not only increases the drying rate but also maintains the quality of the dried product obtained. Food materials such as carrots [106], mushroom and apple [107, 108] and potatoes [109] could be dried successfully using hybrid drying technique. In microwave assisted hot-air drying of potatoes and carrots it was reported the drying rate increases with increment of moisture diffusivity, up to a certain value. For a specific drying condition, if the moisture diffusivity is higher than the threshold value then the drying rate remains unchanged. However, by volumetric heating using microwave the moisture diffusivity of the product increases. The use of microwave has showed changes in microscopic and macroscopic structure of the dried product. There was no much changes in the rehydration characteristics of the product. Researchers have concluded that microwaves in convective drying increases the drying rate than the normal rate for convective drying. Also microwave convective drying results in faster drying and less shrinkage, and hence gives a better quality dehydrated product. Pulsed microwave energy can be employed to improve thermal energy utilization as well as quality of heat sensitive dried product [110].

2.5.1 Microwave assisted hot air rotary drying

It is a combination of microwave and hot air in a rotary dryer. The quality of the final product depends upon the microwave power intensity, drum speed and temperature of the hot air. The microwave hot air drier can be used for drying, sterilization, roasting and cooking application. The rotary dryer gives uniformity in heating than static microwave dryer since the particulates moves and mix well as the drum rotates. This overcomes the uneven distribution of the electromagnetic field [111, 112]. The advantages of this type of dryer are moderate capital cost, efficient drying, reduced drying cost, greater retention of flavor and aroma and reduction in one third of the processing time.

2.5.2 Microwave assisted continuous drying

The major components of this equipment are microwave tunnel fitted with magnetrons, a teflon belt to carry the product through the microwave tunnel, microwave source (power supply), belt feeder, processed product collector from belt, air heating system, extractor fans and control units. For safety purpose microwave tunnel inlet and outlet doors are specially designed for electromagnetic shielding to avoid the leakage of microwaves. Many powdered materials like corn flour, ground spices having moisture less than 10 per cent can be rapidly dried by this type of dryer to decrease the moisture content up to 2-3 %.

2.6 Infrared Hot Air Drying

Synergistic effect of infrared - hot air drying results in rapid heating of product with higher rate of mass transfer. The combined drying reduces the processing time, in addition to less energy consumption for water evaporation compared to hot air drying [113]. The drying kinetics depends on the distance between the infrared lamp and the product, and the air velocity. The air velocity should be considered, since air flowing over the surface of the lamp cools down the emitter and thereby lowers its temperature [114]. Similarly, researchers studied the combined effect of infrared and hot air on drying of onion slices by varying air velocity between 1 and 1.5 m s^{-1}, and found that increase in velocity decreases the moisture removal rate [115]. For vegetables, the combined effect of infra-red and hot air rapidly heats the product resulting in a higher mass transfer rate. The combined drying method reduces around 48% of the drying time, in addition to less energy consumption (63%) for water evaporation when compared to conventional hot air drying [113]. Researchers also reported that the combination of infrared and hot-air drying of onion slices at low temperature (60 ºC) with a moderate air velocity (2 m s^{-1}) and air temperature (40 ºC) retains flavor and color [40]. Drying of Jujube (*Zizyphus jujuba*) slices by short and medium wave infra-red drying results in reduced drying time, higher drying efficiency and good quality product [116]. In infrared drying of fruits and vegetables, it has been reported that energy consumption and drying time are dramatically reduced and thereby the quality of the dried product (volatile components, β carotene, flavours, ascorbic acid etc.) can be maintained. Since, infrared radiation are acts mainly on the surface the thickness of the product is a parameter that affect the efficiency of drying. For food products, the suggested thickness should be no more than 5 mm to obtain efficient drying [117]. Likewise the wavelength have a good effect on the product. The heat sink into the product using near infrared is greater compared to the far infra-red [117]. For infrared heating of hamburger patties using mid-infrared and far-infrared, there was change in core and surface temperature using mid infra-red, while the effect of fat content is higher in far-infrared [118]. The penetration of far infrared and near infrared was reported as 0.26 to 0.36 mm and 0.38 to 2.54 mm, respectively. The energy of the far infrared is mostly converted into heat on the surface. For far infrared around 10% of the radiation of reflected back, while in case of near infrared around 50% reflects back. The use of far infrared for fruits and vegetables have been increased in the recent years.

2.7 Combined Drying Process

2.7.1 Osmotic microwave drying

Osmotic dehydration can be done as pre-treatment prior to combined microwave and hot-air drying. Researchers have concluded that the product can be dried with time consumption, along with reduced shrinkages and improved rehydration properties. Few others also reported the presence of impregnated solute in the tissue decreases the water removal rate during microwave finish-drying and hence the effective diffusivity is little lower. But it results in higher rehydration capacity and overall better quality of product [108, 119]. Osmotic solutions formulated with sugar, salt and calcium lactate for osmotic treatment before microwave- hot air drying, provides shelf stable and better quality product than the traditional product [120]. The microwave power, temperature and air velocity also have effect on the finish drying of osmotically dehydrated products. Studies show that while increasing the microwave power in later stage, the rate of drying increases, thus reducing the drying time. However, higher microwave power also leads to charring of the dried product. While air flow cools the surface of the product and maintains product quality by reducing charring. Also the osmotic dehydration can control overheating of center of the spherical or semi-spherical product like mushroom during microwave drying [121].

2.7.2 Infrared - microwave assisted hot air drying

Microwave infrared hot air drying is a new process with good potential to reduce the capital costs. Infrared has several advantages over conventional drying methods. The major advantages are lesser energy consumption, higher drying rate and uniform temperature distribution giving a better quality product. At present, many driers use infrared radiator to improve drying efficiency, save space and provide clean working environment, etc. The penetration power of infrared is lower, so it generally heats product with less thickness rapidly. For products in which infrared penetrates significantly, addition of infrared energy increases the surface moisture build-up. When the infrared energy is mostly absorbed on the surface, it can reduce surface moisture drastically, beyond a threshold power level, infrared can even reduce the surface moisture lower than its initial value [122]. Hot air drying can also reduce the surface moisture, but not as effectively as infrared heating, perhaps due to the lower heat flux on surface for hot air compared to infrared. Combinations of infrared heating with other conductive, radiative and convective modes of heating have been gaining importance because of increased energy throughput [41]. The penetration depth of infrared influences the level of surface moisture that builds up over time or rise of surface temperature. This hybrid technique of microwave infrared serves as a promising energy conserving method of drying as microwave control internal mass transfer and infrared control surface mass transfer.

2.7.3 Changes in quality of electromagnetic assisted hybrid dehydrated agricultural products

The food quality includes desirable properties, nutritional value, its acceptability and safety. Acceptability includes large stream like visual appeal, aroma, flavour, texture, mouth feel, convenience and cultural appropriateness. Some of these can be maintained by the application of microwave on food materials [123].

Color

The color of the food product is one of the most important quality factors and plays a significant role in appearance, processing and acceptability of food materials by the consumers. It is observed as part of the total appearance, which is the visual recognition and assessment of the surface and subsurface properties of the food material [124]. The color change of food materials during thermal processing takes place because of the reactions inside the food materials like pigment degradation, especially carotenoids and chlorophyll, and browning reactions such as maillard reaction, oxidation, etc. [125-127]. Many research works have been published on assessment of color of various food materials like apple, carrot, garlic, cranberries, maize during hybrid drying. The microwave and infrared power level has pronounced effect on the color of the food materials than the temperature effect. The food products treated under microwave radiation at half the power level retains good color than those dried at maximum microwave power [106, 115]. It was also reported that the materials dried using microwave-infrared have good color retention compared to the hot air conventional drying. Researchers have concluded that there is no much color change when extreme dosage of microwave power and longer treatment time are avoided [119].

Rehydration characteristics

Microwave-infrared drying helps in increasing the rehydration rate of the product. The faster rehydration rate is seen due to the large pores formed by puffing effect during dielectric heating which becomes available for water absorption during rehydration. Also many times microwave dried product results in to higher rehydration compared to other methods. Lin et al. [128] dried the carrot slices by air, microwave vacuum-freeze drying methods and found that the microwave vacuum dried carrots shows higher rehydration ratio than air dried and lower than freeze dried carrots. They concluded that during the microwave vacuum drying the high internal vapor pressure produced by microwave heating and the low chamber pressure provided by vacuum caused the structure of carrot slices to expand and puff. Due to this puffing effect a less dense structure has higher capacity to absorb water when reconstituted. Durance and Wang [129] also reported that microwave-vacuum dehydration causes puffing of the tomato tissue such that the dry product was less than half as dense as air-dried tomatoes and rehydrated more quickly and more completely than the air dried product.

Sensory characteristics

The sensory characteristics mainly the odour of the processed food is considered to be very important for its acceptability and a slight change in the odour of food may affect the consumer appeal. Researchers have studied the sensory attributes of electromagnetic assisted dried products like carrots, garlic, coriander, mint, fenugreek, amaranth and shepu. They reported that microwave assisted hot air drying retain odour better than hot air drying alone. It is also found suitable for amaranth, shepu and fenugreek [128, 130]. The sensory quality of the microwave/ infrared treated product is generally better than the hot air conventional method. The flavour and aroma retention in microwave-infrared treated products is almost equal to the freeze dried product. In green tea processing, the infrared treated and dried teas have highest levels of total phenols and catechins, also taste is pleasant and subtle in odour [131]. Warchalewski et al. [104] studied the microwave treatment on grains and reported that roasted smell is produced during longer time exposure to microwaves but it can be avoided when the exposure time is reduced.

Structural and textural properties

Structural and textural properties are important for the characterization of the quality of a dehydrated product. These properties include the gelatinisation of starch, bulk density, true density, porosity, specific volume and viscoelastic behavior of the materials. Porosity characterises is the state of being porous or the overall open structure of a material. These properties are strongly affected by electromagnetic radiation during drying and other conditions [97].

The gelatinization changes of the product are also dependent on the moisture of the product. Studies shows that the extent of the changes is greater when the moisture content of the microwave-treated starch was greater. The gelatinization level of microwave treated samples was similar to the hot air treated ones [132]. Wheat and corn starches of an intermediate moisture content (30%) subjected to dielectric show alteration of their physico-chemical properties and structure, while waxy corn starch remains almost unchanged. More rigid and firmer structure is obtained at higher air temperature or when applying microwave. Less case hardening is observed in fruits when the product is subjected to dielectric heating. Both continuous and pulsed microwave show lesser hardness in cranberries than those dehydrated by hot air [128, 133].

Nutritional characteristics

Some studied are available related to effect of microwave treatment on protein, gluten, starch and vitamins. Protein content was assessed in food materials like wheat, maize, rice after dielectric heating by few researchers. The protein content for wheat sample does not change and it is same as conventional heated sample. Even in rice and maize the protein content is not much altered. But the gluten content is altered by electromagnetic waves. The gluten loses its stretch-ability and elasticity. There were structural changes in the starch content of the sample and also the microwave treatment increased the damaged starch content. Microwave treatment does not affect the cooking and processing quality of the rice [134, 135]. Food materials dried under microwave conditions show lower vitamin C degradation as compared to conventionally dried materials. Samples dried under microwave conditions retain a much greater concentration of ascorbic acid as compared with air dried samples. Microwave treated samples have double times greater retention of vitamins than the air dried samples. Microwave vacuum dried and microwave freeze dried products have better retention of the vitamins and nutritional components [136]. Apricots dried in microwave drier show higher vitamin A, C and E than the infrared drier [137]. Lin et al. [128] compared the retention of β -carotene and ascorbic acid in the carrots dried by different techniques like air drying, freeze drying, microwave vacuum drying and found that the microwave vacuum dried carrots retained the β-carotene and ascorbic acid more than air dried and less than freeze dried. Cui et al. [133] evaluated the carotenoids retention of carrot slices dried by two methods namely, microwave vacuum and microwave convective methods and compared with those dried by freeze-drying and conventional hot-air drying. Their results showed that the carotenoids retention of carrot slices dried by the microwave assisted drying methods, is very close to or equal to those dried by freeze-drying and much better than those dried by conventional hot air.

2.7.4 Factors affecting dielectric heating

Dielectric properties of materials being heated

The heat produced by electromagnetic is determined by the dielectric properties of the exposed material. The interaction between the electromagnetic field and dielectric material is determined by the electrical parameter, complex permittivity is given in below equation

$$\varepsilon = \varepsilon' - j\varepsilon'' \qquad \text{….. (2.1)}$$

where, ε' is the dielectric constant (real part) and ε" is the dielectric constant (imaginary part). Loss tangent is the material's vulnerability to be penetrated by an electrical field and dissipate electrical energy as heat which is given as the ratio of the dielectric loss to the dielectric constant [39].

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} \qquad \text{…. (2.2)}$$

The ability of the material to absorb electromagnetic wave depends upon the loss factor. Highly lossy materials can absorb microwave energy efficiently while the materials that are transparent like teflon, glass have low loss factor. The dielectric constant represents the ability of a material to store the energy in response to an applied electric field while the loss factor determines energy dissipated in the form of heat. From the equation 2.2 and penetration depth it is seen that the dielectric properties mainly depend upon the moisture and temperature.

Moisture content of the material

The dielectric properties of the food material are mainly influenced by moisture content of the product. Generally, the dielectric property of the exposed material decreases as the moisture content reduces below the critical moisture level. The initial moisture content of the material plays an important role in dielectric heating. The heating behavior of water is dependent on phase and the available free water content. Phase change results in change of dielectric properties. The bound moisture has a hindered ability to rotate along the electromagnetic field due to the immobility of water dipoles. Thereby the ability of the field to extract energy reduces. Researchers have noticed that at initial stage of microwave drying when moisture content is relatively high, heating occurs due to ionic conduction as well as water dispersion. But as the moisture level decreases the ionic conduction dominates the frequency response of the dielectric materials [138].

Temperature of the material

Microwave heating is highly influenced by the product temperature. Both the temperature and moisture have a combined effect on the dielectric heating. The initial temperature of the food product exposed to dielectric heating should either be controlled or known, so that the microwave power can be adjusted to obtain uniform final temperature. The effect of higher initial temperature can be compensated by reducing the power of microwave oven or higher initial mass can be achieved.

Many research works have been done to determine the effect of temperature on dielectric constant and dielectric loss factor. As the temperature increases, the dielectric constant decreases. In few reports it was also mentioned that the dielectric constant increases with temperature (from 20 ° to 65 °C) and gradually becomes constant from 60 ° to 95 °C. The loss factor increases linearly with temperature when ion (salt) concentration is high and it decreases with low or no ion concentration. The loss factor was also found to change quadratically with increased temperature that is first decreasing and then increasing. It was found that loss factor depends on the frequency of the microwave. At a frequency of 915 MHz it increases with temperature but at 2450 MHz the loss factor initially decreases with increase in temperature until reaching a minimum at temperatures between 25° and 75 °C [139, 140].

Shape and size of the foods

The impact of product shape and size is often not considered compared to the attention given to the dielectric properties. Feng et al. [138] discussed the power absorption by different mechanisms in food products. The microwave decays when it travels into the product and the surface of the product experiences more microwave radiation than the center part resulting in surface overheating. The decay of microwaves from both sides of a material may form a central overhead area by super position. Foods with flat geometry are difficult to heat due to overheating of corners and edges. These geometries affect the distribution of energy so that it concentrates around the corner or edge and the center area tends to remain cold. This creates the cold spots and brings out the food safety issue [138].

In case of cylindrical and spherical foods the microwaves may concentrate at the centre. Therefore, products like potato cylinders get puffed during microwave drying. This focusing phenomenon does not occur with flat, rectilinear geometries. If the depth of penetration is small compared with the dimensions of material exposed to the microwave, most of the energy is absorbed near the surface leaving the centre cold. If penetration depth is intermediate, reasonable amounts of energy reach the centre and focusing occurs. As a result cooking of eggs in microwave oven will explode. The power density near the centre of the egg is much higher than in other parts and this cause violent explosion as the interior becomes superheated [141].

Mass to power ratio

The amount of power absorbed has a direct relationship with the mass of the product exposed. Many researchers mention the power density in kW per kg of product. As the power density is increased the heating rate increases and accelerates the drying rate [142]. Also the studies states that for a smaller mass batch type oven will be appropriate, while a larger throughput would be required in large capacity conveyorised equipment [143].

2.8 Sorption Isotherm

The water sorption isotherms represent the equilibrium relationship between the moisture content of foods and the water activity at constant relative humidity, temperature and pressure. At high humidity and temperature accelerated condition it is assumed that water and oxygen are the reactants that are causing failure. However there are other trace substances in the atmosphere, particularly air pollutants that may cause degradation. Only with an understanding of the kinetics of degradation and mass transfer can rational models be developed for lifetime prediction [144]. In accelerated shelf-life testing (ASLT) the products are stored under controlled conditions designed to accelerate the deterioration rate of the product. The deterioration rate can be further related to storage of the product at ambient conditions, and it can be used to predict the shelf life at different storage conditions [145]. The accelerated tests assume that the deteriorative processes will fit a kinetic model [146-148]. Researchers found that there was insignificant effect of temperature on the equilibrium sorption. Generally, Guggenheim, Anderson and de Boer (GAB) model shows better fit than for the moisture sorption isotherm of food products [149, 150].

Chapter 3

Materials and Methods

3.1 Raw Materials

Fresh lemons of same maturity level and approximately same size were procured from the same supplier in the market throughout the experimentation to minimize the deviation in moisture content and other physico-chemical properties. Lemons were washed with water to remove the adhering debris and foreign matter. The thickness and diameter of the lemon slices were maintained at 5 ± 0.2 and 36.5 ± 8 mm, respectively. The moisture content of each lemon was measured separately. The moisture content was determined by Association of Official Analytical Chemists (AOAC) method [151] and the mean initial moisture content was found to be 87.35 ± 1.09% (wet basis). Food grade NaCl (common salt) was used as an osmotic agent. Calcium chloride and ascorbic acid was used for chemical pre-treatments.

3.2 Osmotic Dehydration

3.2.1 Osmotic dehydration experiments

The unpeeled lemon slices were osmotically dehydrated in solutions containing 5, 10, 15 and 20% of NaCl (w/v) at 30, 40 and 50 °C for 30, 60, 90, 120, 150 and 180 min. For all experiments, the sample to solution ratio was maintained at 1:10 (w/v). The experiments were performed without external agitation to avoid additional cost of processing. From each lemon, one slice was used for initial moisture content determination and the other slices for osmotic dehydration experiment. The same value of initial moisture content was used for determination of moisture loss and solid gain for the slices used from those lemons. At a particular combination of concentration and temperature, 6 separate beakers were used for each time interval (30-180 min). One lemon slice was treated in each beaker with proper identification. At the end of the dehydration time, the lemon slices were removed from the solution, rinsed with distilled water, blotted to remove the adhering solute and water present on the surface. At each designated time intervals the lemon slices were analysed for moisture loss and solid gain using oven. Further, oven dried samples were analysed for actual salt gain to estimate the solid loss. The water loss and salt gain with and without solid loss consideration were calculated for all samples. Temperature uniformity of osmotic solution was assured by placing the treatment beaker in a constant temperature water bath. Blank treatment was done by dipping the lemon slices in distilled water without the addition of salt at 30, 40 and 50 °C.

3.2.2 Salt content analysis

The actual salt gain considering solid loss was estimated by determining the NaCl content in osmotically dehydrated samples by AOAC [151], Mohr's titration method. After the determination of moisture content the samples were converted into ash in muffle furnace at 550° C for a period of 12 hours. The ash was dissolved in a known volume of water and filtered to obtain the water soluble ash. Then the chloride ions were titrated against silver nitrate in the presence of potassium chromate until the appearance of light reddish brown color [152].

3.2.3 Modelling mass transfer kinetics

Osmotic dehydration process involves loss of moisture, gain of salt and loss of solids and juice sacs in citrus fruits. A two parameter equation, developed by Azuara, was used to predict the mass transfer kinetics of osmotic dehydration and the final equilibrium point.

Osmotic pressure

Osmotic pressure (π) is the driving force for osmotic dehydration. Osmotic pressure in lemon slices arises due to the difference in concentration between the salt solution and the water inside the sacs which is separated by the semi-permeable membrane (peel and tissues). It is the minimum pressure required for movement of water or solutes through the tissue membrane. The equation for determination of osmotic pressure is given below:

$$\pi = \mathrm{CRT} \qquad \text{….. (3.1)}$$

where π is the osmotic pressure (atm), C is the molar concentration of the solution (mol L^{-1}), R is the ideal gas constant (0.08206 L atm mol^{-1} K^{-1}) and T is the absolute temperature (K).

Moisture loss and salt gain without solid loss consideration

The moisture loss (ML_{wsl}, % initial moisture, IM) and the salt gain (SG_{wsl}, % initial dry matter, db) were calculated by mass balances [142, 153, 154]. The moisture loss was calculated using following equation:

$$ML_{wsl} = \frac{M_i - M_f}{M_i} \times 100 \qquad \text{….. (3.2)}$$

where, M_i and M_f are the moisture (g) present in the sample before and after osmotic dehydration, respectively.

Similarly, the difference between the initial and final dry matter content is considered as the salt gain after osmotic dehydration when solid loss is neglected and hence the SG_{wsl} without solid loss consideration was estimated using following equation:

$$SG_{wsl} = \frac{DM_f - DM_i}{DM_i} \times 100 \qquad \text{….. (3.3)}$$

where, DM_i is the dry matter content (g) without salt before osmotic dehydration, and DM_f is the dry matter content (g) with salt after osmotic dehydration.

In traditional method of osmotic dehydration, solid loss is considered negligible and the moisture loss and solid gain are calculated based on mass balance. This assumption generates error in calculation of mass transfer during osmotic dehydration. Hence, the moisture loss and solid gain without solid loss consideration was determined to indicate the error and compare it with the exact mass transfer (calculated with solid loss consideration) occurring during osmotic dehydration.

Solid loss

Solid loss (SL) is the loss of dry matter content (g) in the osmotic solution and juice sacs losses is the dry matter loss along with moisture in the form of sacs (g). Since solid loss was predominant during osmotic dehydration of lemon slices the general mass balance does not give the exact ML and SG. Therefore, solid loss was determined after experimental determination of NaCl content in the osmotically dehydrated sample. The exact salt gain due to osmosis (SG_{os}) was determined by estimating the NaCl content of oven dried material using Mohr's method, and thereby the SL during osmosis was found using following equation:

$$DM_f = DM_i + SG_{os} - SL \qquad \text{….. (3.4)}$$

During the osmotic dehydration process, the change in dry matter content occurs due to loss of solids.

Moisture loss with solid loss consideration

Moisture loss with solid loss consideration is the sum of ML_{os} (only due to osmosis) and ML_{sl} (moisture lost along with solid loss) in the form of whole juice sacs. To estimate the exact ML_{os}, the ML_{sl} was estimated first from assumption that the solids carry away the same proportion (ratio of moisture to solid in tissue) of moisture during leaching. Further, the ML_{sl} was excluded from the total moisture loss to represent the moisture loss occurring only due to osmosis (ML_{os}).

Dry matter holding capacity (DHC) of lemon slices

DHC index is an assessment for the ability of fruit to retain its solid content during osmotic dehydration. Lemon is highly susceptible to leaching, hence the DHC measurement is necessary to quantify the percentage of solid loss. The DHC (%) varies depending on the nature of the fruit, tissues cohesive nature and mechanical damage during slicing [155]. It was calculated using following equation:

$$DHC = \frac{W_f S_f}{W_i S_i} \times 100 \qquad \text{….. (3.5)}$$

where, W_f is weight of lemon sample (g) after dipping in blank solution, S_f is the percent of dry matter (lemon's solid) retained after dipping, W_i is weight of lemon sample (g) before dipping in blank solution and S_i is the percent of dry matter (lemon's solid) before dipping. The rate at which the lemon slice loses its capacity to withhold the dry matter was determined by the following linear equation for the time ranging from 10 – 180 min.

$$DHC = -a_1\theta + a_2 \qquad \text{….. (3.6)}$$

where a_1 is the rate at which the lemon loses its capacity to withhold the integrity, a_2 is the constant and θ is the time of treatment. The negative sign in Eq. (3.6) is due to the solid loss. The DHC was estimated by immersing the lemon slices in water without the addition of salt at 30, 40 and 50 °C with time interval same as osmotic dehydration experiment.

Moisture loss, salt gain and solid loss kinetics

Peleg (1988) [72] proposed a two parameter sorption equation and predicted the mass fluxes during sorption of various food materials. The model was also used to determine the ML and SG kinetics during osmotic dehydration. The two parameter equation is mentioned as follows:

$$M = M_0 \pm \frac{\theta}{K_1 + K_2\theta} \qquad \text{….. (3.7)}$$

where K_1 gives the initial rate of mass transfer and K_2 corresponds to the minimum attainable moisture content (Peleg capacity constant). The sorption rate (R) can be obtained by first derivative of Eq. (3.7).

$$R = \frac{dM}{d\theta} = \pm \frac{K_1}{(K_1 + K_2\theta)^2} \quad \text{….. (3.8)}$$

At the very beginning when t = t_0, Eq. (3.8) becomes

$$R_0 = \frac{dM}{d\theta} = \pm \frac{1}{K_1} \quad \text{….. (3.9)}$$

The Peleg capacity constant gives the ML or SG at infinite time i.e. equilibrium moisture content. When t = ∞, Eq. (3.10) gives the relation between equilibrium moisture content and K_2

$$M = M_\infty = M_0 \pm \frac{1}{K_2} \quad \text{….. (3.10)}$$

Further the Peleg constants were explained by Azuara [69] as, $K_2 = \frac{1}{ML_\infty}$ or $\frac{1}{SG_\infty}$ and the constant $K_1 = \frac{1}{S_1\,(ML_\infty)}$ or $\frac{1}{S_2\,(SG_\infty)}$. Thus, the equations were derived by Azuara as

$$\frac{\theta}{ML_\theta} = \frac{1}{S_1(ML_\infty)} + \frac{\theta}{ML_\infty} \quad \text{…... (3.11)}$$

$$\frac{\theta}{SG_\theta} = \frac{1}{S_2(SG_\infty)} + \frac{\theta}{SG_\infty} \quad \text{…... (3.12)}$$

where ML_θ and SG_θ are the moisture loss and salt gain at any time, respectively. ML_∞ is the moisture loss at equilibrium and SG_∞ is the salt gain at equilibrium, S_1 and S_2 are the constants related to moisture loss and salt gain during osmotic dehydration. Similarly, SL_θ can be expressed as:

$$\frac{\theta}{SL_\theta} = \frac{1}{S_3(SL_\infty)} + \frac{\theta}{SL_\infty} \quad \text{…... (3.13)}$$

where SL_θ is the solid loss at any time and SL_∞ is the solid loss at equilibrium. S_3 is the constant related to solid loss. The fitting ability of the model was determined using Root-Mean-Square Error (RMSE). The lower RMSE value shows the better fit of the model.

$$RMSE = \sqrt{\sum_{i=1}^{n} \left(\frac{\left(X_p - X_a\right)^2}{n} \right)} \quad \text{…... (3.14)}$$

where X is the variable for ML, SG and SL, the subscripts p and a denote predicted and actual values, respectively and n is the number of data.

Statistical analysis using multiple regression on ML, SG and SL

A multiple regression equation was used to determine the effect of different variables of osmotic solution on ML (%IM), SG (% db) and SL (%db) [142, 149]. Hence, regression was done using Regression Analysis Tool in Microsoft Excel (2013). The correlations were developed considering all the factors and are given as:

$$ML_\theta = a_0\, \theta^{a_1}\, C^{a_2}\, T^{a_3}\, SL_\theta^{a_4}\, SG_\theta^{a_5} \qquad \text{…... (3.15)}$$

$$SG_\theta = b_0\, \theta^{b_1} C^{b_2} T^{b_3}\, SL_\theta^{b_4} \qquad \text{…... (3.16)}$$

$$SL_\theta = c_0\, \theta^{c_1}\, C^{c_2}\, T^{c_3} \qquad \text{…... (3.17)}$$

The coefficients were found by multiple regression. The above equations gives the combined effect of the various parameters on the mass transfer kinetics during osmotic dehydration. The significance of the individual parameters and the interactive effect of the variables were checked.

3.3 Chemical and Thermal Pretreatments

3.3.1 Pretreatment experiments

To inactivate the enzymes and retard the deterioration of the lemon slices few pre-treatments were performed. They were chemical treatment (ascorbic acid and calcium chloride), ultrasound, water blanching and steam blanching. The variables and levels adopted for pretreatments are represented in Table 3.1. The levels of the process variables were determined after several preliminary trials.

For chemical pretreatment, Box Behnken Design was employed to evaluate the effect of different independent parameters such as chemical concentration, temperature and time of treatment. The independent variables with the actual and coded values considered in this study are given in Table 3.2.

Table 3.1 Variables and levels of pretreatments for lemon slices

Pretreatment	Independent Variables	Levels
Osmotic Pre-concentration	Solution Temperature, °C	20-55
	Salt Concentration, % w/v	5-20
	Time of osmosis, min	10-180
Ascorbic Acid	Concentration %, w/v	0.1 to 0.5
	Dipping Time, min	1-10
	Solution Temperature, °C	20-80
Calcium Chloride	Concentration %, w/v	0.1 to 0.5
	Dipping Time, min	1-5
	Solution Temperature, °C	20-80
Ultrasound treatment	Frequency, kHz	20
	Time of application, min	1-5
	Initial water temperature, °C	20-80
Water Blanching	Dipping Time, min	1-5
	Solution Temperature, °C	60-90
Steam Blanching	Steam Exposure Time, min	1-5

Table 3.2 Actual values for different parameters during chemical pre-treatment

Independent variables		Levels		
		-1	0	+1
Chemical Treatment				
a.	Ascorbic acid (w/v)	0.1	0.3	0.5
b.	Calcium chloride (w/v)	0.1	0.3	0.5
c.	Time (min)	1	3	5
d.	Temperature (°C)	20	50	80

For ultrasound, water blanching (4 experiments) and steam blanching (2 experiments) completely randomized design was used. The response variables for all the pre-treatments were ascorbic acid content, pectinesterase and peroxidase activity, and solid loss during treatment.

3.3.2 Determination of ascorbic acid content

Ascorbic acid content was determined by indophenol dye titrimetric method based on AOAC [151]. In this method, ascorbic acid reduces 2, 6 dichlorophenol indophenol dye to a colourless leuco – base. The ascorbic acid gets oxidised to dehydroascorbic acid. At the end point the excess unreduced dye turns into pink solution in acid medium. The titration was performed in the presence of metaphosphoric acid acetic acid (MPAA) solution to maintain pH and avoid auto-oxidation of ascorbic acid at high pH.

3.3.3 Estimation of enzyme activity in lemon

Extraction of peroxidase and pectinesterase

The sample was mixed with 1M NaCl in the proportion of 1:5 (w/v). It was homogenised under chilled condition for 1 min and the homogenate was centrifuged in polypropylene tubes at 6000 xg at 4°C for 20 min. The supernatants was kept in cold condition until analysis [156].

Pectinesterase estimation

Pectinesterase activity was determined by Hagerman and Austin method [157]. The substrate contained, 5 g/L pectin prepared in buffer solution (100mM sodium chloride and 2mM TRIS-HCl, pH 7.5) and 0.1 mL of 0.01% w/v Bromothymol Blue. The activity was observed in UV-spectrophotometer by mixing 2.7 mL of substrate with the 0.2 mL of extracted enzyme at 620 nm [158].

Peroxidase

The substrate for peroxidase was prepared by mixing 0.1 mL of guaiacol, 0.1 mL of 30% hydrogen peroxide and 99.8 mL of phosphate buffer (0.1 mol/L, pH 6.5). The assays was prepared by pipetting 0.12 mL of enzyme extract and 3.48 mL of substrate solution in a quartz cuvette. The absorbance was measured at 470 nm using a UV spectrophotometer for 10 min [159].

3.3.4 Estimation of solid loss during pretreatments

The solid loss during each pre-treatment was determined by the DHC as discussed in osmotic pretreatment section. The effect of temperature was found based on the loss in dry matter during the treatment period.

3.4 Drying of Pretreated Lemon

Osmotically pre-treated samples (30 °C, 20% NaCl concentration for 180 min) were further steam blanched (1 min) to inactivate the enzymes and remove the entrapped air. The pre-treated lemon slices were dried using the combination of infrared hot air and microwave hot air. The optimized condition for infrared hot air drying was selected and further proceeded for microwave hot air drying.

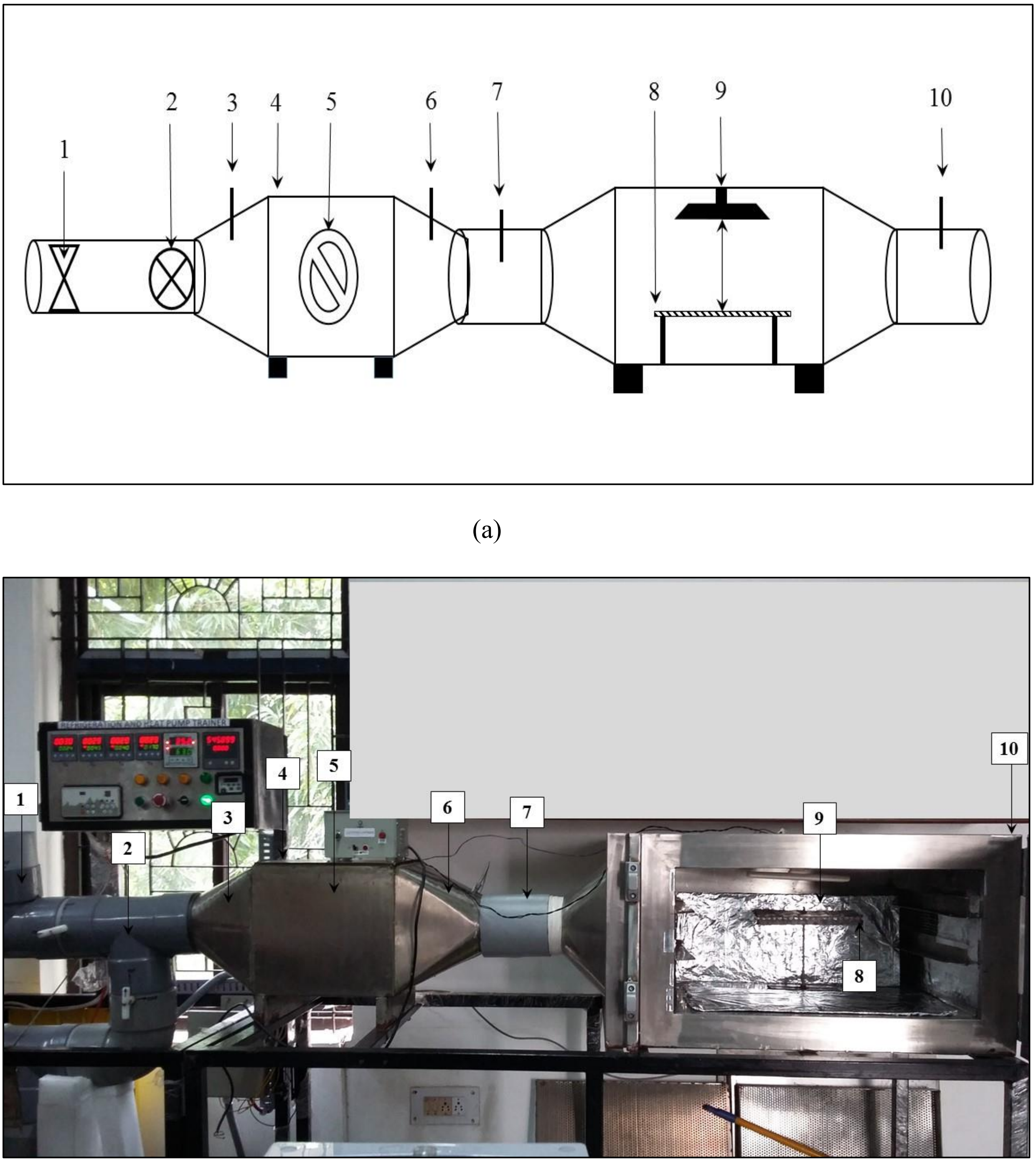
3.4.1 Infrared hot air drying

The schematic diagram of the experimental setup with all accessories is shown in Figure 3.1. The set-up for infrared hot air drier consisted of blower to provide the inlet air and heating arrangement to heat the inlet air. Two separate temperature sensors were inserted in the inlet and outlet of the heating chamber and the air flow was adjusted using the air flow control valve. The infrared heating chamber consisted of 1 kW infrared lamp (FSC longwave IR lamp 8 to 15 μm, 1000W) and a perforated stand with height adjustment. The samples were uniformly spread in a single layer on the stand. On the other side of the infrared chamber air exit was provided and the temperature and RH was monitored using the sensors.

Hybrid design based on Roquemore [160] was used to determine the relative contributions of four variables such as radiation intensity, air temperature, distance and velocity in the range 3000-5000 Wm^{-2}, 50-90 °C, 100-200 mm and 0.5 to 1.5 ms^{-1}, respectively. Fifteen experiments were performed. Actual values of the variables with respect to code values for each combination are given in Table 3.3.

Table 3.3 Actual values of the variables used during infrared hot air drying

Independent variables		**Levels**				
		- α	**-1**	**0**	**+1**	**+ α**
a.	Radiation intensity (Wm^{-2})	2482.30	3000	4000	5000	5517.70
b.	Air temperature (°C)	39.65	50	70	90	100.35
c.	Distance (mm)	74.11	100	150	200	225.88
d.	Velocity (ms^{-1})	0.48	0.5	1.00	1.5	1.87



(a)

(b)

1. Air blow system 2. Air flow control valve 3. Inlet air temperature sensor (K-type thermocouple) 4. Air heating chamber 5. Air Heater 6. Outlet air temperature sensor (K-type thermocouple) 7. Anemometer for air velocity measurement 8. Perforated height adjustable sample keeping arrangement 9. Infrared lamp (FSC longwave IR lamp, 1000 W, 230 V) 10. Air temperature and RH sensor at exit

Figure 3.1 Schematic diagram (a) and image (b) of infrared-hot air drying system

The dependent variables for infrared hot air drying were drying time and drying kinetics. The best condition was selected based on statistical analysis (Analysis of Variance-ANOVA) with target of minimum drying time with respect to drying rate pattern. The selected condition was used for finish microwave hot air drying.

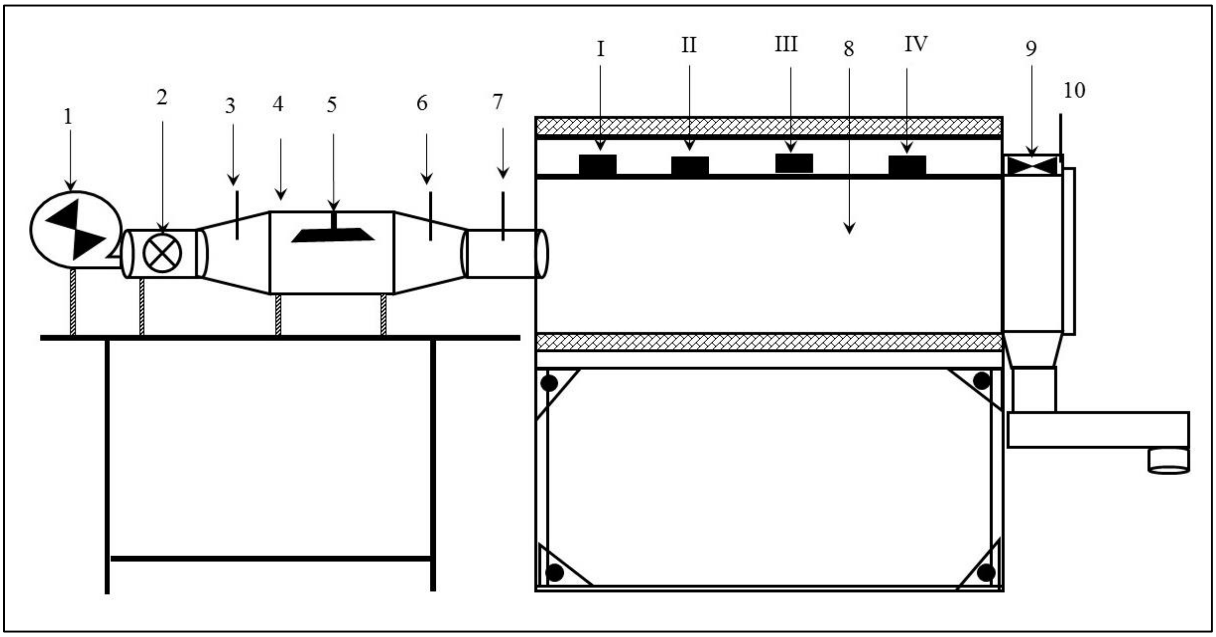
3.4.2 Microwave hot air drying

The experimental set –up consisted of two sections, air heating section and microwave drying chamber where the samples were placed on a perforated tray. The schematic diagram of drying arrangement is shown in Fig 3.2. In air heating section, the air inlet was provided through a blower, a valve was used to control the velocity of the inlet air and the inlet temperature was monitored using a temperature sensor. In the air heating chamber an IR lamp was provided to heat the inlet air and the temperature was measured using a sensor at the outlet of the heating chamber. The heating section was connected to the microwave drying chamber. In microwave drying chamber, four magnetrons were fitted (each 1 KW). An exhaust fan was provided on the outlet of the microwave cavity.

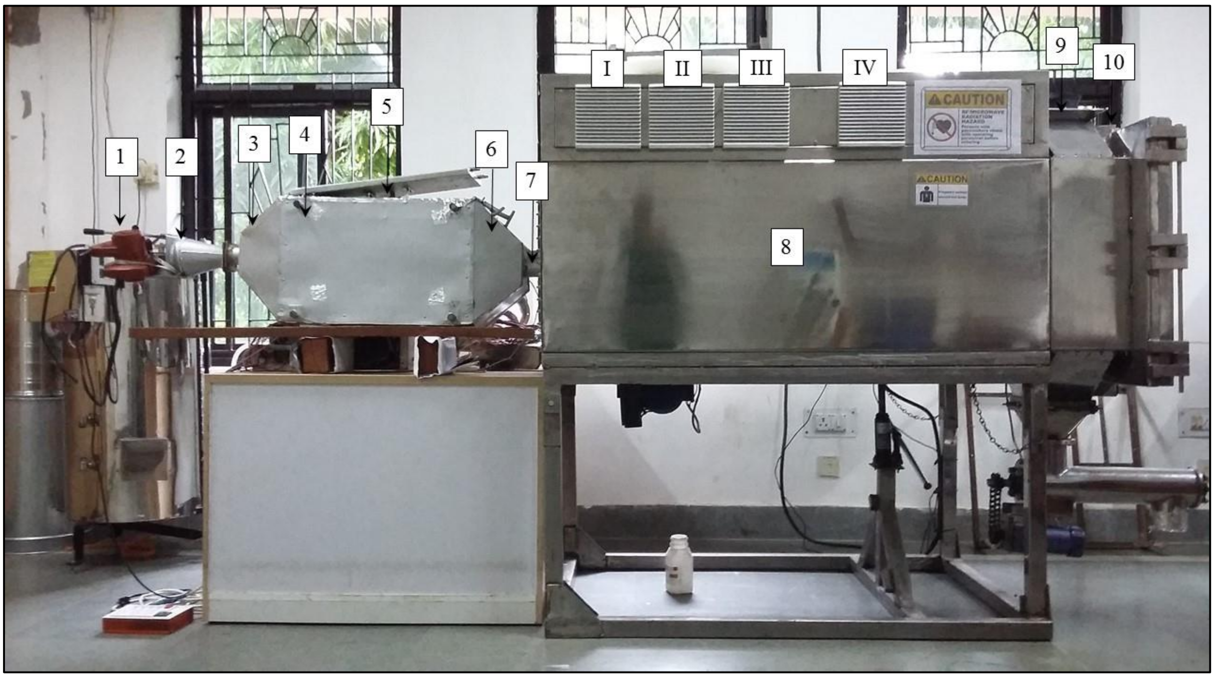
The microwave hot drying of lemon slices was carried out with hybrid design as suggested by Roquemore [160]. After infrared - hot air drying (optimized condition) the microwave hot air was used for finish drying to reduce moisture in lemon slices from approx. 30% (w.b) to 9-10% (wb)). The experimental plan with 11 experiments was used with varying power density, air temperature and air velocity. The levels of the process variables were determined after several preliminary trials, keeping the attention on the product quality. The actual and code values are represented in Table 3.4.

Table 3.4 Actual values of the variables used during microwave hot air drying

Independent variables		**Levels**				
		- α	**-1**	**0**	**+1**	**+ α**
a.	Power density (Wg^{-1})	0.05148	0.3	0.9	1.5	1.74852
b.	Air temperature (°C)	41.716	50	70	90	98.284
d.	Velocity (ms^{-1})	0.2929	0.64645	1	1.35355	1.7071



(a)



(b)

1. Air blow system (13000 rpm, 230 V, 440 W, JKBL-Air Blower) 2. Airflow control valve 3. Inlet air temperature sensor (K-type thermocouple) 4. Air heating chamber 5. IR lamp (FSC IR lamp, 1000 W, 230 V) for air heating 6. Outlet air temperature sensor (K-type thermocouple) 7. Anemometer for air velocity measurement 8. Drying chamber 9. Exhaust fan 10. Air temperature and RH sensor at exit; I, II, III and IV- Magnetron (1kW each)

Figure 3.2 Schematic diagram (a) and image (b) of microwave-hot air drying system

3.4.3 Modeling of drying characteristics

In order to study the effect of the process conditions on the drying characteristics, the average drying rates were calculated for moisture removal up to 9-10 % d.b. The average drying rate was calculated for different time intervals during the drying by dividing the amount of moisture removed by time interval and was plotted against the average moisture content of that time interval. The apparent moisture diffusivity was estimated for infrared and microwave hot dried lemon slices. The lemon slice was considered as infinite slab [115] and the apparent moisture diffusivity (D) of infinite slab was estimated by using following equation [161]:

$$\frac{M_t - M_e}{M_o - M_e} = \frac{8}{\pi^2}\exp\left(-\frac{D\pi^2 t}{4l^2}\right) \qquad \dots (3.18)$$

where, M_o is the initial moisture content; M_t is the moisture content at the sample at any time; M_e is the equilibrium moisture content; t is time (min) and l is the half thickness of the slab.

Another simplified approach to predict drying kinetics for the thin layer drying in the falling rate period has been introduced by Lewis [162].Various mathematical models have been developed based on simplified approach and found useful in describing thin layer drying behavior of agricultural materials. Among those, Page model (Eqn. 3.19) has been used to describe the drying kinetics of various agricultural materials in infrared and microwave-convective drying [106, 130, 163]. The model is of the form:

$$MR = \exp(-kt^n) \qquad \dots (3.19)$$

where, drying rate constant (k, min^{-1}) and n are the parameters of the Page model, t is time in min. The above equations were evaluated through non-linear regression analysis. The goodness of fit of the tested mathematical models to the experimental data was evaluated from the commonly used statistical parameters namely; coefficient of determination (R^2) and root mean square error (RMSE) [142].

3.5 Quality Evaluation of Dehydrated Lemon Slices

3.5.1 Rehydration ratio

Rehydration ratio is the ratio of weight of rehydrated sample to the initial weight of the sample. It is a measure of rehydration characteristics of dried lemon slices. It was determined by dipping dehydrated lemon slice in distilled water at 100°C for 10 min. The water was drained and the lemon was blotted with tissue paper to remove the adhering surface moisture. The weight was recorded and the rehydration ratio was determined by following equation [164]:

$$\text{Rehydration ratio} = \frac{W_R}{W_D} \qquad \ldots (3.20)$$

where W_R and W_D are the weight of rehydrated and dehydrated lemon slices respectively.

3.5.2 Colour

The colour of the dehydrated lemon slices was determined by hunter colour lab. Colour difference (ΔE) as described by Eq. 3.21 was used to describe the colour loss of product

$$\Delta E = [(L-L^*)^2 + (a-a^*)^2 + (b-b^*)^2]^{0.5} \qquad \ldots. (3.21)$$

where ΔE indicates the degree of overall colour change of a sample in comparison to colour values of an ideal sample having colour values of L^*, a^* and b^*. Fresh lemon slices were taken as ideal sample.

3.5.3 Sensory evaluation

The sensory evaluation of dried samples was carried out by panel of 10 members. The panelists were given a proforma for sensory evaluation and asked to indicate their preference for each sample based on the quality attributes such as taste and overall acceptability. On 9-point hedonic scale, where 9 denotes "liked extremely" and 1 "disliked extremely" was used for all the attributes evaluated [165].

3.5.4 Specific energy consumption

The specific energy consumption was estimated as described by Sharma and Prasad [130]. The specific energy consumption for drying the osmotically dehydrated lemon slices from 66% to 9% was estimated. Specific energy consumption, H was expressed as follows:

$$H(\text{kJ/kg}) = \frac{\text{Total energy supplied in drying process}}{\text{Amount of water removed during drying}} \qquad \ldots (3.22)$$

$$H(\text{kJ kg}^{-1}) = \frac{h_1 + h_2 + h_3 + h_4}{W_e} \qquad \ldots (3.23)$$

where h_1 is the energy requirement of the magnetron, kJ; h_2 is the energy required by the infrared lamp, kJ; h_3 is the energy required by the heater, kJ; h_4 is the energy required by the blower and W_e is the amount of moisture removed during the drying process, kg.

3.6 Sorption Study of Dehydrated Lemon Slices

Sorption experiments were conducted for dehydrated lemon slices using accelerated storage study. The moisture content of the product at which it is in equilibrium with the atmosphere is known as equilibrium moisture content (EMC). A presentation of EMC at a given temperature versus the equilibrium relative humidity of the surrounding is expressed as a sorption isotherm. The following formula was used to calculate equilibrium relative humidity at particular equilibrium moisture content.

$$\mathrm{ERH}(\%) = a_w \times 100 \qquad \text{…. (3.24)}$$

The level of temperature was selected to simulate the ambient conditions prevailing in most of the parts of India. Saturated solutions were prepared according to the method described by Ranganna [165]. Solutions were kept in the desiccator to maintain the relative humidity at different levels of 40°C temperature [146, 166]. Approximately, 5-10 g of sample was kept in the desiccator for the study. The desiccator was placed inside temperature-controlled chamber. Moisture content and water activity of each sample was estimated after weight loss study.

The GAB model was used to describe relationship between the water activity (a_w) - equilibrium moisture content (X) and shelf life was predicted. The model is given below:

$$X = \frac{MCKa_w}{(1-Ka_w)\times(1-Ka_w+CKa_w)} \qquad \text{… (3.25)}$$

where, X is the moisture content (kg water.kg dry solid^{-1}); a_w is water activity; C, K and M are the GAB constants. M is monolayer moisture content (g g^{-1} dry solids), C is the Guggenheim constant and K is a molecule multilayer factor. The GAB model constants were determined by fitting a second order polynomial type regression equation between a_w/X and a_w as given below [146]:

$$\frac{a_w}{X} = \left[\frac{K}{M}\left(\frac{1}{C}-1\right)\right]a_w^2 + \left[\frac{1}{M}\left(1-\frac{2}{C}\right)\right]a_w + \frac{1}{MCK} \qquad \text{… (3.26)}$$

$$\frac{a_w}{X} = b_1 a_w^2 + b_2 a_w + b_3 \qquad \ldots (3.27)$$

where, $b_1 = \frac{K}{M}\left(\frac{1}{C} - 1\right)$, …(3.28)

$$b_2 = \frac{1}{M}\left(1 - \frac{2}{C}\right) \qquad \ldots (3.29)$$

$$b_3 = \frac{1}{MCK} \qquad \ldots (3.30)$$

The solution for constants are given as follows:

$$K = \frac{-b_2 + \left(b_2^2 - 4b_3 b_1\right)^{0.5}}{2b_3} \qquad \ldots (3.31)$$

$$M = \left(b_2 + 2b_3 K\right)^{-1} \qquad \ldots (3.32)$$

$$C = \left(b_3 MK\right)^{-1} \qquad \ldots (3.33)$$

The shelf life of the product was calculated using equation 3.34.

$$\theta_{ps} = \frac{W_p}{2k_g b_p l_p P_p^*} \int_{X_i}^{X_{pc}} \frac{dX}{R_{hp} - a_w} \qquad \ldots (3.34)$$

where, θ_{ps} is the shelf life, (s); W_p is weight of the product, (kg); k_g is the permeability of packaging material, (kg water $m^{-2}s^{-1}Pa^{-1}$); b_p is width of the package, (m); l_p is length of the package, (m); P_p^* is saturation vapor pressure of water at Tp, (Pa); X_i is the initial moisture content of the product, (kg water kg^{-1} dry solids); X_{pc} is the critical moisture content of the product, (kg water kg^{-1} dry solids); R_{hp} is relative humidity of the storage environment (fraction) and a_w is the water activity of the product at X_{pc}.

Chapter 4

Results and Discussion

4.1 Osmotic Dehydration Pretreatment

4.1.1 Moisture loss and salt gain kinetics with assumption of no solid and juice sacs losses

The major use of Peleg and Azuara model is to reduce the experimentation and time required to predict the equilibrium conditions. Apart from determining the equilibrium values Azuara model was also used to understand the mass transfer during osmotic dehydration. The ML_∞ and SG_∞ were predicted by fitting θ/ML_θ vs θ (Fig. 4.1) and θ/SG_θ vs θ (Fig. 4.2) in Azuara model. . The elevated trend of 5% NaCl may be due to the increased loss of solids during osmotic dehydration. The values of ML_∞ and S_1 for moisture loss kinetics and SG_∞ and S_2 for salt gain kinetics were determined from Eq. (3.11) and (3.12) and it is given in Table 4.1.

Table 4.1 The constants and equilibrium values of Azuara for moisture loss and salt gain without solid loss consideration

Salt concentration (% NaCl)	**ML_∞ (% IM)**	**S_1 (per min)**	**RMSE**	**SG_∞ (% d.b)**	**S_2 (per min)**	**RMSE**
5%	8.74	0.0203	0.279	-2.0653	-0.0361	1.522
10%	8.51	0.031	0.274	5.9488	-0.0856	1.456
15%	11.91	0.0374	0.541	31.8471	0.003	2.793
20%	16.66	0.0218	0.477	42.9184	0.0067	0.339

The ML_∞ (%IM) varied from 8.74 to 16.66 when the concentration was increased from 5 to 20% at 30 °C. Similarly, SG_∞ (%db) varied from -2.2 to 42 % when the concentration was increased from 5 to 20% at 30 °C.

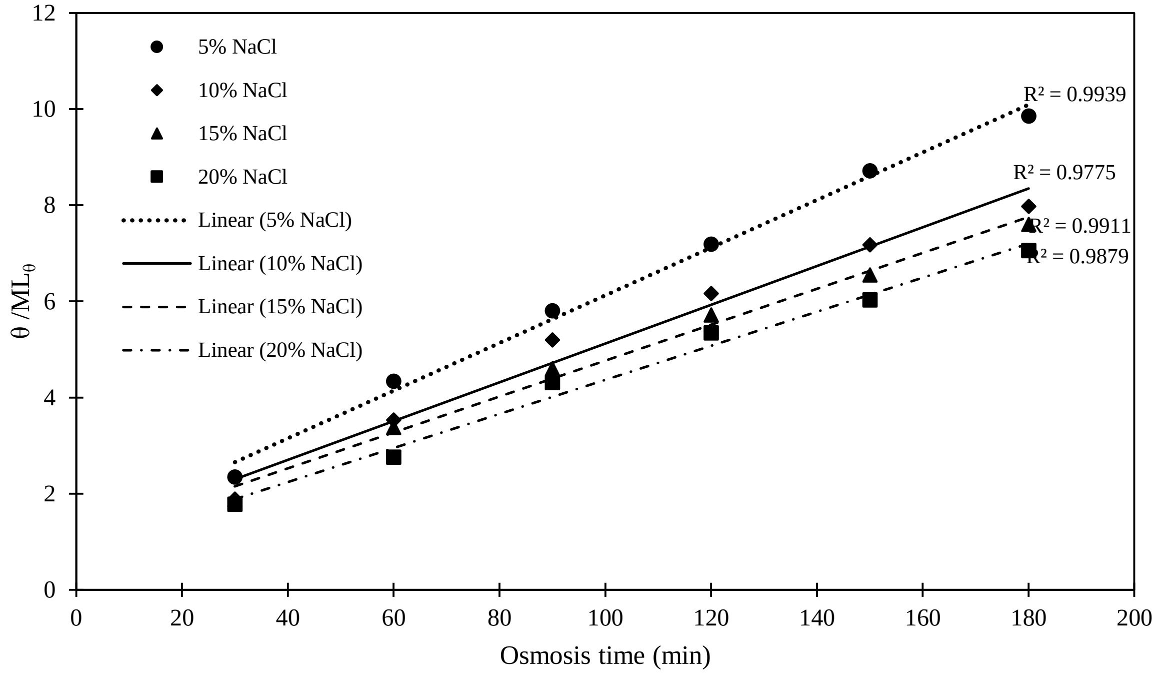


Figure 4.1 Linear plots of Azuara model for determination of ML_∞ and S_1 at different solution concentrations at constant temperature (30 °C)

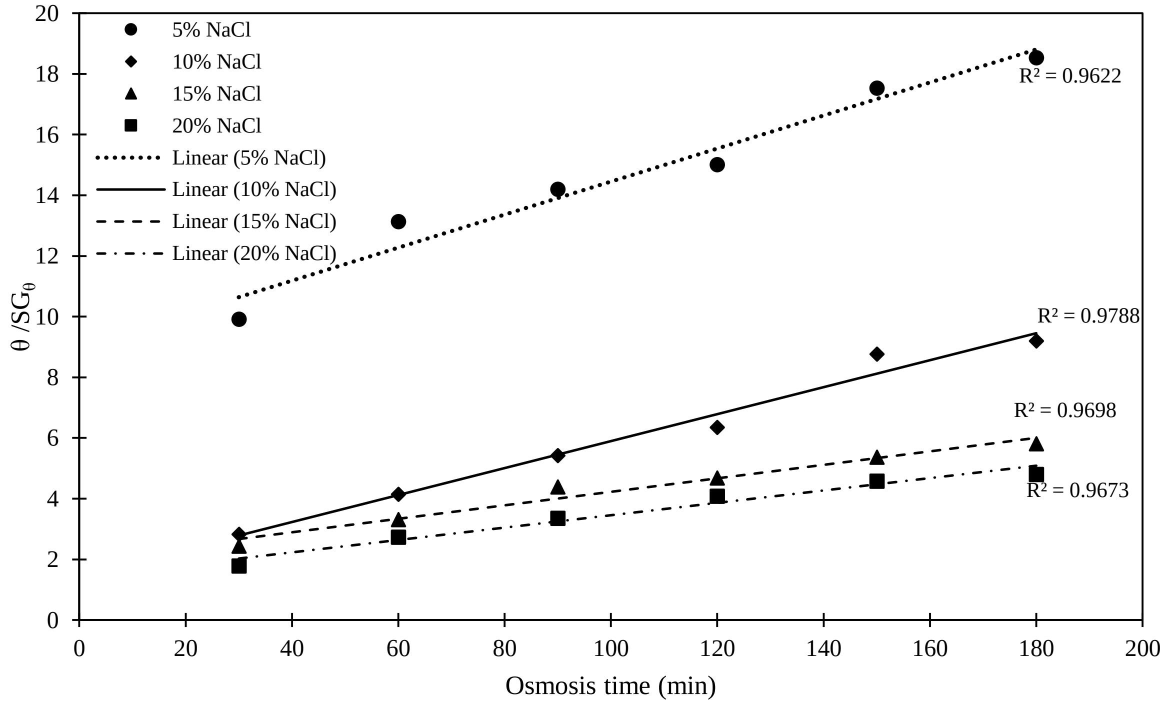


Figure 4.2 Linear plots of Azuara model for determination of SG_∞ and S_2 at different solution concentrations at constant temperature (30 °C)

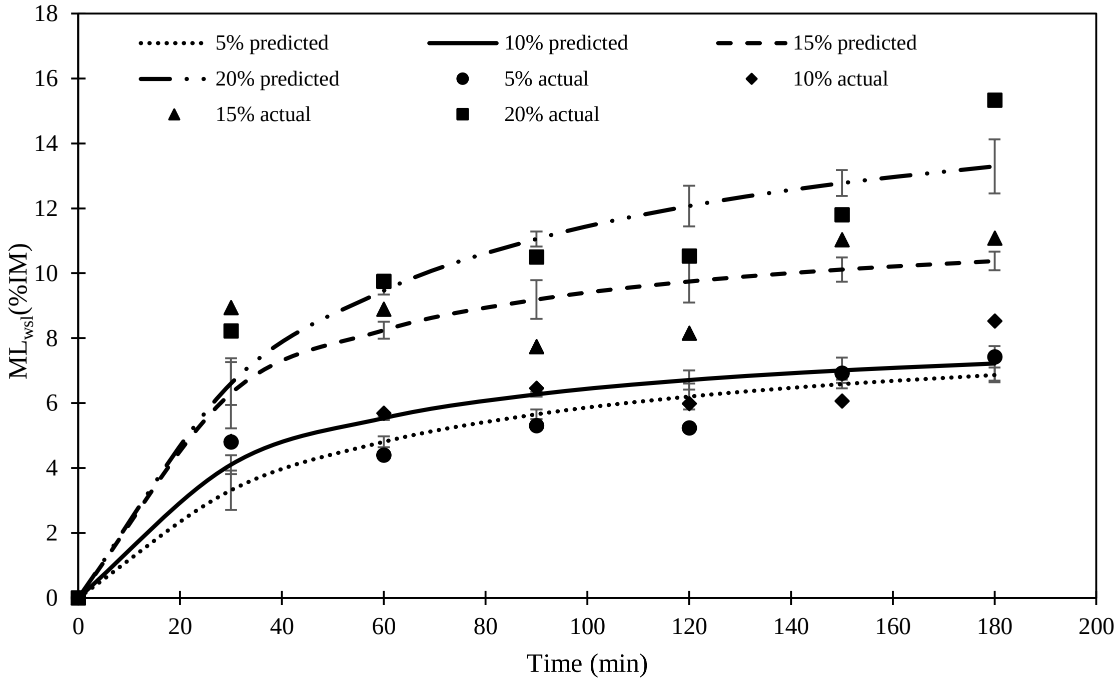


Figure 4.3 Effect of concentrations of osmotic solution on moisture loss at constant temperature 30 °C without solid loss consideration using Azuara model

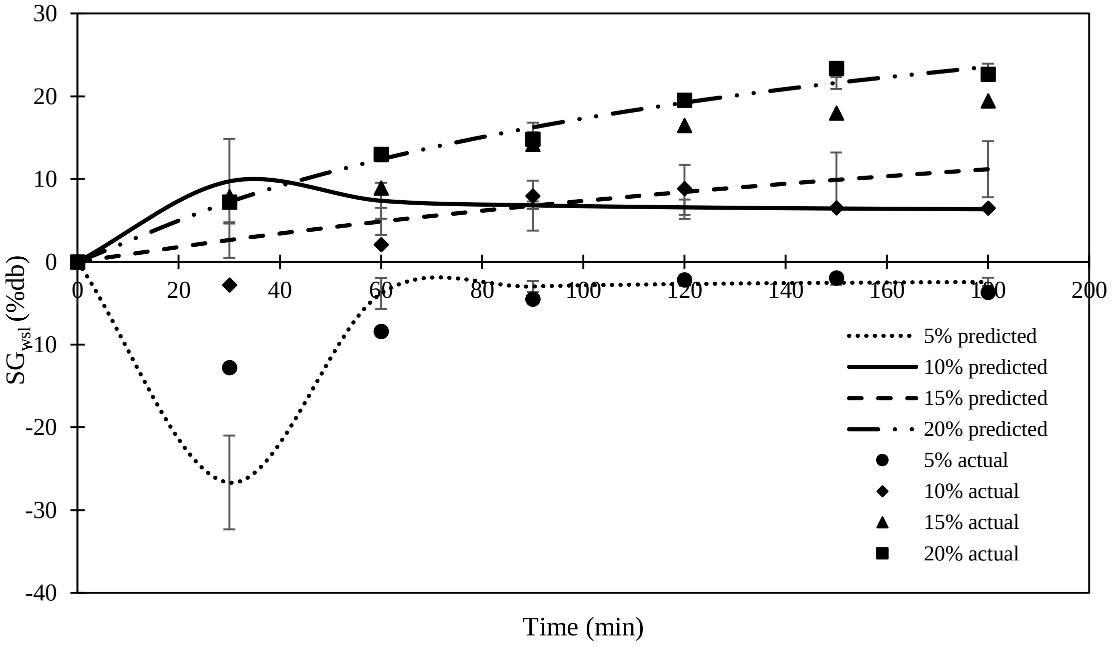


Figure 4.4 Effect of concentrations of osmotic solution on NaCl gain at constant temperature 30 °C without solid loss consideration using Azuara model

The curves of ML_{total} and SG_{total} estimated by mass balance are shown in Fig. 4.3 and 4.4. It was observed that ML and SG increases as the immersion time proceeds and reaches equilibrium condition after a particular period. Comparatively the ML was higher than SG in all the cases. The loss of moisture and salt uptake was rapid in the initial phase and it gradually reduced presumably in the later period. Similar results were reported for other fruits and vegetables [12, 50, 167]. The rate of water removal was in the range of 0.3 - 0.7 g/min during the initial period (30 min) and it gradually reduced to 0.01 - 0.02 g/min during the final stage (180 min). In Fig. 4.4, SG shows negative values especially in the case of 5% concentration of osmotic solution. This behaviour was probably due to the excess loss of solid components than salt gain. The mass transfer kinetics without solid loss consideration were determined since it is the traditional method followed by several researchers to estimate the moisture loss and solid gain. With the aid of traditional method, the difference in equilibrium values with and without solid loss consideration can be obtained.

4.1.2 Dry matter holding capacity (DHC) of lemon slices

Many researchers have stated that the solids loss occurring during osmotic treatment should be considered as negligible. Since the solid loss was predominant in the case of lemon slices, the solid loss during osmotic treatment was related using DHC. The dipping of lemon slices in distilled water without the osmotic agent gave the change in total solid content. The lemon loses its juice sacs in water due to the effect of temperature hence there was change in the dry matter content. The plots for DHC are shown in Fig. 4.5. The average DHC values of lemon decreased from 87.1 to 77.3% when the temperature was varied from 30 to 50 °C, respectively. A linear relationship gave a_1 values 0.113, 0.141 and 0.182 at temperature 30, 40 and 50 °C of water, respectively. The constant a_1 denotes the rate at which the lemon loses its capacity to withhold the integrity. This implied that the DHC of lemon was affected by temperature. High temperature might have disturbed the integrity of the parenchymatic tissues and made lemon feasible for solid loss. It showed that lemon is less capable to hold its dry matter content due to the presence of juice sacs which directly disperse into solution during treatment. Excess solid loss lessens the final yield and can lead to reduced nutritional content in the product.

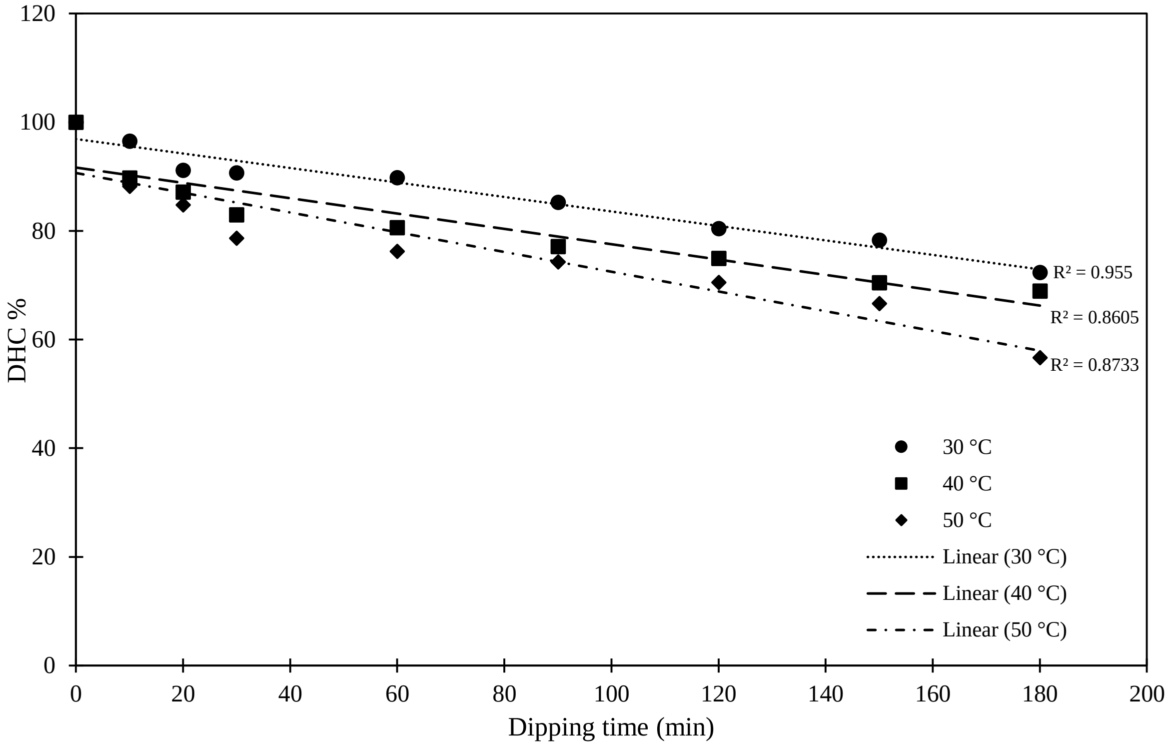


Figure 4.5 Dry matter holding capacity of lemon slices immersed in distilled water at different temperatures

Since the osmotic agent is NaCl, the exact salt gain was determined using Mohr's method. But the knowledge of DHC of lemon slices can be useful to researcher who will be using osmotic agent other than NaCl, like sucrose, fructose, glucose where the quantification of exact solid gain is difficult. Hence, the DHC can give an approximate idea about the losses from lemon tissue when subjected to osmotic dehydration at varying temperature.

4.1.3 Solid loss kinetics during osmotic dehydration

The NaCl content determined using Mohr's method also assured that there was dry matter loss. The curves in Figs. 4.6 – 4.9 shows that SL follows a specific trend, the loss was higher in the initial stage and it almost reached equilibrium in the later phase. At all concentrations, the SL_∞ was higher at 50 °C compared to 30 and 40 °C. At 5% concentration, SL_∞ increased from 18.93 to 35.21 (%db) when the temperature was increased from 30 to 50° C, respectively. Similarly, increase in SL_∞ with respect to temperature increment was found at 10, 15 and 20% of solution concentration and the values are given in Table 4.2. During osmotic dehydration of lemon slices, it was observed that loss of solids was significant and affecting both the ML and SG. The effect of different osmotic solution concentration on solid loss was less while the temperature had pronounceable effect on the loss of lemon's substituents to the osmotic solution. The SL value was found to be inversely proportional to the dry matter holding capacity.

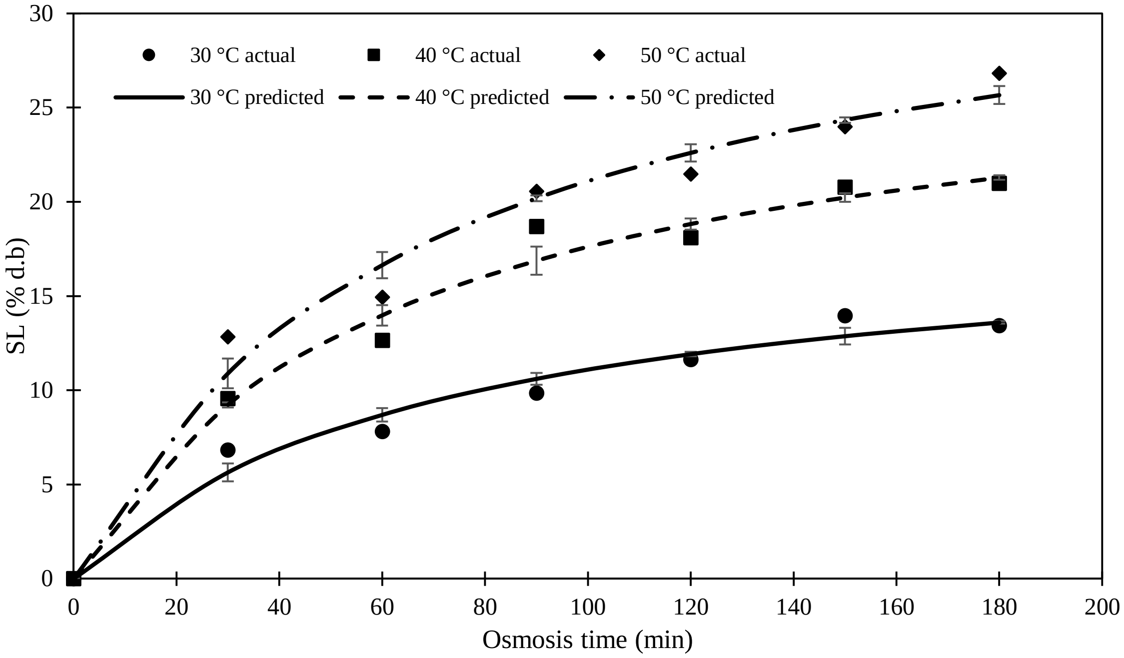


Figure 4.6 Solid loss occurring at different solution temperatures at constant concentration (5% NaCl) of osmotic solution using Azuara model

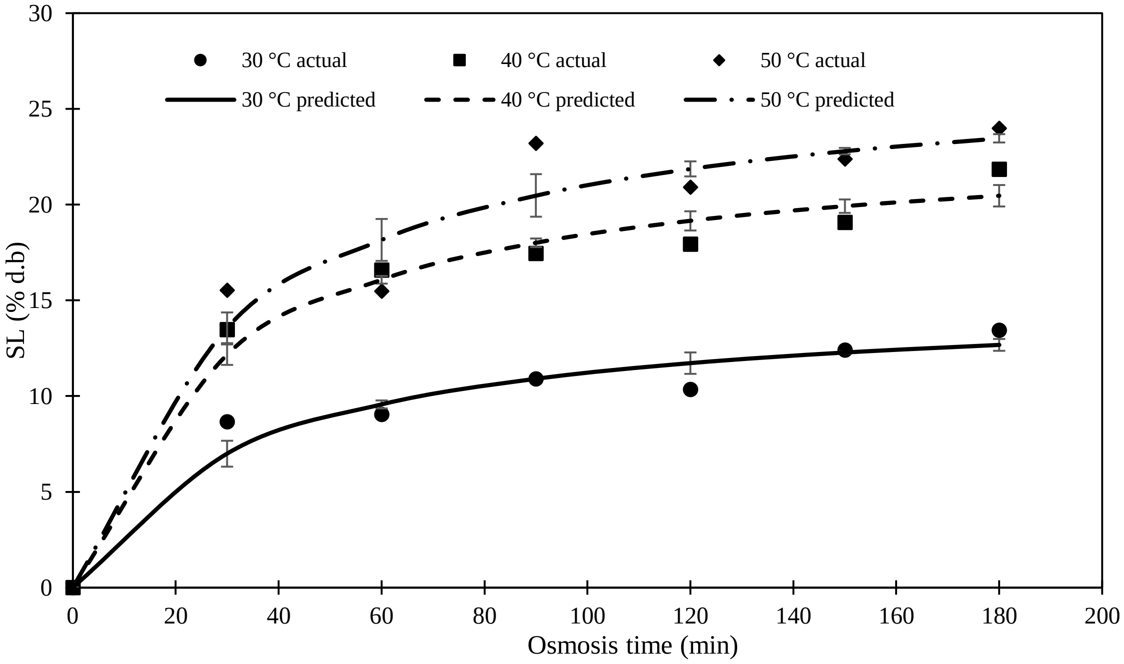


Figure 4.7 Solid loss occurring at different solution temperatures at constant concentration (10% NaCl) of osmotic solution using Azuara model

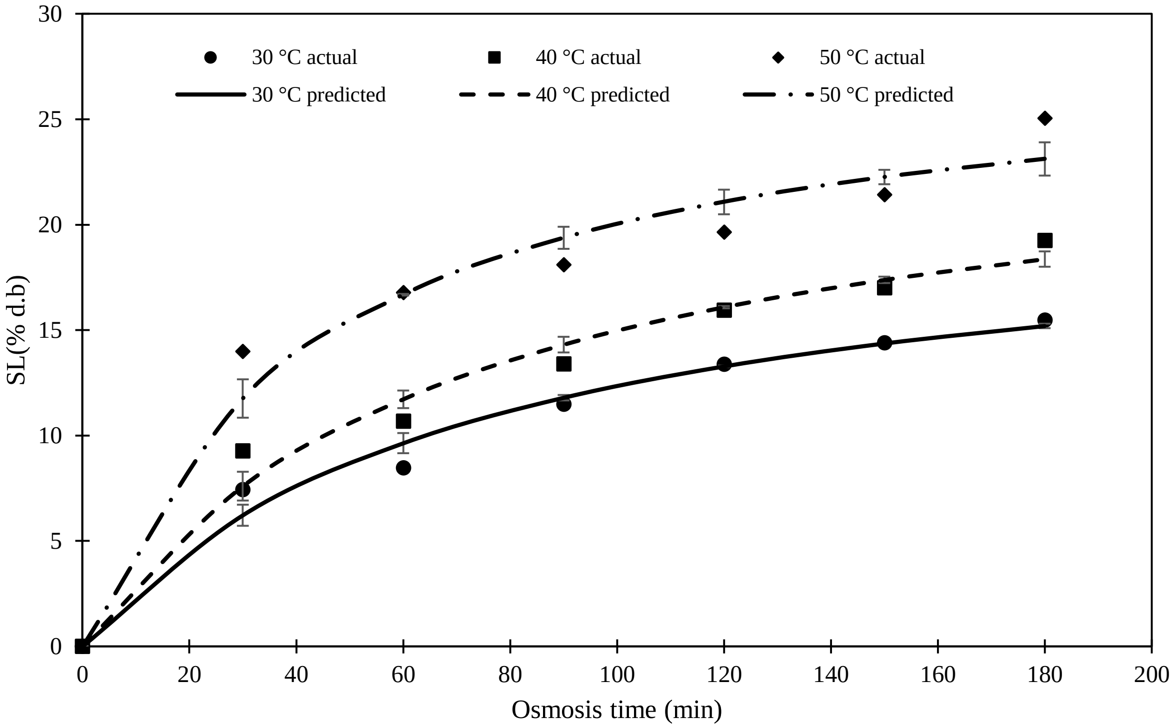


Figure 4.8 Solid loss occurring at different solution temperatures at constant concentration (15% NaCl) of osmotic solution using Azuara model

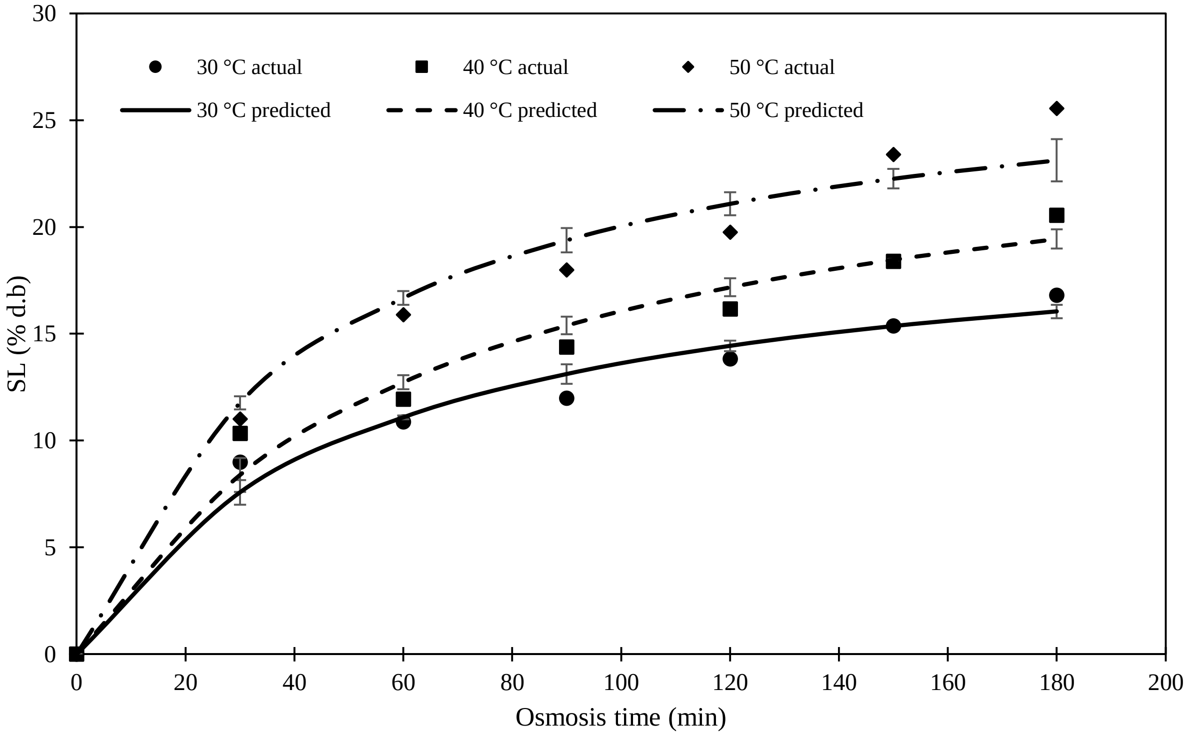


Figure 4.9 Solid loss occurring at different solution temperatures at constant concentration (20% NaCl) of osmotic solution using Azuara model

4.1.4 Moisture loss and salt gain kinetics considering solid and juice sacs loss

The mass transfer kinetics estimated using mass balance showed varied results when the ML and SG were calculated by considering solids and juice sacs losses. This shows that there was significant loss of solids during osmotic dehydration. It was observed that when the moisture leaves it carries away solid matter, especially when the whole sacs of lemon tissue are lost in the osmotic solution. The trends for ML_{os} (% IM) at different temperatures and concentrations of osmotic solution are given in Figs. 4.10 to 4.12. The salt content in dry matter of osmotically dehydrated samples estimated from the Mohr's method were used to determine the exact SG during osmotic dehydration. The SG (% d.b) with osmosis time at different temperatures and concentrations of osmotic solution are shown in Figs. 4.13 to 4.15. The Azuara model was fitted to the ML and SG data and equilibrium values of ML_{os}, SG_{os} with rate constants (S_1 and S_2) were determined. The values are given in Table 4.2. The higher values of S_1 and S_2 indicated the higher diffusion of water and salt per unit time, respectively. The time required for half of the diffusible material to diffuse out or enter in is determined by $\frac{1}{S_1}$ (20 to 30 min) or $\frac{1}{S_2}$ (30 to 150 min), respectively. As the time exceeds the value of $\frac{1}{S_1}$ and $\frac{1}{S_2}$, the ML and SG tends to reach equilibrium, asymptotically. With the constants obtained the predicted ML_{os} and SG_{os} were compared with the actual ML_{os} and SG_{os}, respectively using the RMSE values. For ML_{os}, RMSE ranged between 0.17 and 0.51 and for SG_{os} ranged from 0.08 to 0.56. It was observed that the difference in equilibrium values of ML and SG with and without SL and juice sacs loss consideration was 40 to 60% and 10 to 70%, respectively.

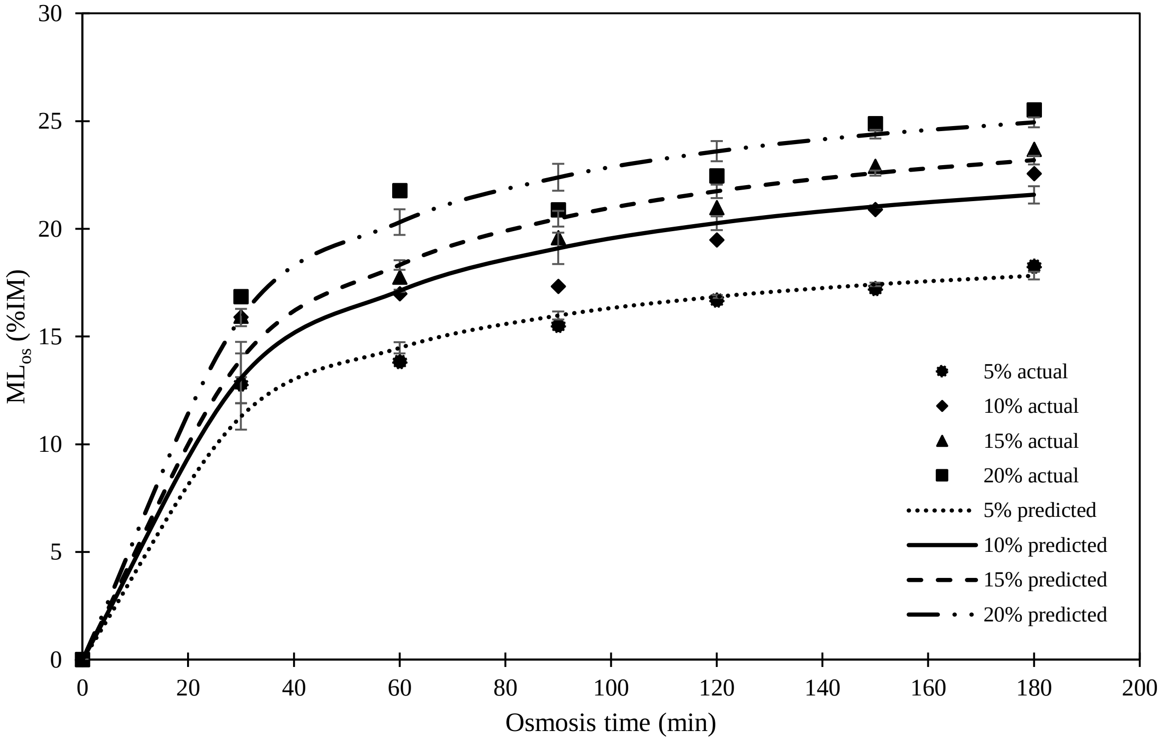


Figure 4.10 Effect of concentrations of osmotic solution on moisture loss at 30 °C with solid loss consideration using Azuara model

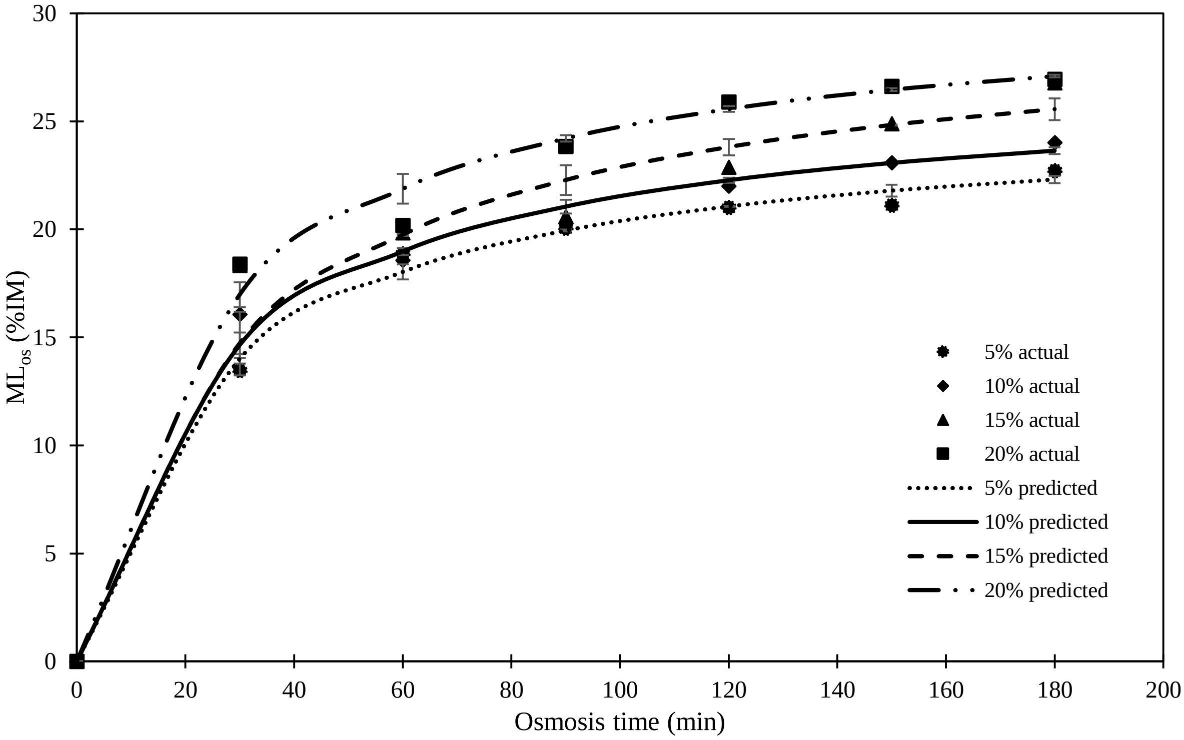


Figure 4.11 Effect of concentrations of osmotic solution on moisture loss at 40 °C with solid loss consideration using Azuara model

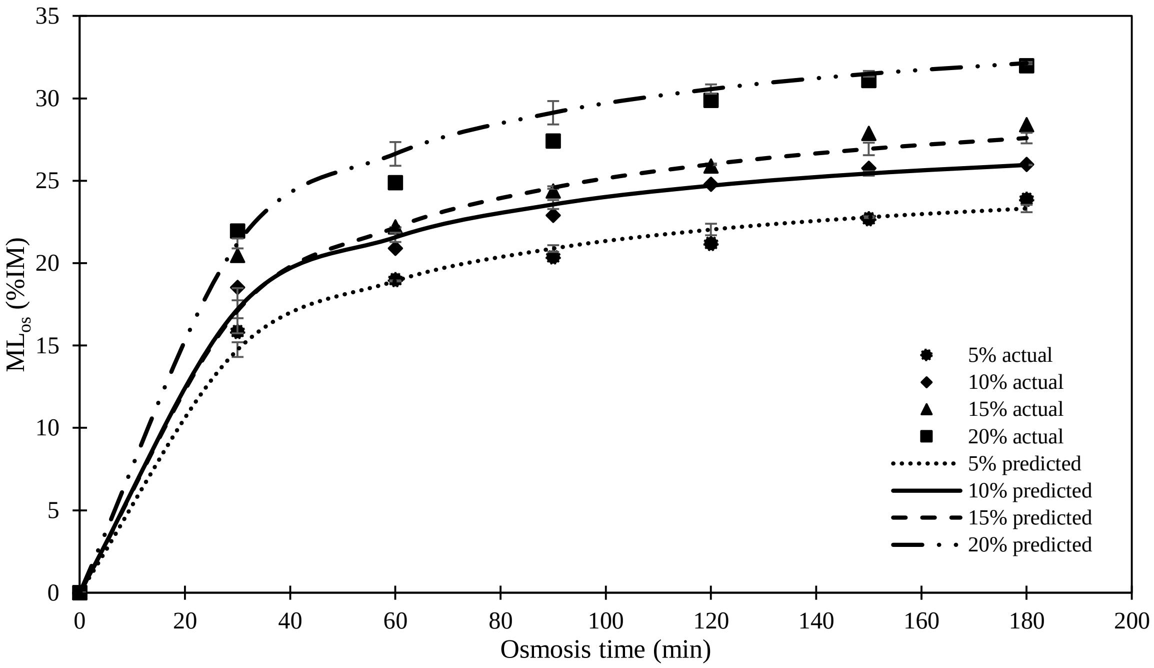


Figure 4.12 Effect of concentrations of osmotic solution on moisture loss at 50 °C with solid loss consideration using Azuara model

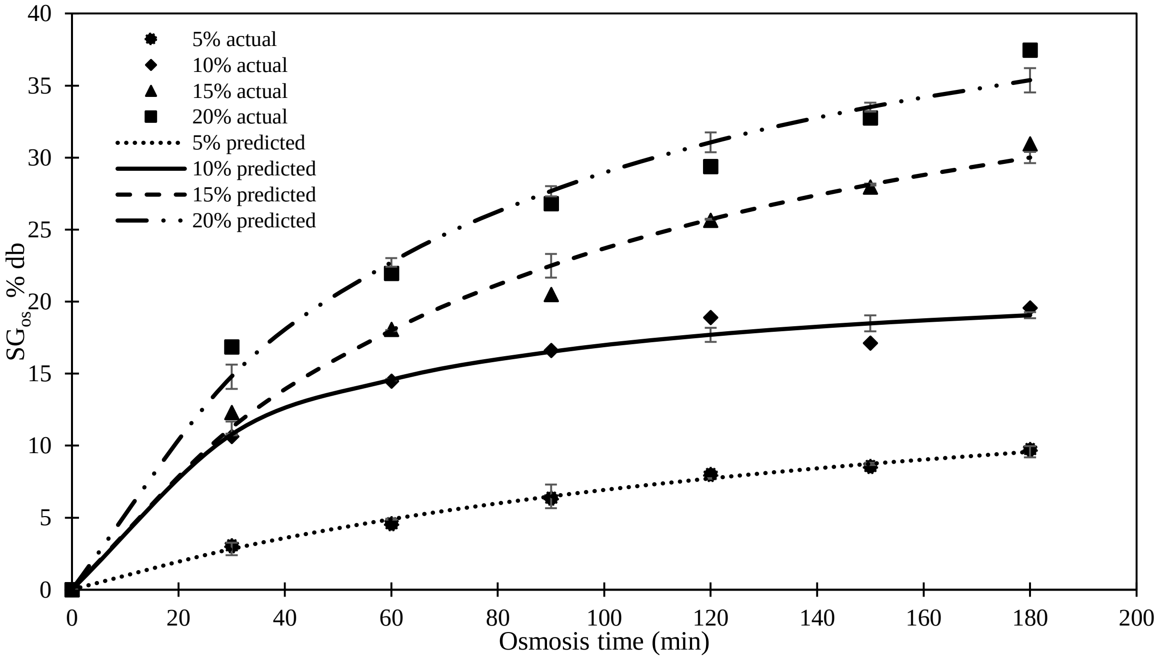


Figure 4.13 Effect of concentrations of osmotic solution on NaCl gain at 30 °C with solid loss consideration using Azuara model

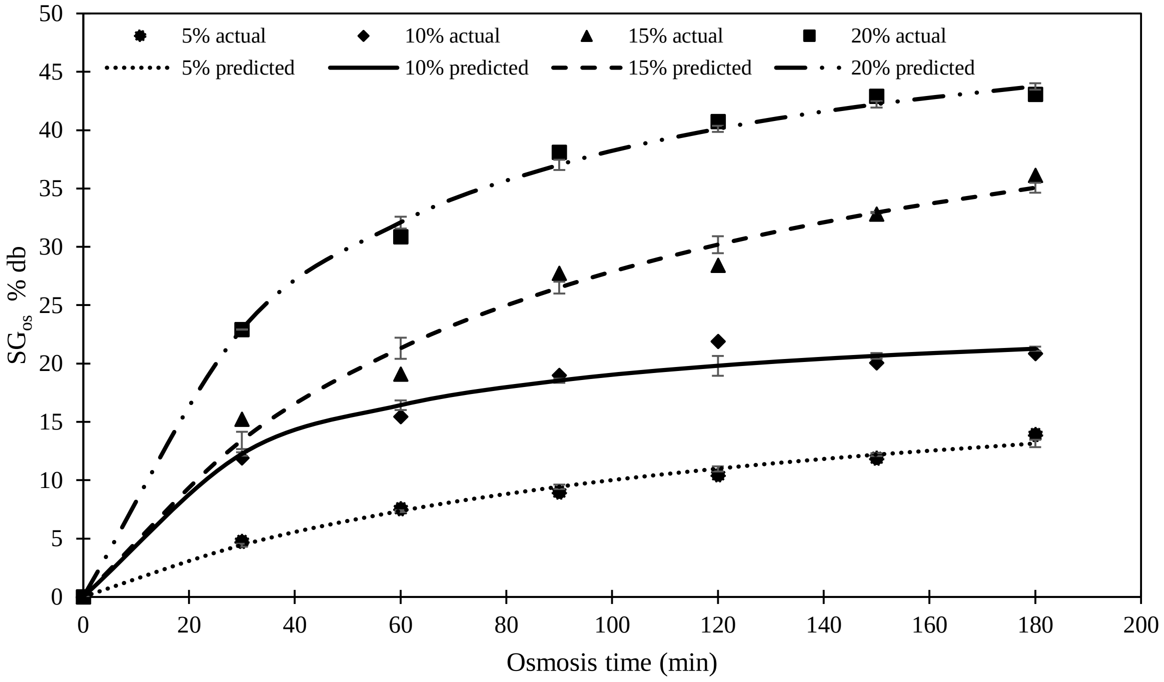


Figure 4.14 Effect of concentrations of osmotic solution on NaCl gain at 40 °C with solid loss consideration using Azuara model

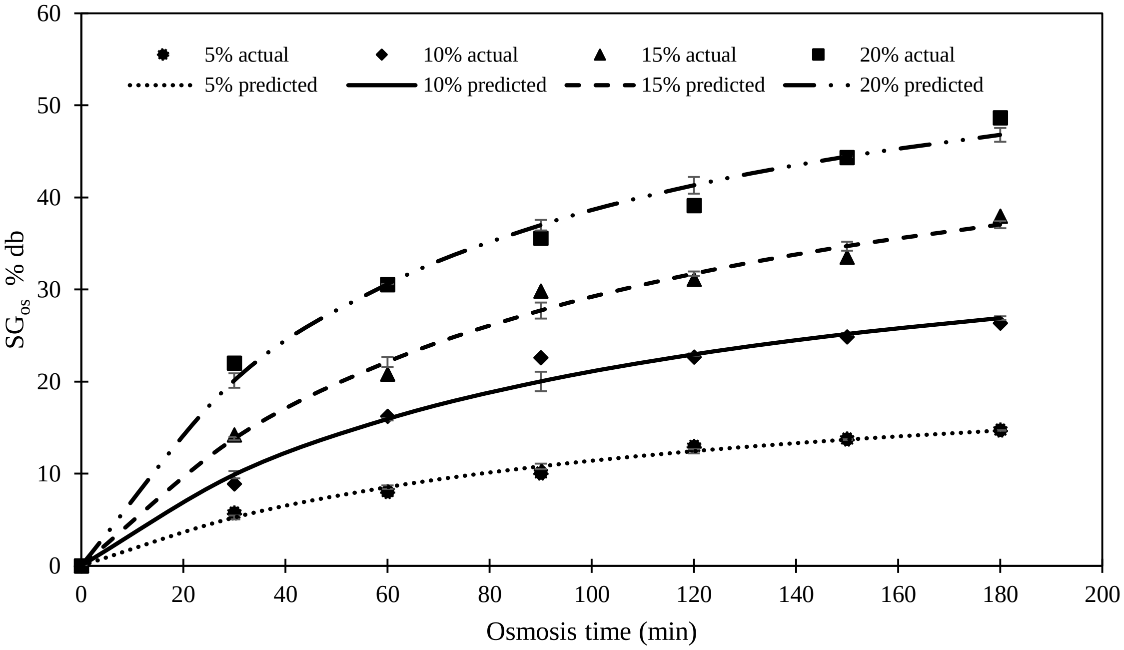


Figure 4.15 Effect of concentrations of osmotic solution on NaCl gain at 50 °C with solid loss consideration using Azuara model

Effect of concentration on mass transfer

The rate of osmotic dehydration increased with increase in concentration of hypertonic solution at a particular temperature, as expected. Similar trend was reported during osmotic dehydration of fruits and vegetables [168-170]. This was because, osmotic pressure between product solution interface increases with increase in solute concentration. The osmotic pressure (π) at different concentrations of NaCl are denoted in Table 4.3. It can be observed that π values increases more with respect to concentration than temperature. The percentage increase of ML_{∞} from 5% to 20% concentration was 39.6, 21.53 and 35.86 % for 30°, 40° and 50 °C. For SG_{∞} the increment was found to be double. The ML_{os} and SG_{os} was rapid in the initial stage and the rate of mass transfer reduced presumably because of the decreasing driving force. In other words the equilibrium condition was almost approaching. In few other cases, the time required to reach equilibrium conditions is higher in case of higher concentration of the osmotic solution. It may be due to the mass transfer resistance in the osmotic solution adjacent to the lemon surface increases with increase in the solution concentration. It is apparent due to the viscous nature of the higher concentrated solution [171]. The initial viscosity of 20% solution was higher (1.142 cP) than 5% concentration (0.864 cP) of the osmotic solution. Also, the NaCl migration towards the sample would have formed a subsurface on the periphery of the sample which forms a barrier for the moisture removal. The Figs. 4.10 to 4.15 indicates that higher solution concentration increased the transfer of water and NaCl molecules.

Table 4.2 The constants and equilibrium values of Azuara model for solid loss, salt gain and moisture loss (only due to osmosis)

Salt concentration (% NaCl)	Temperature								
	30 °C			40 °C			50 °C		
	SL_∞ (% d.b)	**S_3 (per min)**	**RMSE**	**SL_∞ (% d.b)**	**S_3 (per min)**	**RMSE**	**SL_∞ (% d.b)**	**S_3 (per min)**	**RMSE**
5	18.93	0.014	0.295	28.81	0.015	0.344	35.21	0.014	0.452
10	15.12	0.028	0.302	23.69	0.035	0.395	27.47	0.032	0.631
15	21.36	0.013	0.495	25.64	0.014	0.342	28.65	0.023	0.53
20	20.66	0.019	0.281	26.38	0.015	0.404	28.65	0.023	0.531
	SG_∞ (% d.b)	**S_2 (per min)**	**RMSE**	**SG_∞ (% d.b)**	**S_2 (per min)**	**RMSE**	**SG_∞ (% d.b)**	**S_2 (per min)**	**RMSE**
5	18.38	0.006	0.085	21.64	0.008	0.176	22.98	0.009	0.158
10	22.52	0.030	0.232	24.93	0.032	0.332	40.98	0.010	0.337
15	45.04	0.011	0.296	51.81	0.011	0.561	55.86	0.010	0.445
20	49.01	0.014	0.56	53.47	0.025	0.295	63.69	0.015	0.513
	ML_∞ (% IM)	**S_1 (per min)**	**RMSE**	**ML_∞ (% IM)**	**S_1 (per min)**	**RMSE**	**ML_∞ (% IM)**	**S_1 (per min)**	**RMSE**
5	20.16	0.042	0.232	25.31	0.041	0.178	26.38	0.042	0.217
10	24.81	0.037	0.451	26.95	0.039	0.222	28.9	0.049	0.209
15	26.73	0.036	0.341	29.97	0.033	0.518	31.44	0.0381	0.37
20	28.16	0.043	0.417	30.76	0.041	0.279	35.84	0.048	0.374

Effect of temperature on mass transfer

The mass transfer rates gets accelerated at higher temperature. The change in structure of cell membrane and decrease in viscosity of the osmotic solution at higher temperature makes feasible for diffusion of moisture and NaCl. The ML_{os} rate increases with increase in temperature and is evident in Table 4.2. Similar results were found by other researchers during osmotic dehydration of fruits and vegetables [22, 58, 142, 169]. Due to high temperature the semi-permeability of the tissues might have changed, which resulted in higher moisture loss. Also, the high temperature causes decrease in viscosity of the solution which can help in better mass transfer characteristics. From Table 4.3, it was observed that the viscosity was higher 1.142 cP in case of 30 °C and it reduced to 0.792 cP at 50 °C for 20% concentration of osmotic solution. The decrease in viscosity facilitates higher penetration of salt into the lemon slices and thereby restricts the formation of surface layer. This helps in enhanced salt infusion at increased temperature. The SG_{∞} (% db) increased from 49.01 to 63.69 when the temperature was increased from 30 to 50 °C. The equilibrium condition for ML_{os} and SG_{os} increased with respect to temperature since the synergetic effect of higher solution concentration and temperature developed high osmotic potential. Also, from SL data it was noted that there was accelerated loss of lemon's own components to the osmotic solution at higher temperature. Generally the flow of natural solutes of the food to the solution is considered as negligible even-though it is important for organoleptic and nutritional properties of the product [172-174]. Whereas the results prove that the consideration of solid loss is important to increase the yield and maintain the product's organoleptic and nutritional properties.

Table 4.3 Osmotic pressure and viscosity values at different temperatures and concentrations of osmotic solution

Salt concentration (% NaCl)	**Temperature**					
	30 °C		**40 °C**		**50 °C**	
	π (atm)	**η (cP)**	**π (atm)**	**η (cP)**	**π (atm)**	**η (cP)**
5	21.25	0.864	21.96	0.714	22.66	0.600
10	42.54	0.940	43.94	0.761	45.35	0.648
15	63.8	1.02	65.9	0.842	68.01	0.712
20	85.08	1.142	87.89	0.944	90.7	0.792

4.1.5 Regression analysis and development of correlations

The effect of osmosis time, concentration and temperature on SG, SL and ML was found by the multiple regression method using Eq. (3.15), (3.16) and (3.17) and ANOVA. Apart from the concentration and temperature of the osmotic solution, the SG_{θ} and SL_{θ} at different concentration and temperature of hypertonic solution during the osmosis process affects the ML_{θ} from lemon slices. During solid loss the moisture is also taken away in the form of sacs of lemons. Also, during osmotic dehydration higher salt gain forms a sub-layer on the surface of the lemon which hinders the removal of moisture. Hence, the below equations gives the correlations for various parameters.

$ML_\theta = 4.6144\,\theta^{0.1491} C^{0.2375} T^{0.2641} SL_\theta^{0.2008} SG_\theta^{-0.044}$ ($R^2 = 0.96$) …... (4.1)

$SG_\theta = 4.8551\,\theta^{0.3165} C^{0.958} T^{0.3479} SL_\theta^{0.284}$ ($R^2 = 0.98$) …... (4.2)

$SL_\theta = 0.0699\,\theta^{0.3407} C^{-0.002} T^{1.047}$ ($R^2 = 0.92$) …... (4.3)

The results of regression and ANOVA showed that the ML_θ is highly dependent on the θ, C, T and SL ($p<0.05$). But the effect of SG on ML was insignificant ($p > 0.05$). While in the case of SG_θ (Eq. (4.3)), SL has no significant effect and it was highly affected by temperature and concentration of osmotic solution and time of treatment. This indicates the salt gain was independent of SL. Also, it can be observed that concentration of the aqueous solution is not related to the SL whilst the temperature and time has dominant effect. It was expected as lemon loose more solids when subjected to higher temperature for longer time. The same was observed with respect to DHC study. Therefore, higher temperature is not recommended for treatment of lemon. Excess intake of NaCl affect the taste of the product and gives undesirable flavour which is extremely possible while adopting high temperature. Since the DHC of lemon is low the usage of high temperature is to be avoided to achieve more yield and obtain quality product.

4.1.6 Optimization of process parameters

The optimal condition was selected based on the duration at which the maximum moisture loss occurs, salt gain falls within allowable legal limits without affecting the acceptability of dehydrated lemons by the consumers and minimum solid loss during treatment. The limit for salt consumption is based on the intake of sodium. According to U.S. Food and Drug Administration (FDA), the recommended daily intake for sodium is less than 2400 mg per day. Daily Values (DV) were developed by the FDA to help consumers determine the level of various nutrients in a standard serving of food in relation to their approximate requirement for it. For sodium, if the %DV is lesser than 5% then it is considered as low sodium content product and if it is higher than 20% then it is considered as high sodium content product. The optimisation was performed using solver tool in Microsoft excel by GRG (Generalised reduced gradient) non-linear solving method. Maximum moisture loss was kept as objective and the constraints were set using Eq. (4.1), (4.2) and (4.3). Based on that, the optimal time, concentration and temperature were determined. The responses obtained using solver at various conditions are represented in Table 4.4.

Table 4.4 Solutions based on optimisation done using solver tool in excel at 180 min

Salt concentration (% NaCl)	Temperature								
	ML_{os}			SG_{os}			SL		
	30 °C	40 °C	50 °C	30 °C	40 °C	50 °C	30 °C	40 °C	50 °C
5%	18.66	21.21	23.43	9.94	11.97	13.82	14.52	19.62	24.78
10%	21.36	24.28	26.82	19.3	23.24	26.84	14.50	19.59	24.75
15%	23.11	26.28	29.03	28.46	34.27	39.5	14.48	19.58	24.73
20%	24.45	27.79	30.7	37.49	45.13	52.12	14.48	19.57	24.72

The optimum condition for osmotic dehydration of lemon slices was found to be 20% concentration of solution at 30 °C for a period of 180 min. Higher concentration was selected as the required salt gain (to reduce the water activity of the lemon slice) was attainable at 20% concentration in 180 min with low solid loss. During preliminary trials it was observed that the time required to attain the higher moisture loss along with required salt gain was more than 4 hours at low concentrations. Further, to accelerate the mass transfer at low concentration additional agitation was required which increases the processing cost. Hence, the optimum condition was selected based on maximum moisture loss (24.45, %IM), salt gain which reduces the water activity and minimum possible solid loss (14.48%, d.b). From the optimised result, the salt uptake was 39.49% (db). Generally, 100 g of NaCl contains 38.758 g of sodium. Per serving, approximately 10g of dried lemon slice is required which contains 0.52g of salt and its corresponding sodium content is 201.53 mg (0.2 g). The %DV is less than 10% of 2400 mg which is under the legal limit.

4.2 Chemical and Thermal Pretreatments

4.2.1 Effect of chemical treatment on ascorbic acid and enzyme activity in lemon

The effect of chemical pre-treatment (ascorbic acid and calcium chloride) on ascorbic acid content, pectinesterase and peroxidase activity was determined by using Box-Behnken design of experiments. The statistical analysis (ANOVA) for each response is given in Table 4.5. From the table it can be inferred that the model showed significant change ($p<0.05$) in the ascorbic acid content. But the contribution of individual parameters like calcium chloride concentration, time of treatment and temperature was insignificant except ascorbic acid. Similarly, the chemical treatment had insignificant effect on the pectinesterase activity ($p>0.05$) whereas ascorbic acid and temperature showed a significant effect on the peroxidase inactivation. The pectinesterase was found to be highly resistant to temperature in the selected range.

Table 4.5 ANOVA for response surface linear model for ascorbic acid, pectinesterase and peroxidase

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
			Ascorbic Acid			
Model	0.096537	4	0.024134	3.681057	0.0179*	significant
A-Ascorbic acid	0.082582	1	0.082582	12.59587	0.0016**	
B-Calcium chloride	0.006667	1	0.006667	1.016901	0.3233	
C-Time	1.58×10^{-06}	1	1.58×10^{-06}	0.000241	0.9877	
D-Temp	0.007286	1	0.007286	1.111221	0.3023	
Residual	0.157352	24	0.006556			
Lack of Fit	0.157279	20	0.007864	430.5987	< 0.0001	significant
Pure Error	7.31×10^{-06}	4	1.83×10^{-06}			
Cor Total	0.253888	28				
			Pectinesterase			
Model	1449.838	4	362.4596	2.136956	0.1073	not significant
A-Ascorbic acid	204.7324	1	204.7324	1.207043	0.2828	
B-Calcium chloride	603.2155	1	603.2155	3.556384	0.0715	
C-Time	5.254666	1	5.254666	0.03098	0.8618	
D-Temp	636.6357	1	636.6357	3.75342	0.0646	
Residual	4070.756	24	169.6149			
Lack of Fit	4051.99	20	202.5995	43.18334	0.0011	significant
Pure Error	18.76645	4	4.691613			
Cor Total	5520.595	28				
			Peroxidase			
Model	3553.068	4	888.2669	4.651179	0.0064**	significant
A-Ascorbic acid	1813.311	1	1813.311	9.494933	0.0051**	
B-Calcium chloride	48.36142	1	48.36142	0.253232	0.6194	
C-Time	249.4504	1	249.4504	1.306182	0.2644	
D-Temp	1441.945	1	1441.945	7.550367	0.0112*	
Residual	4583.442	24	190.9767			
Pure Error	0.286184	4	0.071546			
Cor Total	8136.51	28				

(* and ** are significance at 5% and 1% level of significance)

The ANOVA indicated that peroxidase and pectinesterase were not simultaneously inactivated by any combination of chemical pretreatment. Therefore, the chemical pretreatment was not used for further study.

4.2.2 Effect of ultrasound and blanching on ascorbic acid and enzyme activity in lemon

The ultrasound had insignificant effect on the inactivation of pectinesterase and peroxidase. The enzyme activity was similar to the fresh sample, while it had an increased ascorbic acid degradation effect. Water blanching and steam blanching had significant effect on the inactivation of pectinesterase and peroxidase. The enzyme activity was low comparatively in steam blanching and the retention of ascorbic acid content was more when compared to the water blanching. In water blanching, dipping of the lemon slices leads to loss of solids and the temperature was insufficient to inactivate enzyme. Also, steam blanching for 5 min comparatively loses more ascorbic acid content than 1 min. Therefore, steam blanching for 1 min was selected as a pretreatment prior to drying to inactivate enzymes as well as to retain the organoleptic and nutritional components. Steam blanching was done after osmotic dehydration as the salt content in the lemon slice maintained the intactness and prevented the loss of other components. The ascorbic acid content was found to be relatively higher for lemon slices, steam blanched after osmotic dehydration than the slices steam blanched prior to osmotic dehydration. Therefore, steam blanching was done to osmotically dehydrated lemon slices for further drying by infrared and microwave hot air drying.

4.3 Drying of Pre-treated Lemon

4.3.1 Infrared hot air drying

The osmotically pre-treated lemon slices at 30 °C, 20% NaCl concentration and 180 min were steam blanched for 1 min prior to infrared hot air drying. From preliminary trials it was found that the drying rate predominantly reduces after 30% moisture content. Radiation intensity, air temperature, distance between the emitter and product, and the air velocity were kept as independent parameters. Unlike microwaves, infrared heats the product surface and availability of surface and free moisture was less after 30% moisture content. Hence for optimization, the cut off time of infrared drying was decided to be moisture content less than 30% w.b (approx.), within 60 min.

The effect of various conditions on moisture content reached after 60 min, Page model constants and apparent moisture diffusivity are given in Tables 4.6 and Table 4.7. From the tables, it is evident that the apparent moisture diffusivity was highest at the combination of high radiation intensity, high air temperature, low distance between the source and product and high air velocity. Also, the diffusivity values were in high range (10 $^{-9}$ m^2/s) compared to other drying methods reported in literature (10^{-12} m^2/s). The infrared radiation impinges on the surface of the product and penetrates to a limited distance. Due to the exposure of the product to infrared radiation, increased molecular vibration generates heat in the product and elevated moisture diffusivity. Accelerated heating leads to the movement of water towards the surface leading to rapid moisture removal from the product and elevated moisture diffusivity. Besides, the hot air removes moisture from the surface, which results in rapid mass transfer. The R^2 values for Page model were between 0.979 and 0.999 indicating its suitability to the drying data. The k value of the combined drying increased with increase in air temperature and radiation intensity. Similar results were reported by other researchers [40].

Table 4.6 Moisture content reached after 60 min, page model constants and apparent diffusivity by infrared hot air drying

Ex No.	**Radiation Intensity (Wm^{-2})**	**Temperature (°C)**	**Distance (mm)**	**Velocity (m/s)**	**MC after 60 min (%w.b)**	**Page model**			**Apparent moisture diffusivity (m^2/s)**
						k (min^{-1})	**n**	**R^2**	
1	4000	70	150	1.86	42.8	0.013	1.047	0.989	3.21×10^{-09}
2	4000	70	150	0.86	44.64	0.010	1.082	0.998	2.53×10^{-09}
3	3000	50	100	1.30	54.58	0.007	1.001	0.999	1.18×10^{-09}
4	5000	50	100	1.30	48.54	0.008	1.051	0.998	1.86×10^{-09}
5	3000	90	100	1.30	29.39	0.018	1.057	0.998	4.05×10^{-09}
6	5000	90	100	1.30	24.45	0.014	1.167	0.997	5.07×10^{-09}
7	3000	50	200	1.30	54.33	0.008	0.962	0.999	1.18×10^{-09}
8	5000	50	200	1.30	52.93	0.004	1.160	0.992	1.52×10^{-09}
9	3000	90	200	1.30	43.11	0.003	1.345	0.979	2.87×10^{-09}
10	5000	90	200	1.30	41.95	0.006	1.231	0.999	2.87×10^{-09}
11	5517.7	70	150	0.47	47.57	0.007	1.115	0.999	2.20×10^{-09}
12	2482.3	70	150	0.47	50.72	0.009	0.999	0.993	1.86×10^{-09}
13	4000	100.35	150	0.47	37.87	0.009	1.180	0.998	3.72×10^{-09}
14	4000	39.64	150	0.47	57.86	0.005	0.987	0.999	8.44×10^{-09}
15	4000	70	225.88	0.47	57.52	0.004	1.055	0.991	1.01×10^{-09}

Table 4.7 gives the ANOVA for infrared hot air drying for moisture removal. It was observed that all the individual parameters are highly significant ($p < 1\%$) in reducing the moisture content.

Table 4.7 ANOVA for moisture content after 60 min during infrared hot air drying

Source	**Sum of Squares**	**df**	**Mean of Square**	**F value**	**p-value**
Model	1389.65	14	99.26	1073.67	0.0239
A-Intensity	26.62	1	26.62	287.99	0.0375
B-Temperature	822.34	1	822.34	8895.01	0.0067
C-Distance	327.21	1	327.21	3539.27	0.0107
D-Velocity	77.37	1	77.37	836.80	0.0220
AB	0.22	1	0.22	2.43	0.3632
AC	8.86	1	8.86	95.86	0.0648
AD	1.25	1	1.25	13.56	0.1688
BC	91.67	1	91.67	991.52	0.0202
BD	16.14	1	16.14	174.54	0.0481
CD	9.96	1	9.96	107.76	0.0611
A^2	2.50	1	2.50	27.07	0.1209
B^2	0.044	1	0.044	0.47	0.6167
C^2	0.13	1	0.13	1.44	0.4426
D^2	5.77	1	5.77	62.45	0.0801
Residual	0.092	1	0.092		
Cor Total	1389.74	15			

The drying rate curves at different conditions are shown in Figs. 4.16 to 4.19. Higher drying rate was observed in higher radiation intensity as expected. The increase in infrared intensity led to the increased absorption of infrared energy by the salt and lemon slices. Further, the temperature and vapour pressure inside the lemon slices might have increased resulting into higher drying rates. Similar results were reported in literature for infrared drying of rice [175]. Increased moisture removal rate was apparent with higher air velocity. Constant drying rate period was absent in all the cases, only falling rate period was evident, it may be because of the unavailability of constant amount of moisture throughout the drying period. Similar trend of falling rate was also reported by other researchers [176]. But in case of lemon after one hour, the falling rate was decreased drastically. Therefore, further drying by infrared was avoided as it was found to be increasing the drying time and energy consumption.

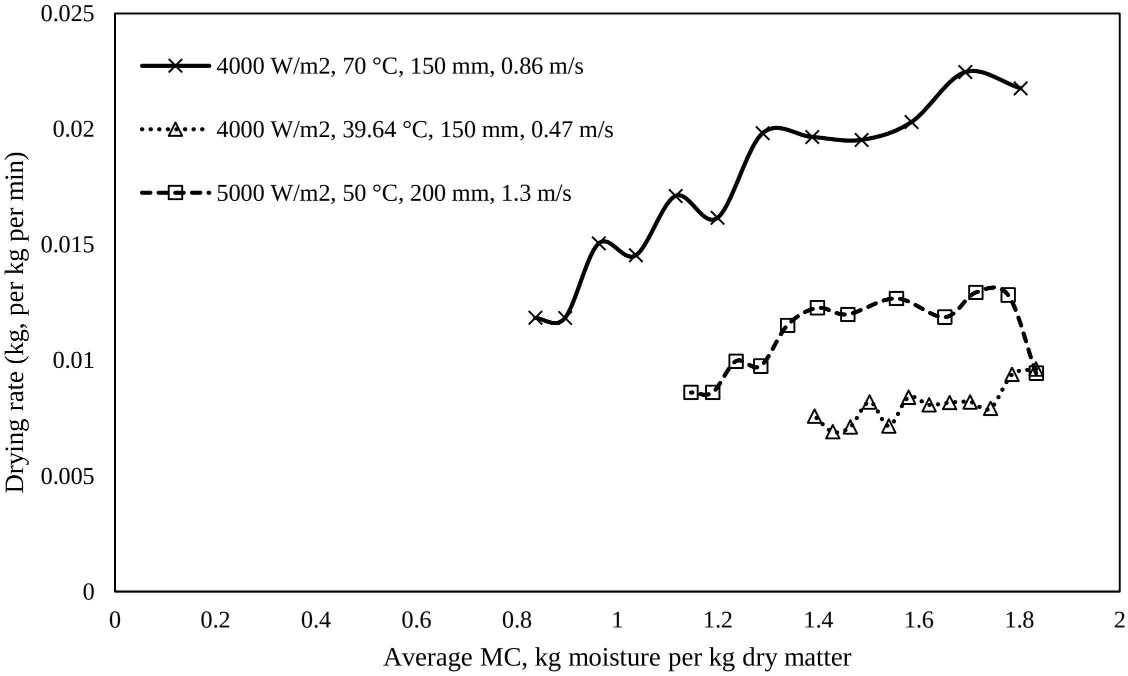


Figure 4.16: Drying rate (kg per kg per min) and average moisture content (kg moisture per kg dry matter) at different conditions (exp no. 2, 8, 14) of infrared hot air drying

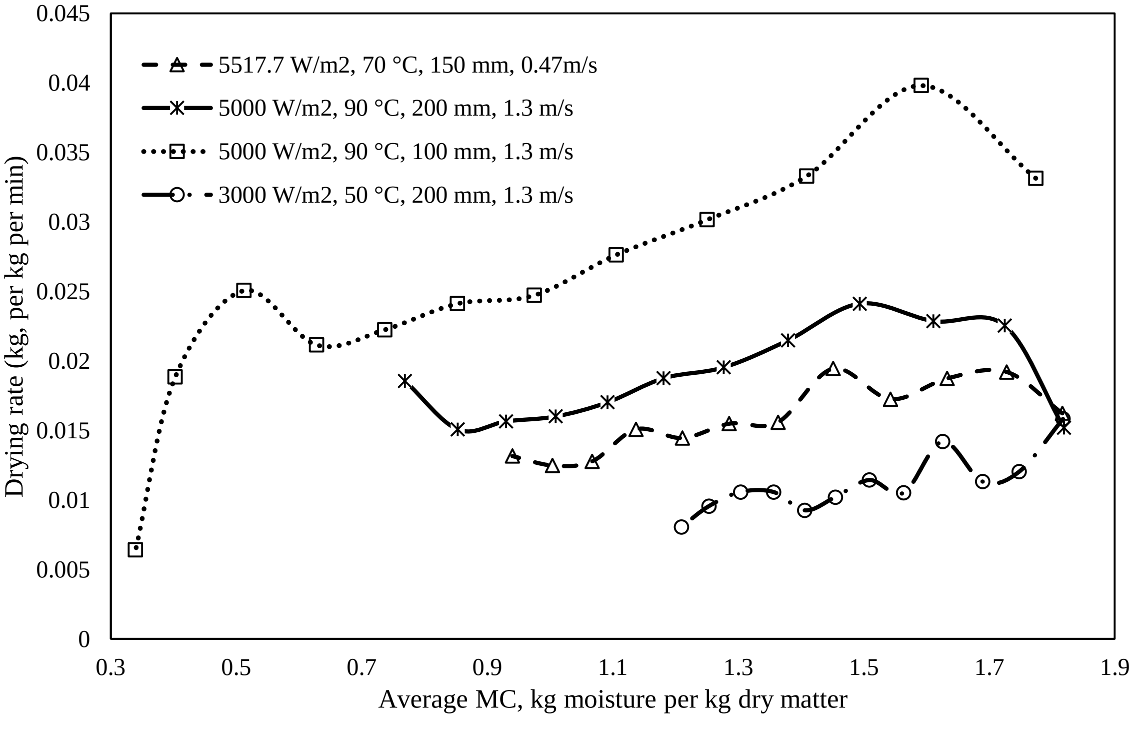


Figure 4.17 Drying rate (kg per kg per min) and average moisture content (kg moisture per kg dry matter) at different conditions (exp no.11, 10, 6, 7) of infrared hot air drying

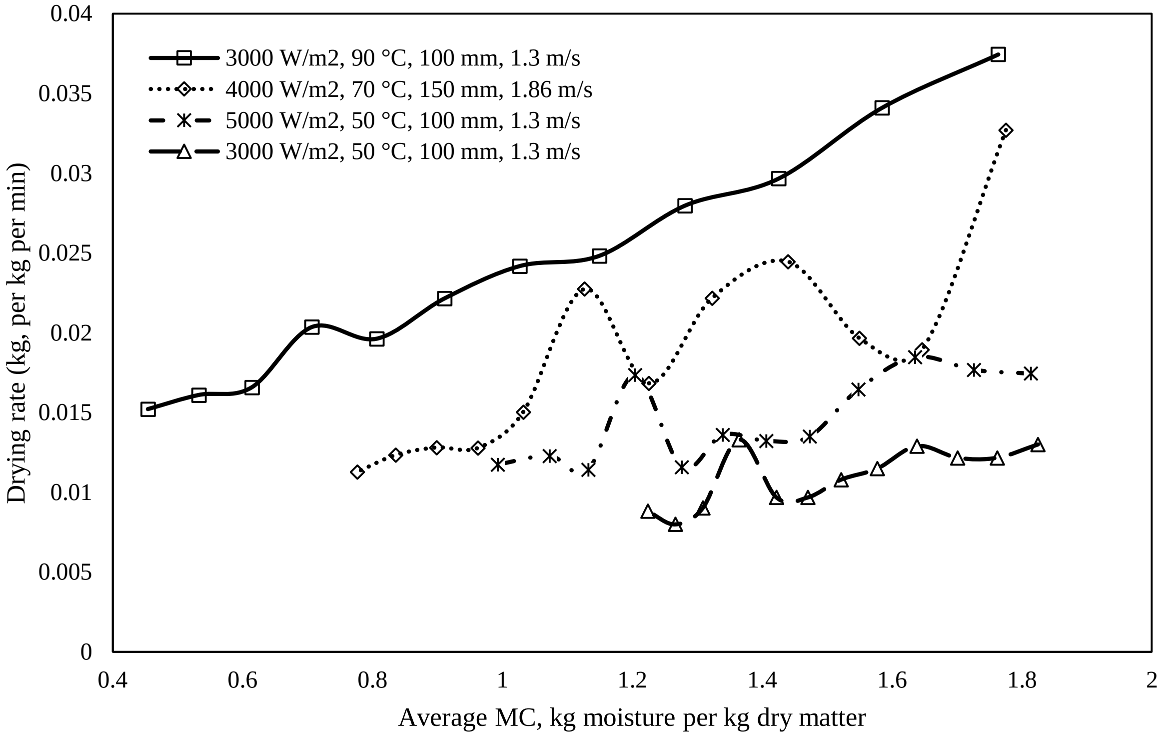


Figure 4.18 Drying rate (kg per kg per min) and average moisture content (kg moisture per kg dry matter) at different conditions (exp no. 5, 1, 4, 3) of infrared hot air drying

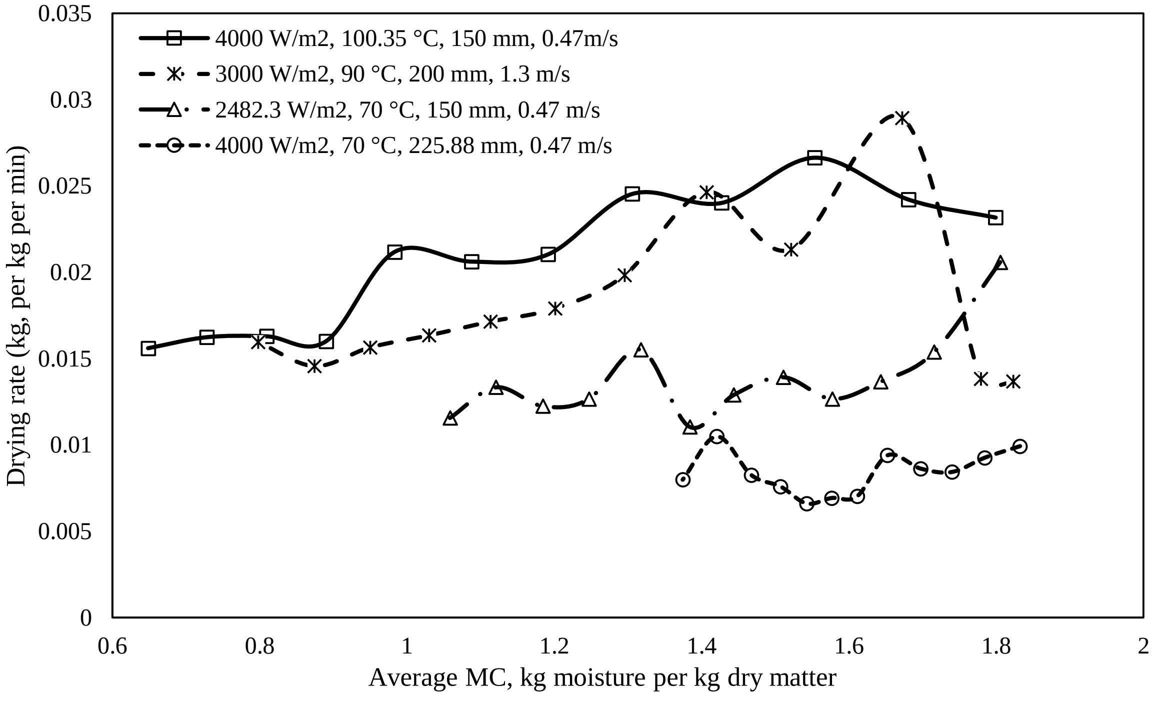


Figure 4.19 Drying rate (kg per kg per min) and average moisture content (kg moisture per kg dry matter) at different conditions (exp no.13, 9, 12, 15) of infrared hot air drying

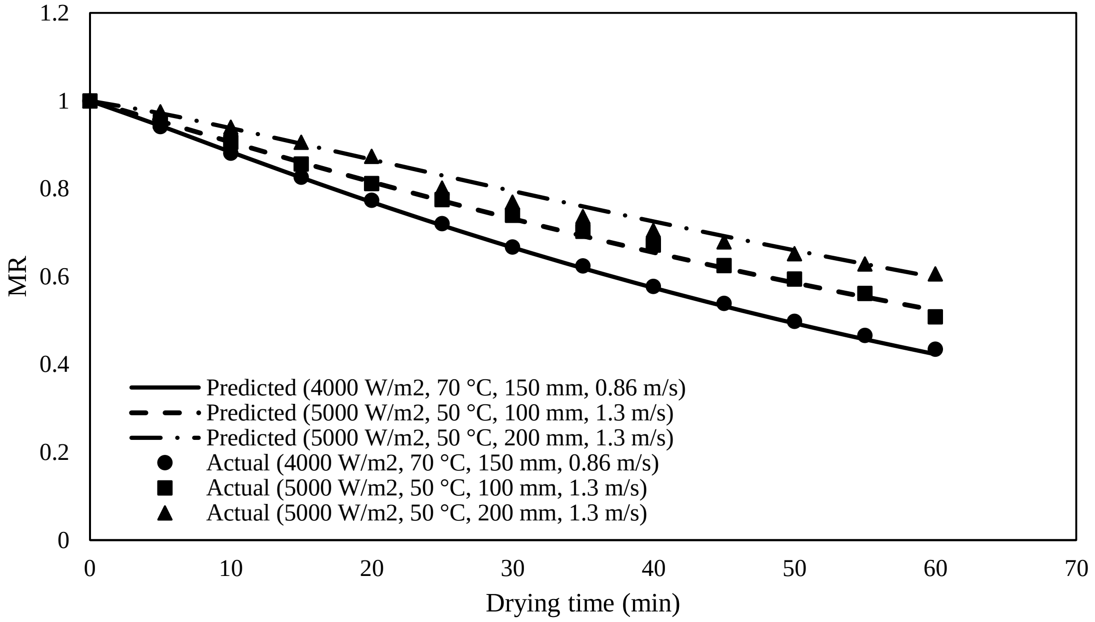


Figure 4.20 Moisture ratio at different conditions (exp no. 2, 4, 7) of infrared hot air drying using Page model

Variation of moisture ratio with time plotted using Page model are shown in Figs 4.20 to 4.23. The curves indicate higher moisture removal at high intensity, lesser distance, and high air temperature and air velocity. Distance and air temperature had noticeable effect on moisture ratio. Page model showed good fit with high R^2 values.

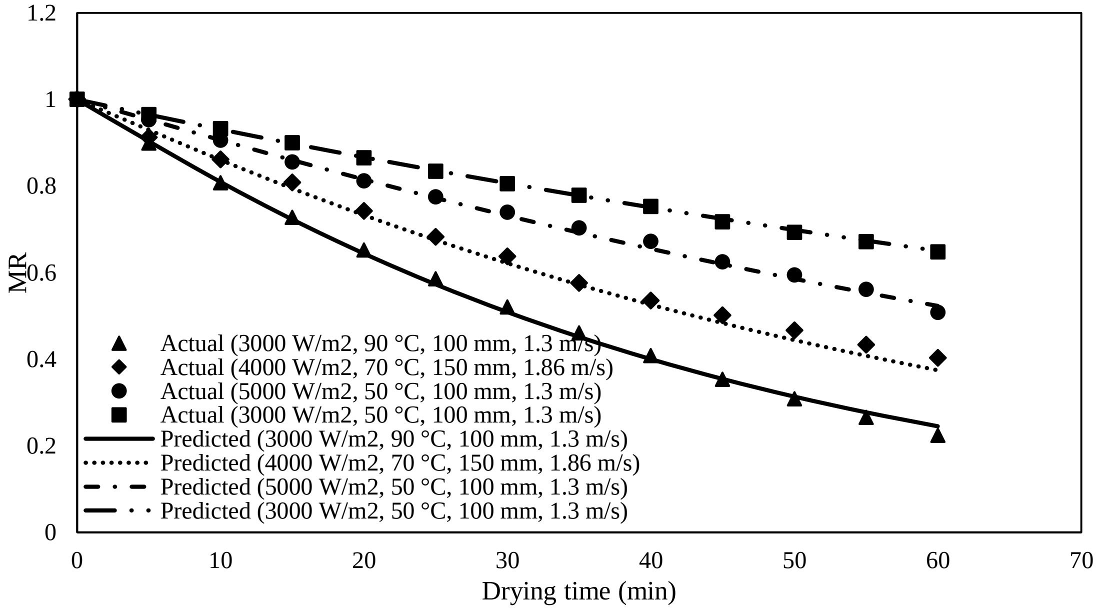


Figure 4.21 Moisture ratio at different conditions (exp no. 5, 1, 4, 3) of infrared hot air drying using Page model

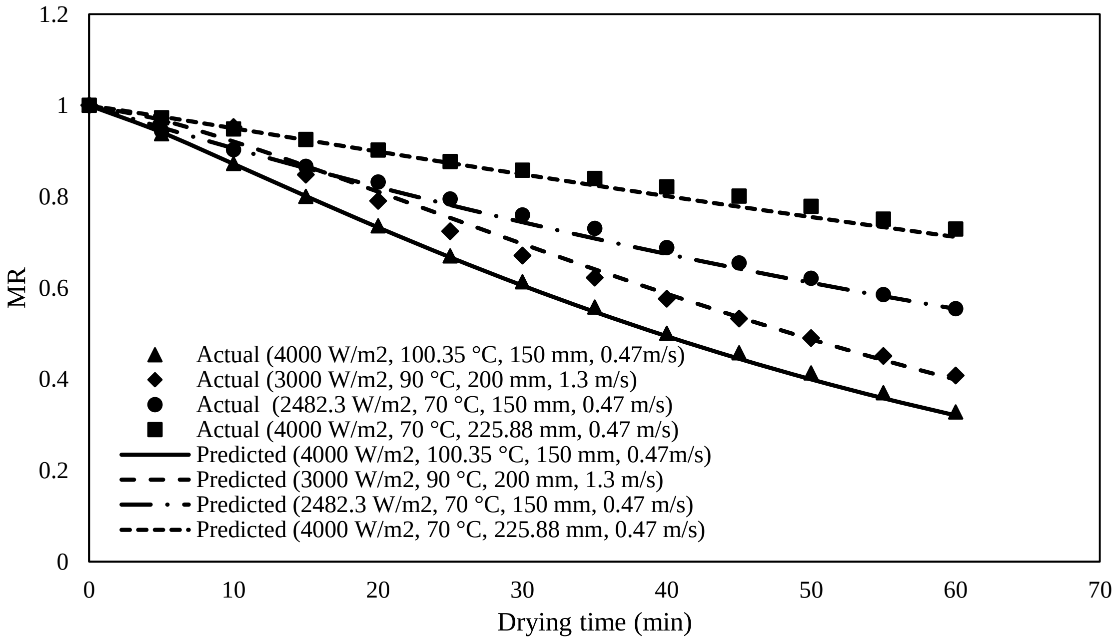


Figure 4.22 Moisture ratio at different conditions (exp no. 13, 9, 12, 15) of infrared hot air drying using Page model

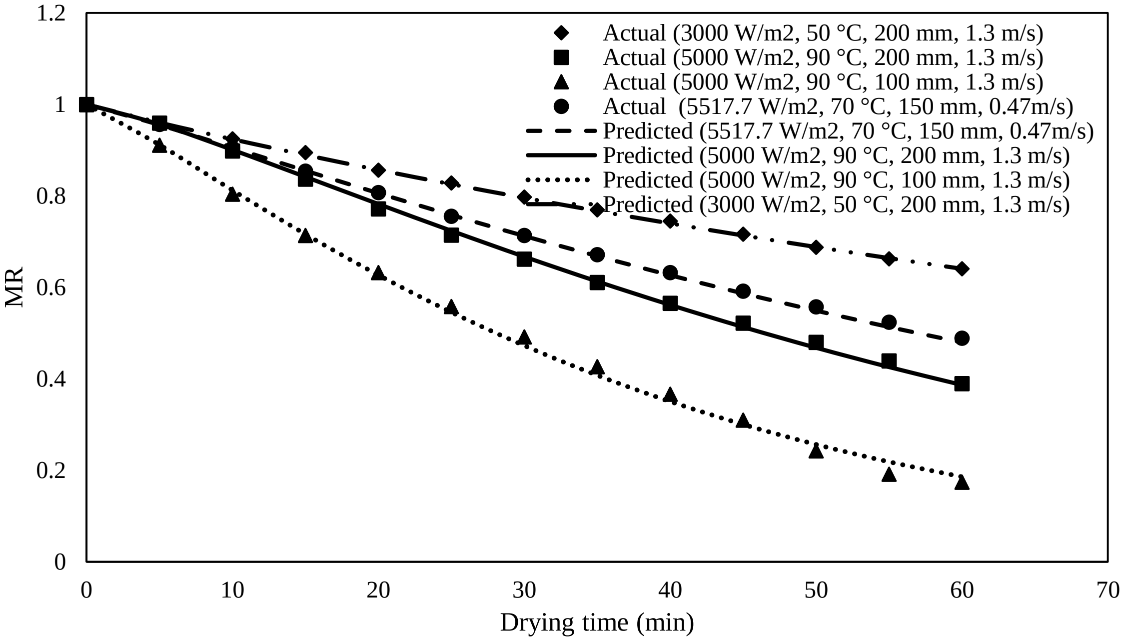


Figure 4.23 Moisture ratio at different conditions (exp no.7, 10, 6, 11) of infrared hot air drying using Page model

The perturbation analysis was performed to study the effect of all factors on responses. For, infrared drying the moisture content reached at 60 min (%wb) and the specific energy consumption were considered as response. The perturbation curves for moisture content are shown in Fig 4.24. It is apparent that the air temperature and distance were highly sensitive to moisture content than radiation intensity and velocity. The R^2 was

0.999. The regression equation for moisture content in terms of actual levels of variables is given below:

$$MC\ (\%wb) = 115.22 - 9.52 \times 10^{-03}\, I - 0.832T - 0.192D + 6.32V + 2.1\, ID + 3.85TD - 0.14\, TV \quad \ldots (4.4)$$

where I, T, D, V are intensity, temperature, distance and velocity respectively.

In case of SEC, the perturbation analysis showed the air temperature and velocity highly significant effect (Fig 4.25). From figure, it can be inferred that at high temperature the energy consumption was low due to the lesser drying time. Additionally, infrared drying was rapid thereby reducing the operating cost and time consumption of the drying process.

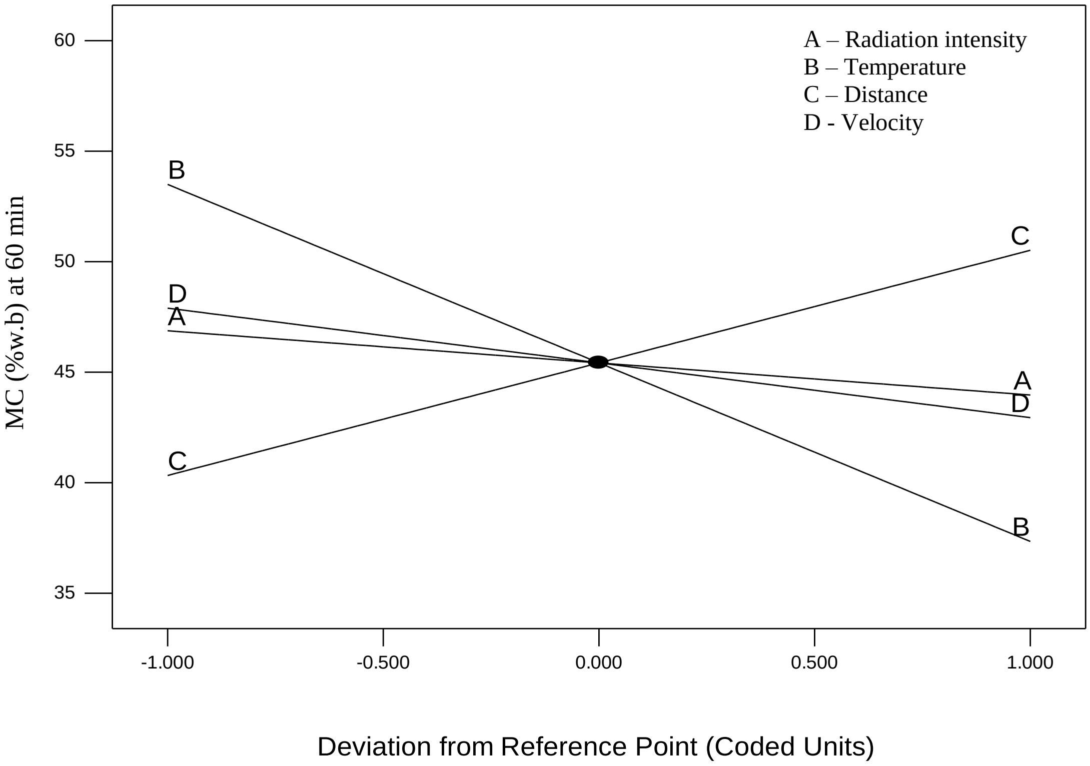


Figure 4.24 Perturbation for MC (%w.b) at 60 min during infrared hot air drying

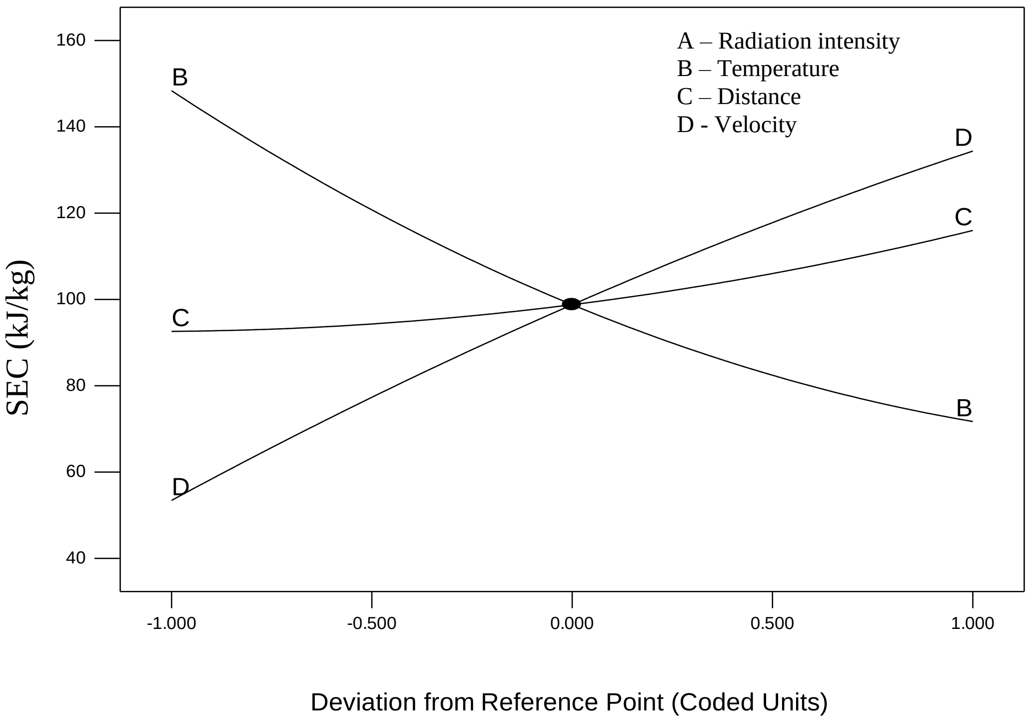


Figure 4.25 Perturbation for SEC (kJ/ kg) during infrared hot air drying

Optimisation of infrared air drying

Numerical optimisation was done based on the moisture reduced at 60 min. The independent parameters were set in range between -1 and +1. The suitable condition for radiation intensity, air temperature, distance between infrared lamp and product, and air velocity were found to be 3000 W/m^2, 90 °C, 100 mm and 1.50 m/s, respectively. The moisture content at this condition after 60 min was 28.8 % w.b with desirability 0.93. Fig 4.26 shows the desirability graph of optimisation. The infrared dried lemon slices using selected condition were further dried by microwave hot air.

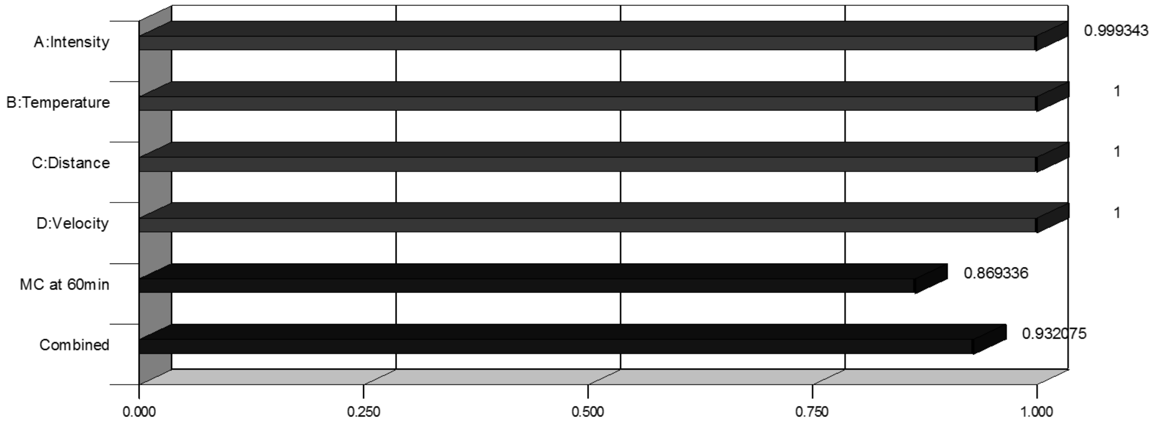


Figure 4.26 Desirability for optimised condition in infrared hot air drying

4.3.2 Microwave hot air drying

Finish drying of the infrared hot dried lemon slices was performed by microwave hot air. Since microwave penetrates inside the product and generates heat, the removal of moisture in the later stage is easier compared to other drying methods. Therefore, microwave hot air drying was chosen to reduce the moisture from 28.8 % to 9% approx. The drying rate curves for microwave hot air drying are depicted in Figs. 4.27 to 4.29. The moisture removal rate was high for microwave hot air than infrared hot air drying. The pulsating ratio for microwave was maintained at 1.5. Pulsation was given to avoid charring of the dried lemon slice. Due to the pulsation of microwave the deviated trend was evident in drying rate curves. The effect of different parameters of microwave hot air on drying kinetics was determined and found that as the power density, air temperature and velocity increased the moisture removal rate. Similar result on the effect of microwave and hot air was reported in literature [108, 130]. The apparent moisture diffusivity was higher for the combination of higher power density and higher temperature and values are given in Table 4.8. The moisture removal rate increased with increase in power density. The drying rate was increased because of a high amount of volumetric heat generation taking place by higher microwave power density, which results in higher product temperature in short time and higher moisture driving force towards the surface. The Page model showed good fit to microwave-hot air drying data with higher R^2 values in the range 0.991 to 0.999. The drying rate constant (k) of the Page model was in the range 0.0008 to 0.0044 min^{-1} (Table 4.8). It showed increasing trend with the increase in microwave power density since higher microwave power level would enhance the drying rate by generating more heat energy inside the product. Similar results were reported in literature for mushrooms and carrots [17, 166]. The moisture ratio curves are shown in Fig 4.30 to 4.32 and the trend was found to be similar to infrared hot air drying but the moisture removal rate was much higher in microwave drying.

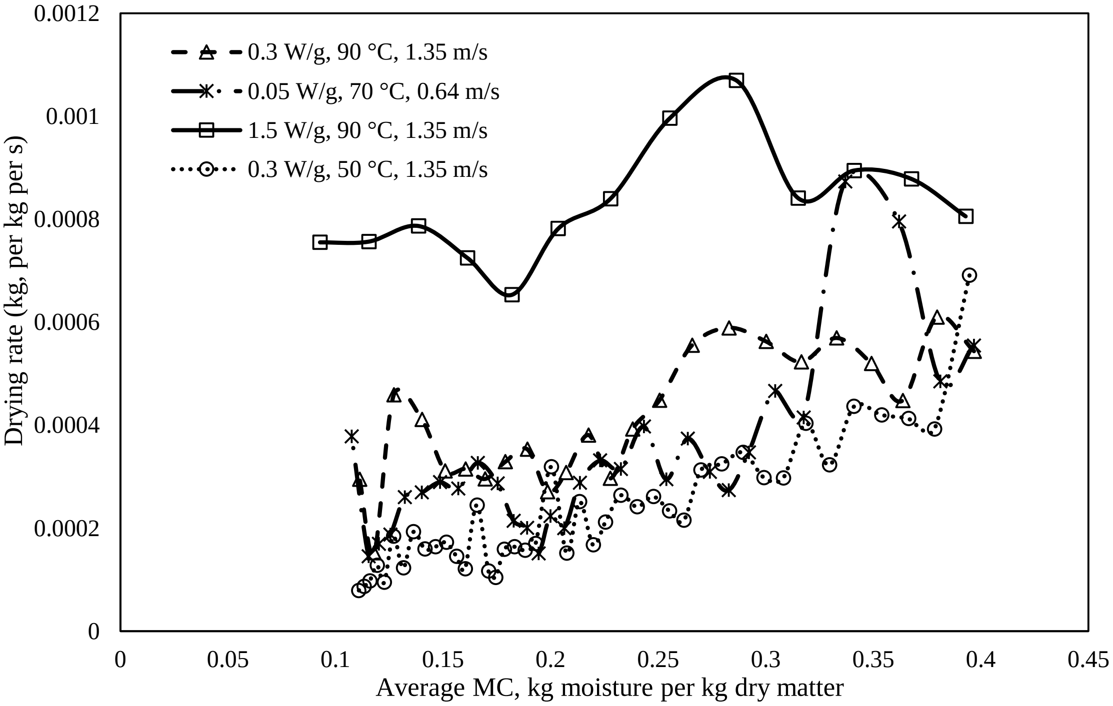


Figure 4.27 Drying rate (kg per kg per s) and average MC (kg per kg dry matter) at different conditions (exp no. 3, 5, 6, 8) during microwave hot air drying

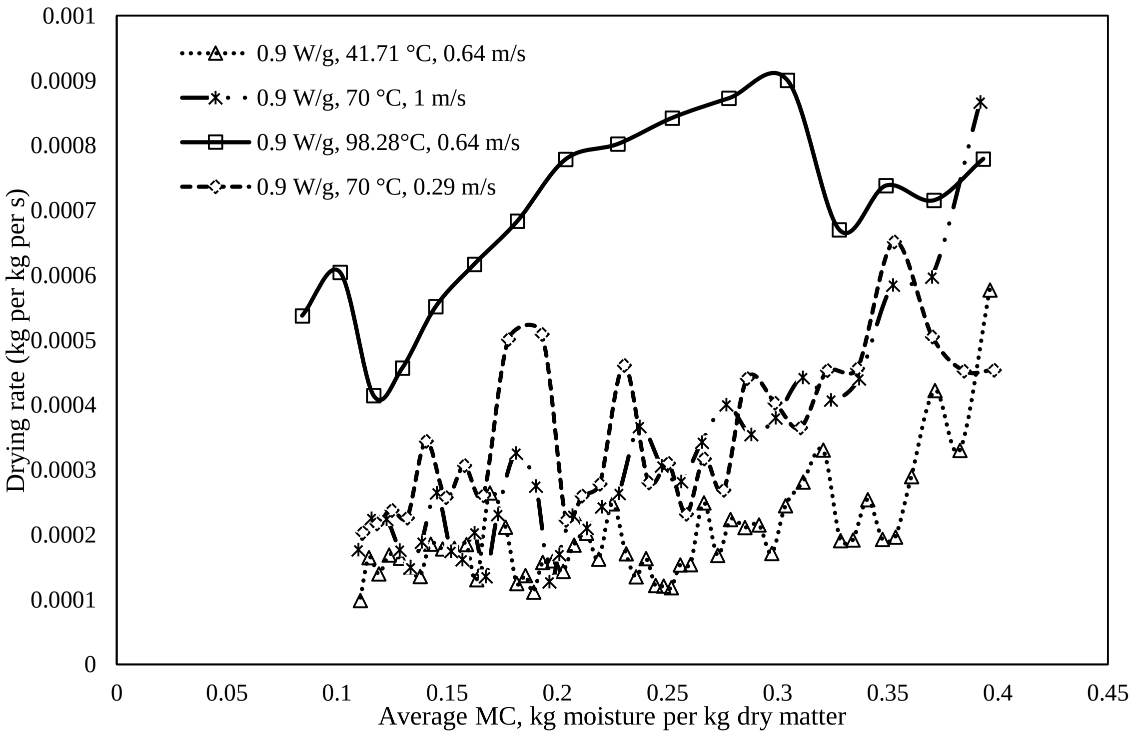


Figure 4.28 Drying rate (kg per kg per s) and average MC (kg per kg dry matter) at different condition (exp no. 2, 9, 10, 11) during microwave hot air drying

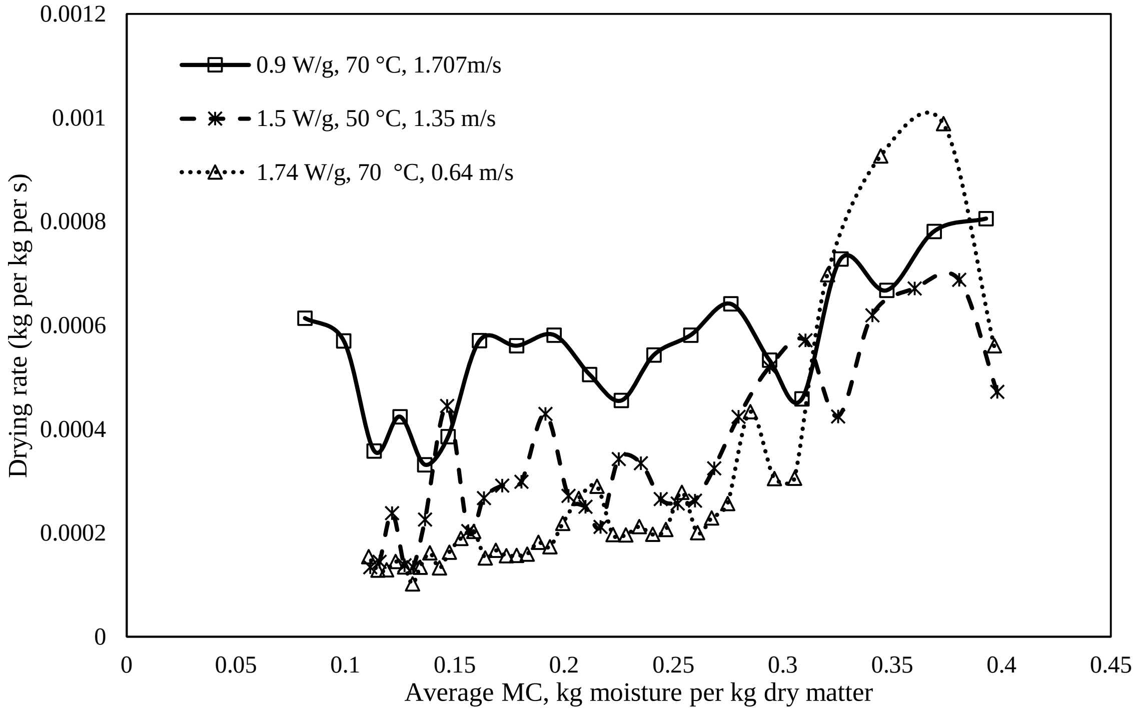


Figure 4.29 Drying rate (kg per kg per s) and average MC (kg per kg dry matter) at different conditions (exp no. 1, 4, 7) during microwave hot air drying

Table 4. 8 Apparent moisture diffusivity (m^2/s) and Page model constants during microwave hot air drying

S. No	**Power Density (W/g)**	**Temperature (°C)**	**Velocity (m/s)**	**Page model**			**Apparent moisture diffusivity (m^2/s)**
				k	**n**	**R^2**	
1	0.9	70	1.87	0.0014	1.09	0.9919	3.04×10^{-08}
2	0.9	70	0.87	0.0009	1.08	0.9965	2.03×10^{-08}
3	0.3	50	1.30	0.0022	0.89	0.9988	1.01×10^{-08}
4	1.5	50	1.30	0.0015	1.01	0.994	1.01×10^{-08}
5	0.3	90	1.30	0.0010	1.09	0.9986	2.03×10^{-08}
6	1.5	90	1.30	0.0008	1.26	0.9959	4.05×10^{-08}
7	1.74	70	1.30	0.0044	0.81	0.9777	1.01×10^{-08}
8	0.05	70	1.30	0.0018	0.97	0.9921	1.01×10^{-08}
9	0.9	98.28	1.30	0.0008	1.23	0.9944	3.04×10^{-08}
10	0.9	41.71	1.30	0.0017	0.89	0.9889	8.11×10^{-09}
11	0.9	70	0.48	0.0035	0.85	0.9984	1.01×10^{-08}

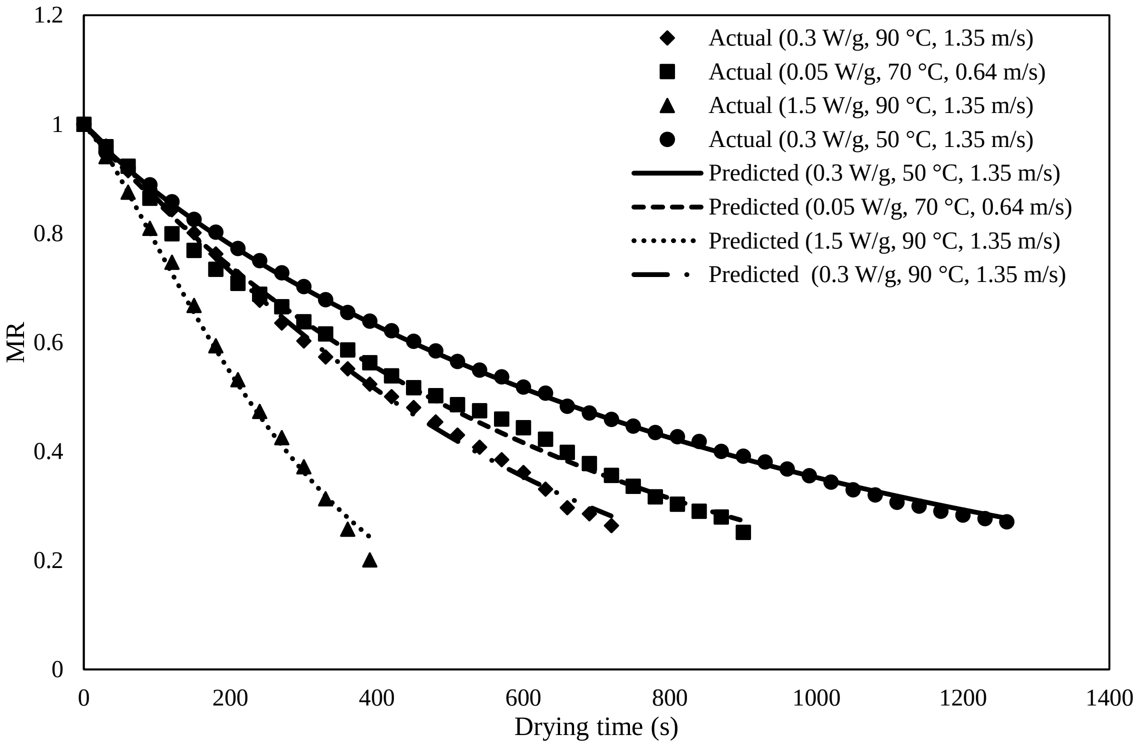


Figure 4.30 Moisture ratio at different conditions (exp no. 3, 5, 6, 8) of microwave hot air drying using Page model

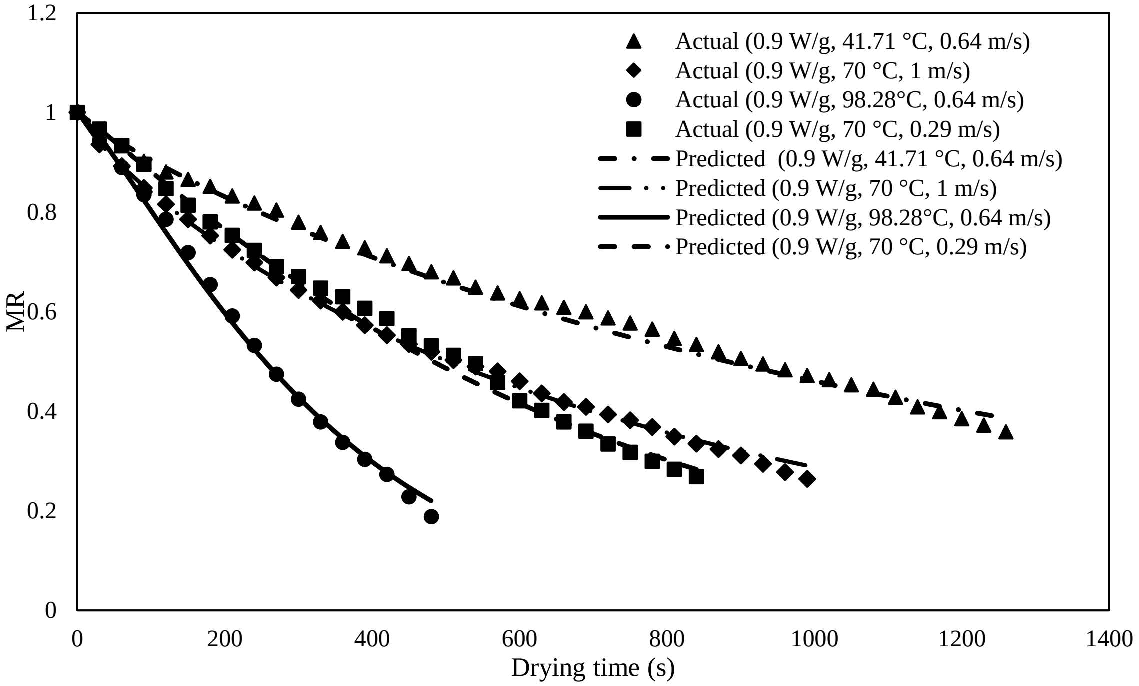


Figure 4.31 Moisture ratio at different conditions (exp no. 2, 9, 10, 11) of microwave hot air drying using Page model

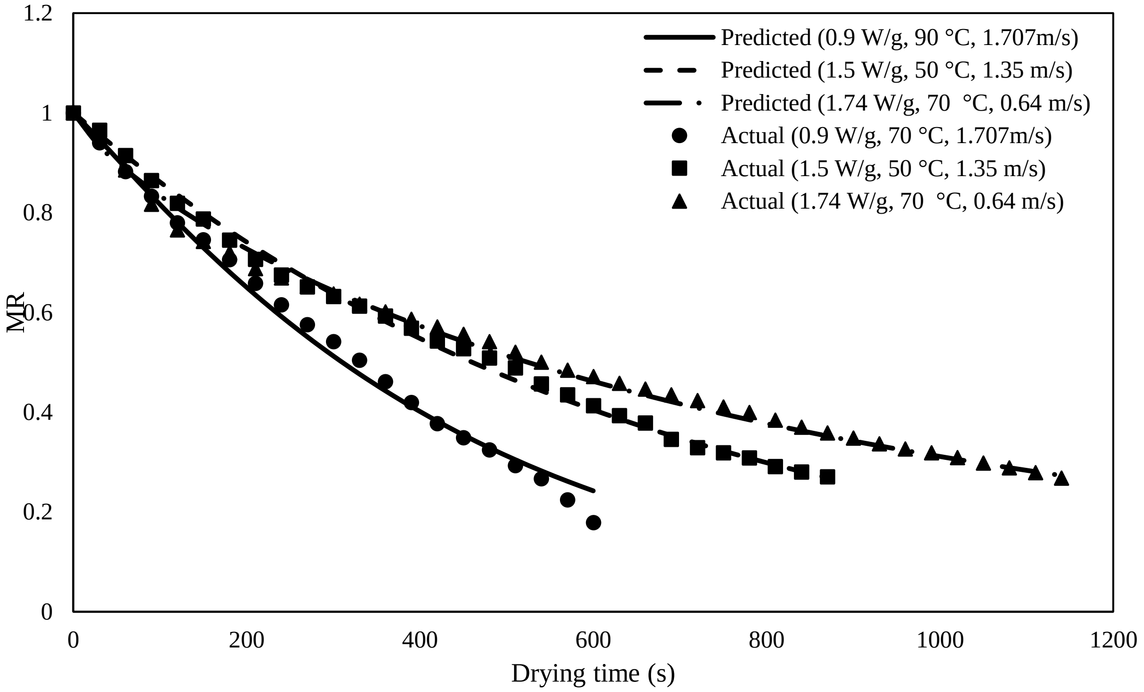


Figure 4.32 Moisture ratio at different conditions (exp no. 1, 4, 7) of microwave hot air drying using Page model

From the perturbation graph in Fig 4.33 it was observed that the variable power density alone had higher effect on ascorbic acid content of the product. When there was increase in power density the ascorbic acid content decreased drastically. The R^2 value was 0.86. The equation in terms of actual levels of variable for AA is given below:

$$\ln(AA) = 8.93 - 0.31\,PD - 0.21\,T - 3.03\,V + 1.37\,V^2 \quad \text{…. (4.5)}$$

where PD and AA are power density and ascorbic acid in lemon slices, respectively

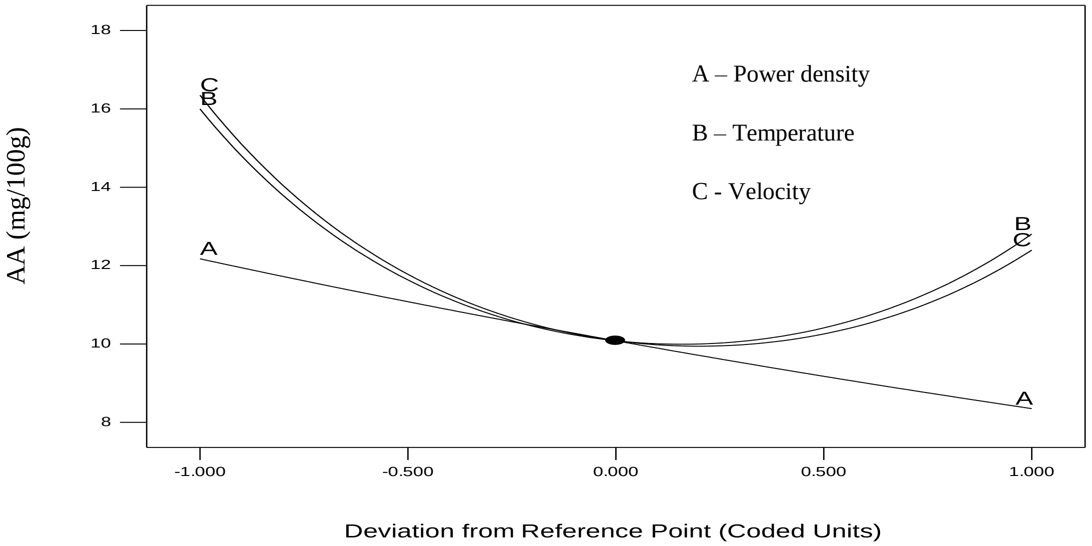


Figure 4. 33 Perturbation for AA (mg/100g) during microwave air drying

Table 4.9 ANOVA for microwave hot air drying

Source	Sum of Squares	df	Mean of Square	F value	p-value
			Ascorbic Acid		
Model	1.35	5	0.27	6.51	0.0302
A-PD	0.28	1	0.28	6.84	0.0474
B-Temperature	0.099	1	0.099	2.38	0.1834
C-Velocity	0.15	1	0.15	3.69	0.1129
B^2	0.66	1	0.66	15.93	0.0104
C^2	0.43	1	0.43	10.44	0.0232
Residual	0.21	5	0.042		
Cor Total	1.56	10			
			Drying Time		
Model	1.73	5	0.35	6.02	0.0354
A-PD	0.066	1	0.066	1.16	0.3309
B-Temperature	1.31	1	1.31	22.90	0.0049
C-Velocity	0.18	1	0.18	3.17	0.1352
B^2	0.075	1	0.075	1.30	0.3056
C^2	0.14	1	0.14	2.49	0.1754
Residual	0.29	5	0.057		
Cor Total	2.01	10			
			Specific Energy Consumption		
Model	2.88	5	0.58	5.56	0.0415
A-PD	0.048	1	0.048	0.46	0.5268
B-Temperature	1.20	1	1.20	11.56	0.0193
C-Velocity	1.22	1	1.22	11.72	0.0188
B^2	0.073	1	0.073	0.71	0.4386
C^2	0.42	1	0.42	4.04	0.1005
Residual	0.52	5	0.10		
Cor Total	3.40	10			

The perturbation curve for specific energy consumption of microwave hot air drying is shown in Fig 4.34. The R^2 value was 0.84. The effect of temperature was found to be significant. Similarly, the perturbation curve for drying time of microwave hot air to reach 9% mc (approx) in lemon slices is depicted in Fig 4.35, and the R^2 value was 0.85.

The temperature and velocity was found to be significant while the power density was insignificant. The regression equations in terms of actual levels for drying time and SEC are given below:

$$\ln(\mathrm{SEC}) = 2.24 - 0.12\ \mathrm{PD} + 0.02\ T + 3.49\ V - 2.91\times10^{-04} T^{2} - 1.35\ V^{2} \qquad \ldots (4.6)$$

$$\ln(\mathrm{Time}) = 2.43 - 0.15\ \mathrm{PD} + 0.02\ T + 1.28\ V - 2.94\times10^{-04} T^{2} - 0.79\ V^{2} \qquad \ldots (4.7)$$

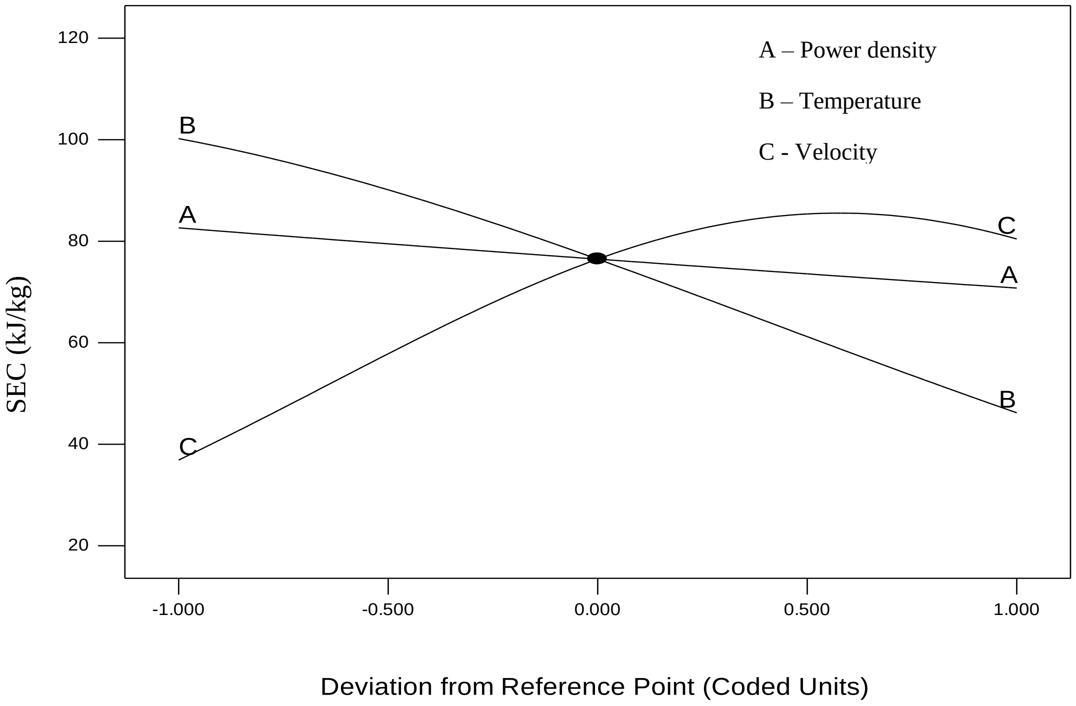


Figure 4.34 Perturbation for SEC (kJ/kg) during microwave air drying

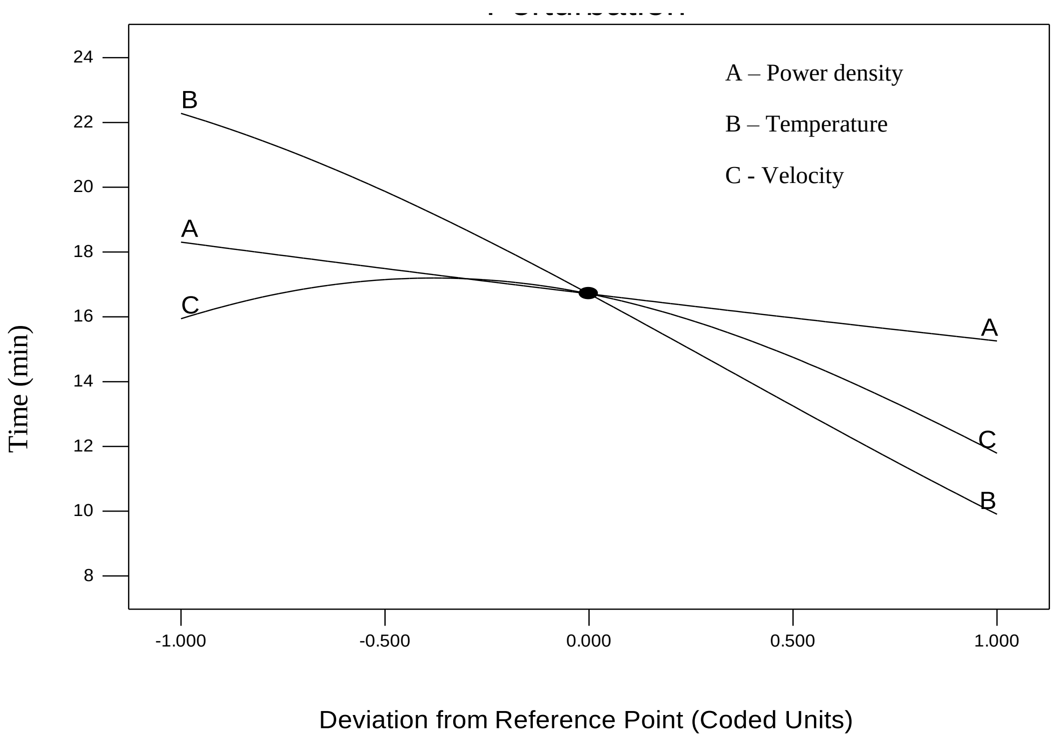


Figure 4.35 Perturbation for time (s) during microwave air drying

The other response variables of quality like color difference, rehydration ratio, hardness and sensory quality (overall acceptability) were found to be insignificantly affected during all parameters of microwave hot air drying. This was expected as the finish drying time is very less (maximum 23 min). Therefore, the quality degradation reactions might have not completely initiated as well as salt was protecting the quality.

Optimization

The optimised condition for finish drying using microwave hot air was selected by taking power density, temperature and velocity in between -1 and +1. The responses drying time (9% mc, w.b) and SEC were set at minimum and the retention of ascorbic acid content was set at maximum. The optimum conditions for power density, air temperature and velocity were 0.30 W/g, 89.9 °C and 0.50 m/s, respectively resulting into 24.13 kJ/kg SEC, 25.08 mg/100g AA and 10.3 min drying time. The desirability value was 0.86 and it is depicted graphically in Fig 4.36.

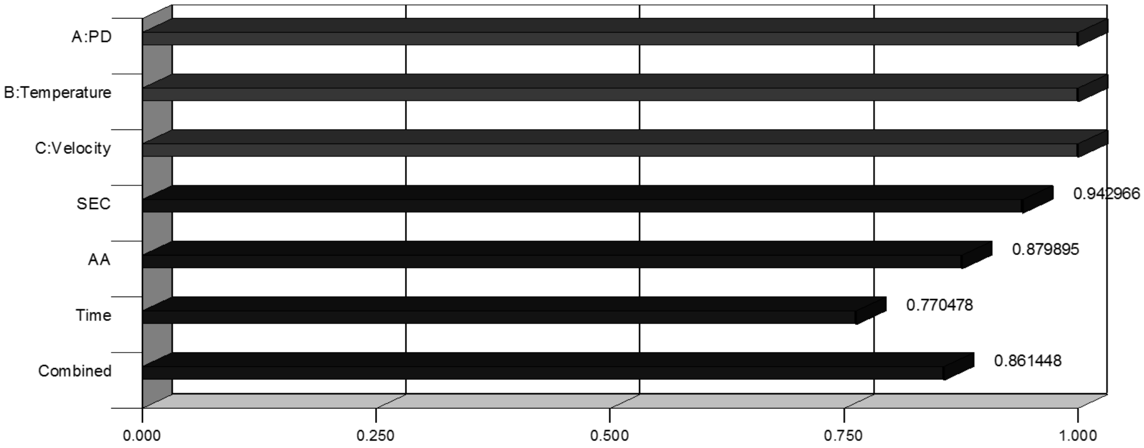


Figure 4.36 Desirability for optimised condition in microwave hot air drying

4.3.3 Sorption Study and Shelf life

The shelf life of the lemon slice dried by optimised conditions (osmotic, steam, infrared-hot air and microwave- hot air) was determined using the water activity analysis. The GAB model was solved by plotting a_w vs a_w/X (Fig. 4.37). The shelf life of 100 g dried lemon slices in 10 cm × 10 cm HDPE packaging at 40 °C storage temperature and 90% RH was found to be 80 days. The GAB model constants were 1.08, 0.02 and 2.08 for K, M and C, respectively.

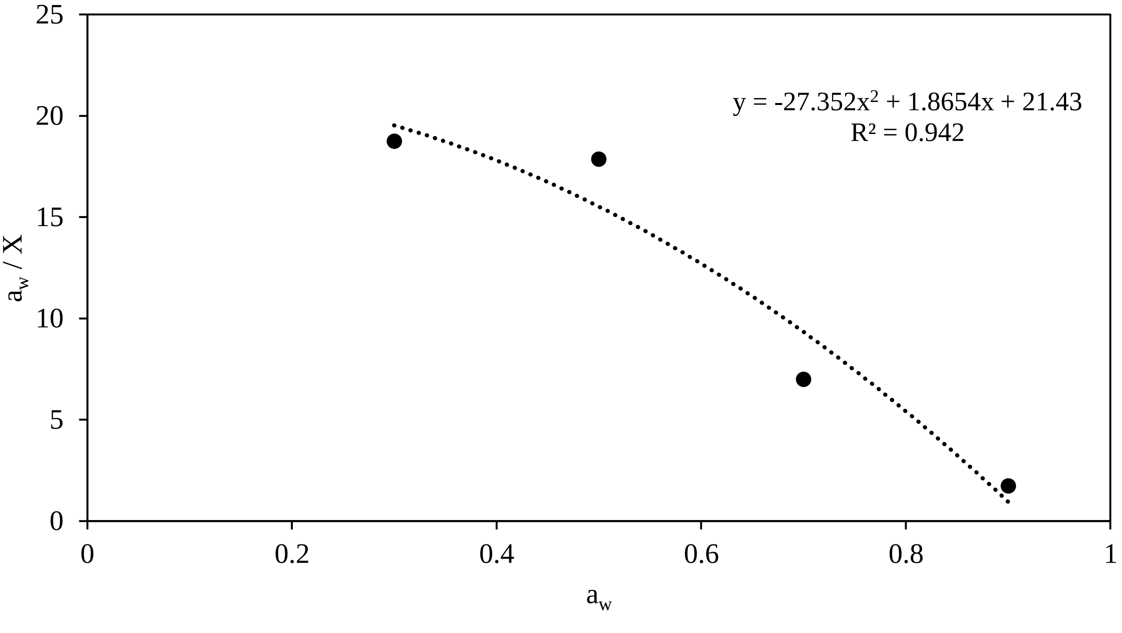


Figure 4.37 Sorption isotherm for dried lemon slices at different water activities

Chapter 5

Summary and Conclusions

The present investigation was undertaken to prepare dried lemon slices. For this purpose, the effect of osmotic, chemical and thermal pretreatments on ascorbic acid retention and enzyme (pectinestarase and peroxidase) inactivation of lemon was studied followed by infrared and microwave hot air drying.

In osmotic dehydration, the independent variables considered were NaCl concentration (5, 10, 15 and 20%), solution temperature (30 to 50°C) and time of treatment (30, 60, 90, 120, 150 and 180 min). The dependent variables were moisture loss, salt gain and solid loss. Best combination of osmosis was found at 20% NaCl concentration, 30 °C solution temperature for 180 min osmosis time. The optimum condition was based on maximum moisture loss (24.45, %IM), salt gain which reduces the water activity (39.49%, d.b) and minimum possible solid loss (14.48%, d.b). During osmotic dehydration the moisture content reduced from 87.5% to 65% (wb). Further, the enzyme inactivation and ascorbic acid retention for chemical and thermal pretreatments were analyzed. For chemical pretreatment, ascorbic acid concentration (0.1 to 0.5%), calcium chloride concentration (0.1 to 0.5%), solution temperature (20 to 80 °C) and pretreatment time (1 to 5 min) were considered as independent variables. Box Behnken Design was used for experiments. Twenty nine experiments were conducted for chemical treatment. For ultrasound, water and steam blanching, completely randomized design was used. The enzyme inactivation for chemical pretreatment was insignificant except ascorbic acid which had significant effect on peroxidase inactivation. Pectinesterase and peroxidase was not inactivated during ultrasound treatment. Water blanching and steam blanching had higher level of enzyme inactivation but the ascorbic acid loss was high in case of water blanching. Therefore, steam blanching for 1 min was selected as pretreatment to inactivate enzymes. In infrared hot air drying, the independent variables were radiation intensity (3000 to 5000 W/m^2), air temperature (50 to 90 °C), distance between lamp and product (100 to 200mm) and air velocity (0.5 to 1.5 m/s). Hybrid experimental design was used for infrared hot air drying. The optimized condition of infrared drying was based on the moisture reached after 60 min of drying. The optimum levels for radiation intensity, air temperature, distance between infrared lamp and product, and air velocity were found to be 3000 W/m^2, 90 °C, 100 mm and 1.50 m/s, respectively. The moisture content at this condition after 60 min was 28.8 % w.b. Further finish drying was done using microwave hot air. For microwave hot air drying, the independent parameters were power density (0.3 to 1.5 W/g), air temperature (50 to 90 °C) and velocity (0.5 to 1.5 m/s), hybrid design was used and the response variables were color difference, texture, shrinkage, rehydration ratio and sensory quality (overall acceptability). The optimum levels of power density, air temperature and velocity were 0.30W/g, 89.9 °C and 0.50 m/s, respectively with 24.13 kJ/kg SEC, 25.08 mg/100g AA and 10.3 min drying time. The drying characteristics were studied using Page model and moisture diffusivity was estimated. Sorption behavior of the lemon slices dried at optimum conditions of osmotic infrared and microwave hot air drying was studied using Guggenheim, Anderson and deBoer (GAB) model. GAB constants were estimated and shelf life of lemon slice was predicted at 40°C environmental temperature and 90 % relative humidity which was 80 days in HDPE.

Based on the results obtained from various experiments following conclusions were drawn within experimental ranges of the variables.

Osmotic pretreatment of lemon slices at various salt concentrations and solution temperatures was found to be advantageous compared to all other pretreatments. During osmotic dehydration the lemon’s own solid constituents gets lost along with the two –way counter current diffusion. The DHC of the lemon fruit and estimation of salt gain reveals the significant SL occurring during osmotic treatment particularly at higher temperatures. Therefore, high temperature is not recommended for product like lemon slice with low DHC. There is difference between the ML_{total} and ML_{os} when SL is present during osmosis. Also, the solid gain in the osmotic dehydration study should be corrected after estimation of exact SL as there is difference between solid gain without and with assumption of negligible SL.

The inactivation of the enzymes is necessary to retard the deterioration of the processed product. Effect of chemicals and ultrasound on enzyme inactivation in lemon was less compared to other pretreatments. Steam blanching should be performed after osmotic dehydration as the salt content in the slice avoids the cell disruption and maintains the intactness of the final product.

Infrared hot air drying of osmotically pretreated and steam blanched lemon slices is effective to reduce moisture content less than 30% w.b. during one hour without entering in drastically falling rate period. The higher moisture removal rate can be obtained at combination of high radiation intensity, high air temperature, low distance between IR source and lemon, and higher air velocity. Also, the infrared hot air drying reduces the specific energy consumption compared to conventional drying while maintaining the product quality.

Microwave hot air drying saves energy and drying time if applied as finish drying for osmotic-infrared hot air dried lemon slices. Higher moisture diffusivity and drying rate were obtained by the synergistic effect of higher power density, temperature and velocity, in final stage of drying where removal of moisture is very difficult. The quality of the product is also maintained with minimum specific energy consumption in microwave hot air drying due to very short drying time (10.3 min). From the sorption study it was found that the osmotic dehydration reduced the water activity of the product. Therefore, the final moisture content of the osmosed and dried lemon slices can be higher than dried product without osmosis.

Scope for Further Research

Further research is required on exploration of combined osmotic-infrared–microwave drying process for other fruits and vegetables. A cost benefit analysis of the conventional, microwave, infrared and the combination of any of these could be done.

Appendix

Sensory Evaluation Form

Name: - Date:-

Product: - Dried lemon slices Time:-

1. Kindly evaluate the product using 9 point hedonic scale followed by your valuable remarks and comments.
2. Remember you are the only one who can tell us what you like.
3. An honest expression of your personal feeling will help us.

Sensory characteristics / Samples No.	Appearance	Colour	Texture (firmness)	Flavor	Overall acceptability

9 - Like extremely

8 - Like very much

7 - Like moderately

6 - Like slightly

5 - Neither like nor dislike

4 - Dislike slightly

3 - Dislike moderately

2 - Dislike very much

1 - Dislike extremely

Remarks/Comments:-

Signature

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Dissemination

1. Internationally indexed journals (*Web of Science, SCI, Scopus, etc.*)

1. Deepika S. and P. P. Sutar. (2016). Osmotic Dehydration of Lemon (*Citrus limon* L.) Slices: Modelling Mass Transfer Kinetics Correlated with Dry Matter Holding Capacity and Juice Sacs Losses. *Drying Technology*, Accepted. Publisher Taylor and Francis.

2. Deepika S. and P. P. Sutar., Simultaneous Inactivation of Pectinesterase and Peroxidase Enzymes in Lemon (*Citrus limon* L.) Slices during Osmotic-Infrared-Microwave Hot Air Drying. *Innovative Food Science and Emerging Technologies*, Under Revision. Publisher Elsevier.

2. Book chapters

1. Deepika S. and P.P. Sutar (2015) Microwave Assisted Hybrid Drying of Food and Agricultural Materials. In *Drying Technologies for Foods: Fundamentals and Applications*, New India Publishing Agency, New Delhi, pp. 121-154

3. Conferences

1. Deepika S. and P.P. Sutar (2016) Post-Harvest Processing of Lemons to Inactivate Peroxidase and Pectinesterase Enzymes. Presented at all India seminar on "Post-Harvest Management of Fruits and Vegetables" during June 01-02, 2016, Kolkata.