BEHAVIOURS OF THERMOLABILE MATERIALS IN MICROWAVE VACUUM DEHYDRATION

(TINGKAH LAKU BAHAN SENSITIF HABA DALAM PENGERINGAN VAKUM GELOMBANG MIKRO)

MOHD ROZAINEE TAIB

PUSAT PENGURUSAN PENYELIDIKAN UNIVERSITI TEKNOLOGI MALAYSIA

2009

UNIVERSITI TEKNOLOGI MALAYSIA

BORANG PENGESAHAN LAPORAN AKHIR PENYELIDIKAN

TAJUK PROJEK :**BEHAVIOURS OF THERMOLABILE MATERIALS IN**
MICROWAVE VACUUM DEHYDRATION

C		
5	av	a

PROF. MADYA DR. MOHD ROZAINEE BIN TAIB (HURUF BESAR)

Mengaku membenarkan Laporan Akhir Penyelidikan ini disimpan di Perpustakaan Universiti Teknologi Malaysia dengan syarat-syarat kegunaan seperti berikut :

- 1. Laporan Akhir Penyelidikan ini adalah hakmilik Universiti Teknologi Malaysia.
- 2. Perpustakaan Universiti Teknologi Malaysia dibenarkan membuat salinan untuk tujuan rujukan sahaja.
- 3. Perpustakaan dibenarkan membuat penjualan salinan Laporan Akhir Penyelidikan ini bagi kategori TIDAK TERHAD.
- 4. * Sila tandakan (/)

SULIT

TIDAK

TERHAD

 $\sqrt{}$

(Mengandungi maklumat yang berdarjah keselamatan atau Kepentingan Malaysia seperti yang termaktub di dalam AKTA RAHSIA RASMI 1972).

TERHAD (Mengandungi maklumat TERHAD yang telah ditentukan oleh Organisasi/badan di mana penyelidikan dijalankan).

TANDATANGAN KETUA PENYELIDIK

Nama & Cop Ketua Penyelidik

Tarikh : _____

CATATAN : * Jika Laporan Akhir Penyelidikan ini SULIT atau TERHAD, sila lampirkan surat daripada pihak berkuasa/organisasi berkenaan dengan menyatakan sekali sebab dan tempoh laporan ini perlu dikelaskan

ACKNOWLEDGEMENT

I would like to acknowledge Ministry of Science, Technology and Innovation (MOSTI) for sponsoring this research through the granting of Science Fund (Project Vote 78223). Without the research fund, this project will not meet successful completion.

I would also like to extend my thanks to all the laboratory technicians who involved in assisting this project.

Head

Assoc. Prof Dr. Mohd Rozainee bin Taib

ABSTRACT

BEHAVIOURS OF THERMOLABILE MATERIALS IN MICROWAVE VACUUM DEHYDRATION

(Keywords: microwave, vacuum, dehydration, thermolabile, drying)

The objectives of this research were; 1) to design and develop a laboratory scale microwave vacuum dehydration testing rig for drying of thermolabile materials; 2) to study the drying mechanism of thermolabile material; 3) to determine the kinetics of microwave energy transfer in a vacuum testing rig. A laboratory-scale microwave vacuum dehydration system was successfully developed for dehydration of thermolabile materials. Microwave is recognized as a fast heating process characterised by volumetric heating. Contrary to conventional dehydration method which heats from the surface of material, microwave heats material from inside out. Incorporation of vacuum enhances the microwave dehydration process by allowing water to vaporize at lower temperature. Thus, dehydration of thermolabile materials can be achieved at low temperature and shorter time thereby preventing degradation of nutritional values and texture. Behaviours of thermolabile materials such as jackfruit, guava and papaya in microwave vacuum dehydration were studied. Operating parameters such as microwave power and vacuum pressure were found to influence the dehydration process. Increment of microwave power resulted in higher drying rates but increased risks of charring. However, the charring could be reduced with combination of microwave power. Products dried by combination of microwave power has lesser shrinkage and softer texture due to porous (puffing) structure induced by vacuum condition. It also possessed better reconstitution ability. In addition, papaya dried using microwave vacuum was found to have high enzyme activity retained.

Researcher:	Assoc. Prof. Dr. Mohd Rozainee bin Taib (Head)
E-mail:	rozainee@fkkksa.utm.my
Tel. No:	07-5535578
Vote No:	78223

<u>ABSTRAK</u>

TINGKAH LAKU BAHAN SENSITIF HABA DALAM PENGERINGAN VAKUM GELOMBANG MIKRO

(Kata kunci: gelombang mikro, vakum, dihidrasi, termolabil, pengeringan)

Objektif kajian ini adalah; 1) untuk merekabentuk dan membangunkan sistem pengeringan vakum gelombang mikro bagi pengeringan bahan sensitif haba; 2) untuk mengkaji mekanisme pengeringan bagi bahan sensitif haba; 3) untuk memahami kinetik penukaran tenaga gelombang mikro dalam sistem pengeringan vakum gelombang mikro. Sistem pengeringan vakum gelombang mikro berskala makmal telah berjaya dibangunkan untuk pengeringan bahan sensitif haba. Gelombang mikro dikenali sebagai proses pemanasan secara menyuluruh. Berlainan dengan kaedah pengeringan konvensional yang memanas dari permukaan bahan, gelombang mikro memanas dari dalam ke luar bahan. Gabungan vakum dan gelombang mikro meningkatkan proses pengeringan menggunakan teknologi tersebut dengan membolehkan air mengewap pada suhu yang lebih rendah. Oleh itu, pengeringan bahan sensitif haba dapat dicapai pada suhu rendah dan masa yang lebih pendek sekaligus mengelak penurunan kualiti pada nilai nutrisi dan tekstur bahan. Tingkah laku bahan sensitif haba seperti nangka, jambu dan betik dalam pengeringan vakum gelombang mikro telah dikaji. Parameter operasi seperti kuasa gelombang mikro dan tekanan vakum didapati mempengaruhi proses pengeringan. Peningkatan kuasa gelombang mikro dapat meningkatkan kadar pengeringan tetapi risiko charring adalah tinggi. Walaubagaimanapun, charring dapat dikurangkan dengan penggunaan kombinasi kuasa gelombang mikro. Produk yang dikeringkan menggunakan kombinasi kuasa gelombang mikro didapati mengalami kurang pengecutan dan tekstur yang lebih lembut disebabkan struktur poros yang dihasilkan oleh kondisi vakum. Produk turut mempunyai kebolehan untk kembali ke bentuk asal yang lebih baik. Di samping itu, betik yang dikeringkan menggunakan vakum gelombang mikro berjaya mengekalkan aktiviti enzim yang tinggi.

Researcher:Assoc. Prof. Dr. Mohd Rozainee bin Taib (Head)E-mail:rozainee@fkkksa.utm.myTel. No:07-5535578Vote No:78223

TABLE OF CONTENTS

CHAPTER TITLE

PAGE

1

ACKNOWLEDGEMENT	i
ABSTRACT	ii
ABSTRAK	iii
TABLE OF CONTENTS	iv
LIST OF TABLES	vii
LIST OF FIGURES	viii
NOMENCLATURE	X
LIST OF APPENDICES	xi

INTRODUCTION

1.1	1.1 Research Background	
1.2	Microwave Dehydration System	2
1.3	Advantages of Microwave Dehydration System	3
1.3	3.1 Significant of Volumetric Heating Imposed by Microwave	5
1.3	3.2 Incorporation of vacuum to microwave dehydration system	6
1.4	Problem Statement	7
1.5	Objectives of Research	8
1.6	Scopes of Research	8
1.7	Layout of Report	9
LITER	ATURE REVIEW	10
2.1	Fundamental Principles of Drying	10

2.1.1 Definition of Moisture Content	12
2.1.2 Sorption Isotherms	15
2.1.3 Drying Curves for Constant Drying Condition	16
2.1.4 Drying Kinetics	17
2.1.5 Rehydration of Dried Products	19
2.1.6 Shrinkage and Casehardening	20
2.2 Microwave Vacuum Drying	21
2.2.1 Fundamentals of Microwave Drying	21
2.2.2 Mechanism of Microwave Drying	21
2.2.2.1 Dipolar Rotation	23
2.2.2.2 Ionic Conduction	24
2.2.3 Physical Processes During Microwave Drying	25
2.2.4 Operational Constraint and Controversy of Microwave	26
2.2.5 Microwave Vacuum Dehydration	27
2.2.6 Material Study in the Design of Vacuum Microwave System	29
2.3 Sensory Evaluation	30
2.4 Mathematical Model	31
METHODOLOGY	33
3.1 Design of Microwave Vacuum Dehydration System	33
3.1.1 Calibration of Microwave Power	37
3.2 Material Preparation and Experimental Procedure	38
3.2.1 Jackfruit (Artocarpus heterophyllus Lam.)	38
3.2.2 Guava (<i>Psidium guajava</i> L.)	39
3.2.3 Papaya (Carica papaya L.)	39
3.3 Rehydration Test	40
3.4 Enzymatic Activity Analysis	41
3.4.1 Reagent Preparation	41
3.4.1.1 Concentrated Buffer (stock reagent)	41
3.4.1.2 Dilute Buffer Solution	42
3.4.1.3 Milk Substrate	42
3.4.1.4 Enzyme Buffer	42
3.4.2 Procedures	43
3.4.2.1 Milk substrate standardization	43

v

3.4.2.2	Enzyme preparation	44
3.4.2.3	Enzyme evaluation	44
3.4.2.4	Calculation	45

STUDY ON THE DRYING MECHANISM AND ENERGY TRANSFER OFJACKFRUIT AND GUAVA IN VACUUM MICROWAVE SYSTEM46

4.1 Microv	wave Power Output Measurement	46
4.2 Vacuu	m Microwave Drying Characteristics	47
4.2.1 Ja	ckfruit	47
4.2.1.1	Development of Proper irradiation Cycle	49
4.2.2 Gu	uava	52
4.2.2.1	Effects of Microwave Power Setting	52
4.2.2.2	Effects of Microwave Power Densities	55
4.2.2.3	Combination of Microwave Power Densities	58
4.3 Rehydr	ration	61
4.3.1 Res	ult of Rehydrated Jackfruit	62
4.3.2 Re	esult of Rehydrated Guava	63

STUDY ON THE DRYING MECHANISM AND ENERGY TRANSFER OF PAPAYA IN VACUUM MICROWAVE SYSTEM AND THE ENZYMATIC QUALITY 66

5.1 Combination of Microwave Power Mode		66
5.2	Effects of Sample Thickness	68
5.3	Rehydration Characteristic	70
5.4	Enzymatic activity	71
CONC	LUSION AND RECOMMENDATIONS	72
6.1	Conclusion	72
6.2	Recommendations	73
REFEI	RENCES	74
APPEN	NDICES	78

LIST OF TABLES

TABLE	NO. TITLE	PAGE
4.1	Power output for each microwave power setting	47
4. 2	Rehydration ratio and coefficient of rehydration	62
5.1	The effects of drying methods and papaya thickness on enzyme a	activity 71

LIST OF FIGURES

FIGURE NO

TITLE

PAGE

2.1	Wet basis and dry basis moisture content conversion curve	14
2.2	Typical sorption isotherms at a given temperature	15
2.3	Various type of moisture content (Mujumdar and Devahastin, 2000).	16
2.4	Typical drying curve (Geankoplis, 1995)	17
2.5	Typical drying-rate curve (Geankoplis, 1995)	17
2.6	Diagrammatic illustration of a plane monochromatic electromagnetic wa	ve.
	$E_{\rm o}$ and $H_{\rm o}$ are the amplitudes of the fields. (Schiffmann, R. F. 2006)	22
2.7	Typical drying curve for microwave drying	25
2.8	Vapour pressure versus boiling point of water (Felder and Rousseau, 198	86)
		28
0 1		2.4
3.1	Design of Microwave Vacuum Dehydration Experimental Set-up	34
3.2	The radiation cycles of various power levels of microwave (SHARP Mo	del:
	R-958A)	35
3.3	Manual mechanical moving tool of vacuum microwave dehydration systemeters	tem
		36
4.1	Dehydration curves for different power levels	48
4.2	Microwave irradiation of the combination of different power mode	50
4.3	The algorithm in determining proper irradiation cycle of microwave	
	vacuum drying	51
4.4	Drying curve for guava dried using high microwave power setting with	
	power density of 1kW/kg and vacuum pressure of -600mmHg	54

4.5	Drying curve for guavas dried using different microwave power setting	
	with power density of approximately 1kW/kg	55
4.6	Drying curves of microwave vacuum dried guavas dried using different	
	power densities	56
4.7	Drying curve for the MWVD of guava using the combination of differe	nt
	power settings	58
4.8	The algorithm in determining the suitable microwave power combination	on
	for the MWVD process	60
4.9	Drying curve for MWVD of guava using combination of different power	er
	settings	61
4.10	Effect of microwave power density on the rehydration ratio of microwave	ve
	vacuum dried guavas	63
4.11	Effect of different microwave power settings on the rehydration ratio of	
	microwave vacuum dried guavas	64
4.12	Effect of different drying conditions on the rehydration ratio	65
5.1	Drying curve of microwave-vacuum dried (MVD) papaya slices	67
5.2	Effect of papaya thickness on drying time during microwave-vacuum dr	ying
	(MVD) of papaya slices using combination of microwave power mode a	at -
	86.66 kPa system pressure.	69
5.3	Effect of microwave-vacuum dried papaya thickness on rehydration rati	o at
	30°C water temperature at -86.66 kPa system pressure.	70

NOMENCLATURE

Α	Exposed surface area for drying
C_p	Specific heat capacity of sample $(\frac{kJ}{kg.K})$
COR	Coefficient of Rehydration
m	Mass of sample (kg)
Р	Vacuum pressure (mmHg)
Q_{abs}	Energy absorbed by sample per unit time (KW)
R	Drying rate
RR	Rehydration Ratio
Т	Temperature
ΔT	Temperature rise in sample (^{o}C)
Δt	Drying time (s)
W	Weight of wet bone sample
W_{s}	Weight of dry sample
X	Free moisture content
X^{*}	Equilibrium moisture content
X_{A}	Residue Moisture Content

LIST OF APPENDICES

APPENDIX

TITLE

PAGE

A	Microwave Vacuum Dehydrated papaya	79
В	Calculations of Rate of Drying, Penetration Depth & Dielectric properties	81

CHAPTER 1

INTRODUCTION

1.1 Research Background

Biological and agricultural products are organic materials which cannot last longer and easily spoiled. Thus, the products need to be preserved in order to make it last longer for later usage. Drying or dehydration is one of the oldest methods in food preservation and its primary objective is to remove by evaporation a large fraction of the water present in the food. The reduction in moisture content inhibits or decreases microbial and enzymatic activity, which otherwise would lead to food damage or spoilage. Besides that, volumetric shrinkage and weight losses make the dehydrated products easily to be handled, transported and distributed. Dehydration provides opportunities for maximum convenience, flexibility and economics as industrial ingredients because dried products can be sized, shaped and formed to fit almost any requirement.

Dehydration of heat sensitive material such as agricultural and biological products is a difficult and cumbersome process. Conventional drying which is characterised as surface heating such as sun drying takes long drying time and the materials are exposed to elevated drying temperatures resulting in degradation of nutritional value such as vitamin C, beneficial enzyme, etc. It also results in unattractive appearance of final products in terms of colour, texture and structure.

Nowadays, hot air drying is mostly employed in dehydration process of food materials such as red pepper (Doymaz and Pala, 2002) and mushroom (Giri and Prasad, 2007). Thermal damage incurred by a product during drying is directly proportional to the temperature and drying period. The high temperature and long drying time associated with hot air convective drying tend to cause heat damage and hence adversely affect texture, colour, flavour and nutritious properties of the dehydrated product. Besides, high temperature and "outside to inside" heat transfer mechanism cause the material to be dried tend to shrink and in turn result checking, cracking and warping (McCabe *et al.*, 1993). Shrunken cell makes the drying rate decrease and hence does prolong the drying period, besides contributes to the poor appearance of dehydrated product. There is no ideal temperature for hot air drying. High temperature associated with low relative humidity cause case hardening, while low temperature or high relative humidity lead to long drying time and the fruit may spoil before dried. The temperature between 35 and 63 $^{\circ}$ C is satisfactory for most fruit processing (Kordylas, 1990).

1.2 Microwave Dehydration System

Due to the disadvantages imply by conventional convective drying, a better drying process is developed to improve drying process. Drying by means of microwave is increasingly popular in spite speculation regarding radiation effect imposed by microwave. Microwaves are electromagnetic waves with frequencies ranging between 300 MHz and 300GHz. Materials that absorb microwave energy are known as dielectrics and they are characterized as non-conductive materials. However, dielectrics can be heated if the molecules possess an asymmetrical structure. Water is the typical case of such molecule. In the presence of an electrical field these asymmetric or polar molecule will align themselves according to the electrical field. Contrary to conventional heating process, these movements result in internal heat development and an increase of the temperature of the material.

Microwave drying is a fast drying process, where electromagnetic energy is transferred to material to be dried directly without passing through heating medium. Microwave has the advantage of high penetration to the solid material and this yields uniform drying throughout the entire portions of material. Besides, microwave is preferentially absorbed by the water molecules and thus the energy losses to nontargeted material heating are no significant. However, microwave has the disadvantage of non-homogeneous energy distribution in the cavity that creating non-uniform heating (Drouzas *et al.*, 1999). Continuous microwave irradiation causes the drying temperature to rise uninterrupted and consequently results burning or overheating. The boiling point of water in microwave drying is slightly higher than normal condition. This makes process control of microwave irradiation is becoming more complex owing to high tendency of overheating as the results.

1.3 Advantages of Microwave Dehydration System

Applications of microwaves in dehydration process have been widely investigated due to its unique features in energy transfer. There are many advantages of applying the microwaves technology as stated below:

- a) Volumetric heating: Microwave-assisted drying is characterised as volumetric heating in which the microwave energy is dissipated as heat to the total volume of mass rather than a surface heating as conventional convective drying (Roberts, 1999).
- **b) Uniform heating:** Microwaves pass through most materials uniformly (if the material thickness is relatively smaller than

penetration depth). All parts of the material are heated simultaneously and resulting a uniform heating (if the material is homogeneous), avoiding the large temperature gradients (Schiffmann, 1987; Baysar, 1992).

- c) Selective heating: Microwaves selectively heat up the lossy dielectric materials (such as water, etc.) of a matter composed of different dielectric properties, avoids heating of the air, walls or other parts (Schiffmann, 1987; Baysar, 1992).
- **Internal vaporisation:** Microwaves couple directly into the core of the material and vaporise the moisture inside. As a result, an internal total gas pressure gradient builds up and most of the moisture removed as a vapour (Feng, 2000).
- e) Energy efficiency: Microwave energy tends to couple toward the wetter areas, heat losses are considerably reduced. By selectively heating the moisture, which eliminates the energy wastage to heat up the environment or other parts, leads to significant energy savings. In contrast, microwave energy is 'cool' and plant cooling savings can be realised (Schiffmann, 1987).
- f) Time saving: As a result of the volumetric heating and internal vaporisation, the drying time can be reduced substantially. This is more dramatically observed for materials having poor thermal diffusivity (Stuchly and Stuchly, 1983; Feng, 2000).
- g) Space saving: A substantial reduction in process time due to the more rapid heating, a long, stretched out processing lines can be compressed into a fraction of the space (Decareau and Peterson, 1986).
- **Economic saving:** In view of the cost of plant construction, energy consumption and process time, it can represent substantial economic saving (Decareau and Peterson, 1986).
- Process improvement: Drying can be done at lower temperature, no need to maintain high surface temperatures, this make possible to eliminate case-hardening, surface cracking or overheating, which are commonly occurred with conventional heating methods (Schiffmann, 1987). Microwave heating preserves product quality. This was

successfully reported in food drying, such as apples, potato chips, strawberries and carrots by a combined microwave-assisted convection process. Microwave dried products show a higher rehydration values (Drouzas & Schubert, 1996; Litvin *et al.*, 1998; Funebo & Ohlsson, 1998; Lin *et al.*, 1998; Nijhuis *et al.*, 1998; Feng and Tang, 1998; Maskan, 2000).

- j) Instant process control: The instantaneous on-off operation of microwave generation and the ability to change the microwave output power make a high degree of flexibility and process control is easily achievable (Stuchly and Stuchly, 1983; Schiffmann, 1987).
- k) Environmental friendly. Microwave heating is non-polluting; at least it removes the source of pollution from the processing plant to the electric generating station.

1.3.1 Significant of Volumetric Heating Imposed by Microwave

As compared to conventional drying methods, microwaves penetrate into much greater depths. That is called volumetric heating which has the following advantages:

- A temperature gradient directed towards the surface, such as temperatures inside are higher than on the outside giving rise to a higher partial pressure that drives the evaporating liquid to the surface.
- 2. Consequently, the superficial layer does not dry out completely and the surfaces remain permeable.
- The liquid evaporating inside the product is emitted through the pore structure of the solid material's macro-capillary system, resulting in a high drying velocity.

- 4. The heating of water and most organic solvents occur selectivity due to the greater dielectric losses of water as compared to the product to be dried.
- 5. Swift and thorough drying of moist products with low thermal conductivity.
- 6. Stationary drying of thick layers without frictional losses.
- 7. High total efficiency of energy application.
- 8. High-speed control of the energy transport.
- 9. Short processing times, such as suitable for automated manufacturing.

1.3.2 Incorporation of vacuum to microwave dehydration system

Microwave can do only raising the product temperature in order to change moisture to vapour, but it requires other technique to remove the vapour. Convective drying is a conventional way for removing vapour. Another unique technique is the vacuum. Vacuum microwave drying offers an alternative way to improve quality attributes. Low pressure associated with this technology reduces the boiling point of water to the level at where the effects of thermal degradation and discoloration are not significant (Drouzas *et al.*, 1996). High vacuum level causes the structure of material expand, high penetration of microwave into the interiors, fast mass transfer of moisture and consequently, shortening the drying time (Lin *et al.*, 1998).

However, due to high costs and longer process, the application of vacuum in drying process is still limited. The idea to combine fast heating of microwave and low temperature processing of vacuum has been investigated by a number of researchers. The results show that the vacuum-microwave drying is an alternative way to improve the quality of dried products. Thus, a hybrid drying technique of the microwave vacuum dehydration was introduced in this study. A vacuum drying technology has many advantages such as follow:

- 1. Low drying temperatures treat the product gentle
- 2. No oxygen attack for the product.
- 3. Highly nutritious instant properties.
- 4. Better flavour.
- 5. Less hydroscopic final product.
- 6. Minimal product losses.
- Static drying of thick layers without frictional losses, therefore no mechanic stress for the product.

1.4 Problem Statement

Conventional drying of thermolabile materials such as biological and agricultural products results in undesirable changes in terms of physical and chemical properties, affect the quality of the preserved product, which leads to reduction of its commercial value. Besides, conventional drying process is inefficient because of the requirement of long hour processing.

Due to these drawbacks, an alternative way is desirable to dehydrate the thermolabile materials and to improve the quality of dehydrated products. Incorporation of vacuum to microwave system is found as an effective way to overcome the drawbacks of conventional drying. The vacuum microwave system combines the advantages of vacuum drying and microwave application where the presence of vacuum condition in drying system results in a lower boiling point of water, thus allowing water to vaporize at low temperature. Drying at low temperature leads to prevention of heat damage and nutritional losses. In addition, low-temperature vaporization also leads to lower rate of oxidation resulting in an improvement of colours and flavours of dehydrated products.

1.5 Objectives of Research

The purpose of the research work is to study the behaviours of thermolabile materials in microwave vacuum dehydration. The objectives of the research were:

- a) To design and develop a laboratory scale microwave vacuum dehydration testing rig for drying of thermolabile materials.
- b) To study the drying mechanism of thermolabile material.
- c) To determine the kinetics of microwave energy transfer in a vacuum testing rig.

1.6 Scopes of Research

The scopes of this research were:

- a) Design and development of laboratory scale microwave vacuum dehydration testing rig for drying of thermolabile materials
- Investigation of drying mechanism of three thermolabile materials such as jackfruit, guava and papaya.
- c) Determination of the kinetic of microwave energy transfer in the designed vacuum microwave dehydration testing rig.

1.7 Layout of Report

This report presents research works on behaviours of thermolabile materials in microwave vacuum dehydration.

Chapter 1

This chapter presents research background, problem statement, justification and objectives of the research.

Chapter 2

This chapter presents literature review regarding dehydration of material using microwave vacuum technology.

Chapter 3

This chapter describes experimental set up and calculations in this research.

Chapter 4

This chapter presents the result of the drying mechanism of jackfruit and guava, as well as the energy transfer in the microwave vacuum system. Besides, sensory evaluation test and rehydration test are evaluated to compare the quality attributes of dehydrated products such as colour, texture, flavour and aroma, in order to investigate the feasibility of vacuum microwave drying for these particular thermolabile materials.

Chapter 5

This chapter presents the drying mechanism of papaya in microwave vacuum system. The quality of dried papaya in term of enzymatic activity is discussed.

Chapter 6

This chapter presents conclusion and recommendations derived from this research works.

CHAPTER 2

LITERATURE REVIEW

2.1 Fundamental Principles of Drying

Drying or dehydration, in general, usually means removal of relatively small amounts of water from material (Geankoplis, 1995). The term drying is also used to refer to removal of other organic liquids from solids. Drying is perhaps the oldest, most common of chemical engineering unit operations. Drying processes can be classified as batch, where the material is fed into the system for a given period of time, or as continuous, where the material is fed and removed continuously. It competes with distillation as the most energy consuming unit operations in the process industries due to the high latent heat of vaporisation and inefficiency of energy transfer using convective hot air as the most common heating medium.

In order to reduce the intensive thermal load for vaporisation, dewatering which minimising water content of the wet feed materials is thus important prior to the drying by mechanical means using vacuum or pressure filters, decanters, centrifuges, etc. (Mujumdar, 1996). Mechanical dewatering gives an economical option in removing water than thermal means. Nevertheless, thermal drying is applied when lower final moisture content is required, a condition that could not be achieved through mechanical dewatering.

Drying is a complex process which involving transient heat and mass transfer simultaneously. Generally, convective drying is applied in most conventional method, which characterised as surface heating. Heat is transferred by convective hot air from surroundings to the process material through its surface. Thus, surface area plays the main role in the external transport phenomena. From surface, by conduction, heat further transfer into the core layers of the material. The internal transport phenomena are determined entirely by the inherent physical properties of the material: its specific heat, thermal conductivity and density, which lumped together into one parameter, the thermal diffusivity (Meredith, 1998). The drying force for the overall heat transfer from surroundings to the core layers of the material is the negative temperature gradient (outside is hotter than inside) between these two mediums. Thus, conventional drying methods always suffered from the high surrounding temperature required for heat transfer.

In contrast, moisture transfer is in the opposite direction from within the material toward the surface, where evaporation takes place to remove the moisture by convection to the surroundings. The driving forces and mechanisms of mass transfer for moisture movement may be complex as summarised below (Fortes and Okos, 1980; Chirife, 1983; Mujumdar and Devahastin, 2000):

- a) Liquid diffusion due to moisture concentration gradients.
- b) Vapour diffusion due to partial vapour pressure gradients caused by temperature gradients.
- c) Liquid movement due to capillary forces by the interfacial tension between the water and solid.
- d) Knudsen diffusion, if drying takes place at very low temperatures and pressures, e.g. in freeze drying.

- e) Liquid or vapour flow due to the shrinkage and total pressure gradients.
- f) Flow due to the vaporisation-condensation sequence.
- g) Liquid movement due to gravitational forces, but is negligible when the pore dimensions inside material are very small as in most foodstuffs.
- h) Surface diffusion, possible although not proven nor taken into account by existing drying models.

All these possible moisture movement mechanisms are all lumped into a single measurable parameter, the effective moisture diffusivity, regardless of the dominating mechanism (Feng *et al.*, 1999). The opposite direction of heat and mass transfer is not favourable for energy coupling that makes the drying an energy consuming process. A parallel direction of heat and mass transfer will occur if microwave heating is employed, which will be discussed in subsequent section.

2.1.1 Definition of Moisture Content

Before further discussions on the drying kinetics, the definitions of moisture content are needed to be clear and well defined for avoiding from misinterpreting, since there are many definitions quantifying the moisture content of materials are in used. Moisture content usually expressed as the mass of moisture per unit weight of dry basis (d.b.) or wet basis (w.b.). Moisture content on a volumetric basis is rarely employed (Gardner, 1971). Moisture content on wet basis, $M_{w.b.}$ is the ratio of moisture to total weight, but the more scientific basis of moisture content on dry basis, $M_{d.b.}$ or simply referred to M is adopted in drying calculations and throughout the texts. The equations of these two definitions are shown as below:

$$M_{w.b.} = \frac{W_{w}}{W_{w} + W_{s}} \times 100\%$$
(2.1)

$$M_{d.b.} = \frac{W_{w}}{W_{s}} \times 100\%$$
 (2.2)

where, W_s and W_w are the weight of dry solid and water respectively. The $M_{w.b.}$ is non-linear, while $M_{d.b.}$ is a linear function to the evaporated moisture. Thus, the drying rate can be easily interpreted by observing directly from the drying curve gradient of $M_{d.b.}$. The relationship between wet and dry basis can be expressed as Equation (2.3) and graphically in Figure 2.1.

$$M_{w.b.} = \frac{M_{d.b.}}{100\% + M_{d.b.}} \times 100\%$$
(2.3)

The above discussed moisture content is based on the total moisture basis without consider the free and equilibrium moisture of the solids at a given condition. Because of the equilibrium moisture content is relatively temperature and humidity dependency for different materials (Geankoplis, 1995); thus, it is seldom been used in many journal papers and technical reports.

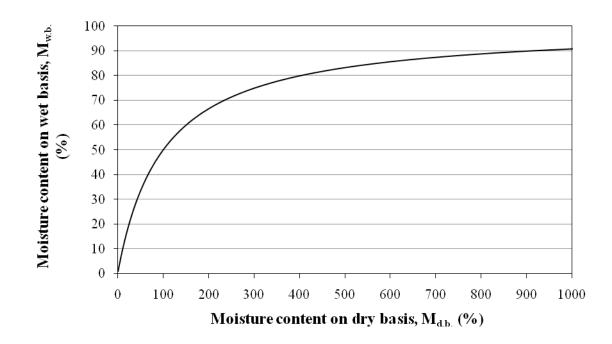


Figure 2. 1: Wet basis and dry basis moisture content conversion curve

When a wet solid is brought into contact with air-vapour mixture at a given temperature and humidity, after exposure for sufficiently long for equilibrium to be reached, the solid will be remained at certain moisture content, which so-called equilibrium moisture content at that given condition, which expressed by:

$$M^* = \frac{W^*}{W_s} \times 100\%$$
 (2.4)

where, W^* is the equilibrium moisture at that given condition.

A free moisture content, M_f is representing the moisture content at a given equilibrium state, as expressed below:

$$\mathbf{M}_{\mathrm{f}} = \mathbf{M} - \mathbf{M}^* \tag{2.5}$$

where, M is moisture content (d.b.) and M^{*} is equilibrium moisture content at that given condition.

2.1.2 Sorption Isotherms

A different value of equilibrium moisture content is obtained according to the direction of sorption from which equilibrium is approached. A sorption isotherm is simply a curve showing the equilibrium moisture content versus the relative humidity at a given temperature. By exposing the solid to air-vapour mixture of increasing humidity to adsorb moisture gives the adsorption isotherm and of decreasing humidity to dry gives the desorption isotherm. Apparently, the latter is of particular interest in a drying process. Figure 2.2 shows the typical sorption isotherms at a given temperature.

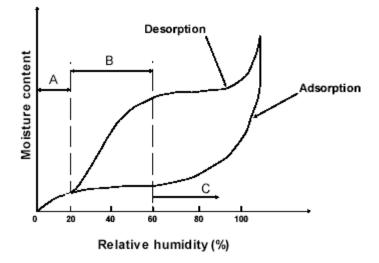


Figure 2. 2: Typical sorption isotherms at a given temperature (Mujumdar and Devahastin, 2000).

There are three distinct regions, A, B and C in an isotherm, which are indicative of different water binding mechanisms at individual sites on the solid matrix (Fortes and Okos, 1980; Mujumdar and Devahastin, 2000). In region A, water is tightly bound to the sites and unavailable for reaction. In region B, water is more loosely bound in smaller capillaries. In region C, water is relatively free and even more loosely held in larger capillaries. Generally, water in these regions up to the 100% relative humidity line, is so-called bound water or bound moisture. Due to this water is physically and/or chemically bound to the solid matrix, so it exerts a vapour pressure lower than that of pure water at the same temperature. The excess water that indicated by intersection with the 100% relative humidity line is called unbound water or unbound moisture. The unbound water exerts vapour pressure as high as that of liquid water at the same temperature. Figure 2.3 shows the various type of moisture content.

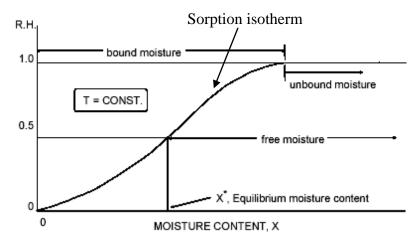


Figure 2. 3: Various type of moisture content (Mujumdar and Devahastin, 2000).

2.1.3 Drying Curves for Constant Drying Condition

In drying, it is necessary in most cases to obtain some experimental data for curve plotting. It is desired to design the drying process. There are two typical curves, one is moisture content versus time (drying curve) and another is drying rate versus moisture content (drying rate curve). From the curves, the different period of drying process can be observed. Figure 2.4 and Figure 2.5 show the typical drying curve and drying rate curve respectively.

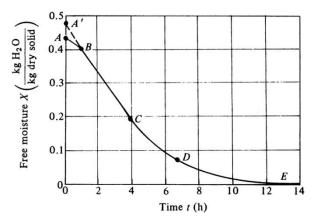


Figure 2. 4: Typical drying curve (Geankoplis, 1995)

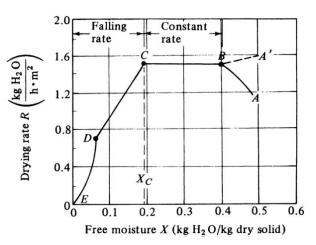


Figure 2. 5: Typical drying-rate curve (Geankoplis, 1995)

2.1.4 Drying Kinetics

According to Figure 2.5, under a constant drying condition, the drying can be divided into three distinct periods, namely initial heating period (A-B), constant rate period (B-C) and falling rate period (C-E).

(i) Initial heating period

During this period, the heat transfer to the wet solid initially goes through the conditioning stage. The surface temperature rises towards the wet bulb temperature of the air-vapour mixture. If the starting solid temperature is hot, the rate may start at point A'. The duration to achieve point B depends on the types of wet solid to be dried, as different material would have different thermal diffusivity. Furthermore, the duration to achieve the point B is proportional to the size of the sample.

(ii) Constant rate period

During this period, the drying rate is almost constant due to equilibrium condition is achieved. The wet solid forms a layer of water on the surface area, where the evaporation removes the water. The formed water is unbound, and this is referred to as the evaporation process as occurs on the free water surface. Because of the saturated vapour, the surface temperature is approximately the wet bulb temperature. Since a film of free water is always available at the evaporating surface, this period is governed fully by the external transport phenomena, nearly independent of the material being dried. The capillary movement is dominating the moisture transfer inside the solid. The volume shrinkage is approximately equal to the volume of the evaporated water. This period continues as long as evaporation rate is limited by the water being supplied to the surface, which the mechanism depends to the internal transport phenomena. In reality, many foodstuffs, however, do not display this period at all, due to the internal transport limitations (Foster and Okos, 1980; Mujumdar and Devahastin, 2000).

(iii) Falling rate period

The point at which the departure from constant rate period is first observed is so-called the critical moisture content, M_c where indicating the start of falling rate periods. The surface temperature raise toward the dry bulb temperature of air-vapour mixture. This period is determined by the internal transport phenomena. At the first falling rate period (C-D), due to insufficient water to maintain a continuous layer of water on solid surface, the surface is partially unsaturated. Capillary forces still controlling the moisture movement, but the surface layer of water starts to recede below the surface. The surface area continually decreases until it is completely dry, at where the second falling rate period (D-E) begins. At this period, the moisture movement is governed by the vapour diffusion. In falling rate periods, shrinkage still takes place, but to a much lesser extent. The surface would develop a hard layer on the surface, which known as case hardening, yields a higher resistance for heat and mass transfer. Generally, this period lasts for a longer time than constant rate period until the solid reaches its equilibrium moisture content at the given drying condition.

2.1.5 Rehydration of Dried Products

Rehydration or also called reconstitution is an important quality attribute in dried products which provides a good indication as to whether various processing conditions have been correctly applied. Rehydration involves a reverse physiochemical changes that occur during dehydration process. During the rehydration process, water transfer occurring from rehydration liquid to the dry solid by liquid diffusion until equilibrium is reached (Khraisheh *et al.*, 2003). Generally, the rate of water absorption and the extent of restoration of the dried fruits are affected by the degree of dehydration and disruption of cellular integrity. Rehydration is maximized when cellular and structural disruption is minimized.

The rehydration ratio RR is defined as the ratio of the mass of the rehydrated sample to the mass of the dehydrated sample (Khraisheh *et al.*, 2004).

$$RR = \frac{m_{rh}}{m_{dh}}$$
(2.6)

The coefficient of rehydration COR is calculated using (Prabhanjan *et al.*, 1995):

$$COR = \frac{m_{rh} (100 - X_o)}{m_{dh} (100 - X_{dh})}$$
(2.7)

The rehydration rate is defined as the slope of rehydration ratio versus rehydration time (Khraisheh *et al.*, 2004).

Rehydration Rate=
$$\frac{d(RR)}{dt_{rh}}$$
 (2.8)

2.1.6 Shrinkage and Casehardening

Colloidal and fibrous materials, especially for those high in initial moisture content do undergo shrinkage when bound water is removed during dehydration process. Since the outer layers necessarily lose moisture before the interior portions, the moisture concentration in outer layers is less than that in the interior. The surface layers shrink against an unyielding, constant-volume core; and cause checking, cracking and warping. Generally, diffusivity is sensitive to moisture concentration and decreases with decreasing concentration; the resistant to diffuse in the outer layers is increased. This accentuates shrinkage effects by impended the flow of moisture to the surface and so increasing the moisture gradient near the surface. The rate of drying is subsequently being reduced with the progressing of drying time. In extreme cases, casehardening occurs owning to shrinkage and drop in diffusivity to develop a layer of closed packed, shrunken cells, which are sealed together at the surface (McCabe *et al.*, 1993).

The effects of shrinkage and casehardening can be minimized by reducing the rate of drying; thereby flattening the concentration gradients in the materials to be dried and makes the diffusivity throughout the solid is more constant. The moisture gradient at the surface is flattened and thus, the entire piece is protected against shrinkage. In practice, humidity of the drying air is regulated to control the rate of drying (Geankoplis, 2003).

2.2 Microwave Vacuum Drying

2.2.1 Fundamentals of Microwave Drying

Microwave is an electromagnetic wave of radiant energy. It consists of a band of electromagnetic spectrum ranging from 0.3 to 300 GHz, fall in the range between radio waves and infrared radiations which corresponded to wavelengths ranging from 1 mm to 1 meter (Gould, 1996). The allowable frequency of microwave for domestic purpose in Malaysia is 2.45GHz, corresponding to 12.24cm wavelength or 4.9cm depth of penetration in vacuum (Mujumdar, 1987). For industrial, scientific and medical (ISM) use, however, the frequency of 915GHz is allowed under special authorization (Gould, 1996).

2.2.2 Mechanism of Microwave Drying

Generally, the periodic changing of wave's polarity and decay through zero that causes stress on ions, atoms, and molecules, which is converted to heat. Figure 2.6 show the plane electromagnetic wave with electric, E and magnetic, H as the component of the wave. Both component are perpendicular to each other and the direction of the propagation that is X-direction, make them a plane wave. Furthermore, the wave can be representing by sine or cosine function, which makes it monochromatic waves.

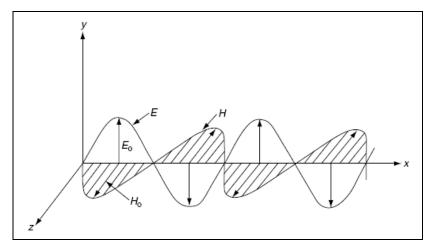


Figure 2. 6: Diagrammatic illustration of a plane monochromatic electromagnetic wave. E_0 and H_0 are the amplitudes of the fields. (Schiffmann, R. F. 2006)

When both field with vectors E and H lie in one direction, they are linearly polarized. Moreover, this energy wave changes its energy content and amplitude as it travels through a medium. This phenomenon can be seen from the diagram, both components undergoes cycles and firstly it is at zero, and then builds up to maximum value and again decays to zero, continue in the same pattern but with changing polarity after half cycle. As stated earlier, this phenomenon causing stresses and then converted into heat.

It is a crucial fact that microwaves are forms of energy that are manifest as heat through their interaction with materials. There are many mechanism of the energy conversion, for instance, ionic conduction, dipole rotation, interface polarization, dipole stretching, etc. However, the first two mechanisms are primarily interested in microwave drying.

2.2.2.1 Dipolar Rotation

Food and other materials contain polar molecules that act as dipole. For instance, water molecules are polar molecules with the negative charge centered near the oxygen atom and positive charge nearer the hydrogen atoms and can be say to possess an asymmetric charge center. Other molecules become induced dipoles due to stress causes by the electric field. Dipoles are influence by the rapidly changing polarity of the electric field.

When microwaves pass into foods, the electric field tend to pull them into alignment but then, they returned to random orientation as the field decays to zero. The patterns will be continuing with opposite polarity. This buildup and decays of field will occur as much as the electric field reverses 915 or 2450 million times per second. The molecules attempting to oscillate at such frequencies generate intermolecular friction, which causes energy conversion from electrical field energy to stored potential energy in the material and then to stored random kinetic or thermal energy, thus causes the food to heat.

The frequency in this drying is unknown as relaxation frequency. For small molecules, such as water, alcohols, etc the relaxation frequency is higher than the microwave frequency and will rises as temperature increases, causing slow energy conversion. In contrast, large molecules will have better energy conversion since they have lower relaxation frequency at room temperature. However, it implies that water, alcohols, are better microwave absorbers, thus, often drying of materials such as foods and medicinal, can be conduct at lower temperature even at cold or subfreezing temperatures.

A common misconception is microwave cook food from the inside to the outside. In reality, the outer layers of food absorbed microwave in a manner similar to heat form dehydration methods. However, due to the capability of microwave to penetrate dry nonconductive substances at the surface of food, this method usually deposit initial heat more deeply than other methods (Bilbao *et al.*, 2006). Depending on water content, the depth of initial heat deposition may be several centimetres or more with microwave ovens, in contrast to broiling (infrared) or convection drying, which deposit heat thinly at the food surface. Depth of penetration of microwaves is dependent on food composition and the frequency, with lower microwave frequencies being more penetrating.

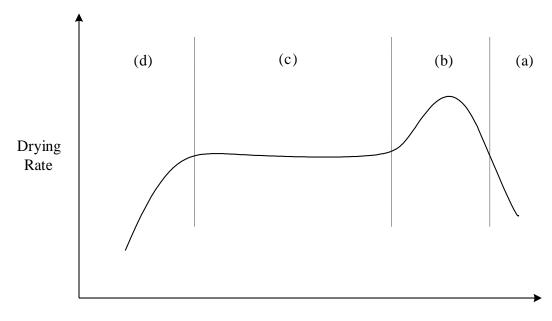
2.2.2.2 Ionic Conduction

Ionic conduction is not dependent largely upon either temperature or frequency. It involve in two-step energy conversion: electric field energy is converted to induced ordered kinetic energy, then converted to disordered kinetic energy, which is regarded as heat.

Ions, as charged units are accelerate by electric fields. The electric field will cause them to move in the opposite direction of their polarity. As a result, they will collide with unionized water molecules, giving up kinetic energy and causing them to accelerate and collide with other water molecules, and when the polarity changes, the ions accelerate in the opposite pattern. For electric field reverses 915 or 2450 million times per second, high transferred of energy occurred for large numbers of collisions.

2.2.3 Physical Processes During Microwave Drying

Figure 2.7 describes the physical processes which involved during a typical microwave drying.



Moisture Content

Figure 2. 7: Typical drying curve for microwave drying

(a) Initial heating up period

During this period, the material temperature rises towards the wet bulb temperature of the liquid.

(b) Liquid movement period

When the temperature of the wet solid approaches the boiling point of the liquid then internal evaporation can occur and an increase in the total pressure is developed within the pores. Mass transfer is now mainly governed by the total pressure gradients due to rapid formation of the vapour phase in the wet solid. If the

material in very high moisture content, some moisture may be removed as liquid due to filtrational flow driven by the total pressure gradient. During this period, the rate of moisture removal increases beyond the constant drying rate limit that does not exist in conventional convective drying.

(c) Constant-rate period

During this period, the moisture content is very high. Evaporation will take place from the surface at a constant rate as long as the ambient conditions remain constant. The moisture is supplied continuously through capillary forces from the interior layers towards the surface to replace the water being evaporated.

(d) Falling-rate period

When the moisture content reduces below a critical level, due to the limitation by the reduction of water migration from the interior towards its surface, the rate of evaporation becomes gradually less. The moisture migrates towards the surfaces by mass flow of the liquid and vapour phases. As the solid dries out the network of capillaries ceases to be continuous introducing pockets of air which impedes the liquid movement towards the outer layers, vapour flow is now the dominant flow mechanism during this period.

2.2.4 Operational Constraint and Controversy of Microwave

1. Non-homogeneity of microwave

Although microwave processing of food is fast, there are several potential problems that cause undesirable quality changes of dried fruits. The uneven distribution of microwave energy and different rates of energy absorption in different portions of food cause uneven heating or burning in microwaved food (Kelen *et al.*,

2005). Two main factors that cause non-uniform distribution of microwave energy are cavity effects (design limitation, location of feedstock, shape of cavity, hanging parts such as spray gun, mixer or thermometer, etc) and workload interactions (loss factor, penetration depth and thickness of the workload and particle's features)(Kelen *et al.*, 2006). The uneven distribution of microwave can be reduced by a stirrer, a type of fan that rotates and does change the radiated area of food continuously.

2. Fluctuation of microwave power output

In general, microwave power output is somewhat different from the rated capacity that is stated in the manufacturer's literature and this may be due to magnetron filament and heating effects. As the magnetron ages, it takes the filament a longer time to reach the emission condition. The power variations may also occur if the magnetron is operated for long period of time, as the prolonged heating of the permanent magnets (which is part of the magnetron) causes reduction in the magnetic field and hence reduction in the power output (Zheng *et al.*, 2004).

3. Normal Boiling Point Rise

Liquid with a smooth surface does undergo superheat and reaches temperature a few degrees Celsius above their normal boiling point during microwave irradiation. The boiling process start explosively when liquid is disturbed, such as when the microwave power is pause, which can result burning (Zheng *et al.*, 2004).

2.2.5 Microwave Vacuum Dehydration

The main purpose of vacuum dehydration is to enable the removal of moisture at less than the boiling point. The boiling point of water does decrease with the reducing of vapour pressure, as showed in Figure 2.8. At the high vacuum level, evaporation occurred at relatively low temperature and thermal damage on dried products can be avoided. The qualities of dried products such as colour, texture, nutritious properties, flavour and aroma can be preserved after dehydration process (Tsami *et al.*, 1999). An important feature vacuum drying is the absence of air during drying and this makes the process attractive for drying materials that may deteriorate as a result of oxidation or may be modified chemically as a result of exposure to air at elevated temperature (Contreras *et al.*, 2005). Puffing effects owing to microwave heating enhanced by high vacuum level causes fast mass transfer and therefore, makes shorter of the drying period.

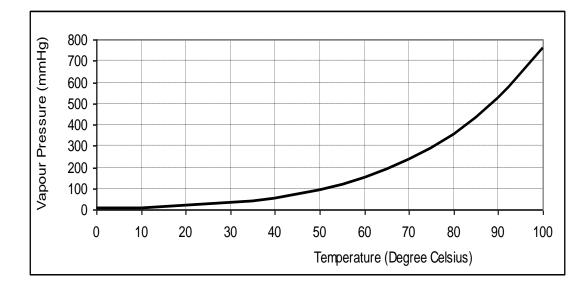


Figure 2. 8: Vapour pressure versus boiling point of water (Felder and Rousseau, 1986)

Microwave acts as heat supply in this technology and thus, can be classified as non-adiabatic dehydration process. The disadvantage of microwave is superheating of free liquid molecules may occurs and makes the heating temperature higher a few degrees Celsius than the actual boiling point at a fixed pressure (Zheng *et al.*, 2004).

2.2.6 Material Study in the Design of Vacuum Microwave System

The interaction of electromagnetic fields with materials is important in the design of microwave vacuum system to avoid materials failures such as burning of constructed materials, explosion of high vacuum system and ineffective drying. Materials can be classified into four categories depending on their characteristic when interact with electromagnetic field (Stewart and Amerine, 1982).

Conductors such as metals are materials with free electrons. These materials reflect electromagnetic waves without energy absorption and usually are used to contain and directly disposed to microwave.

Insulators such as glass are electrically nonconductive materials. Insulators reflect and absorb electromagnetic waves to a negligible extent and primarily transmit them (transparent to the waves). These materials are useful to support or contain materials to be heated by the electromagnetic field.

Dielectrics or lossy dielectrics are materials which properties range from conductors to insulators. These materials absorb electromagnetic energy and convert it into heat. The examples of dielectrics are water, oils, wood, sugar and fat.

Magnetic compounds such as ferrites are materials interact with the magnetic component of the electromagnetic wave. These materials are often used as shielding or choking devices that prevent leakage of electromagnetic energy.

2.3 Sensory Evaluation

The total sensory quality of food products, i.e. colour, texture, flavour and aroma can only be predicted to a certain extent by instrumental, physical and chemical methods and the accuracy of these predictions may differ to consumer's need. Sensory evaluation is a non-instrumental method that applies natural human's sense on the food products. The points in favour of using sensory analysis over instrumental methods as the measure of product quality are as follows (Arthey and Dennis, 1991):

- Sensory analysis uses human senses and can measure many variables at one time.
- 2. More rapid and cheaper than other instrumental methods.
- Sensory analysis has no larger measurement noise than instrumental methods. Since human beings are involved, there is an intelligent selfcorrection in a sensory panel that is often lacking in machinery instruments.
- 4. More reliable in marketing because human beings are involved.

Selecting assessors of sensory evaluation must be carried out with as much care as possible because the wrong selection causes unreliable results. It is important that the assessors is well motivated and has correct attitude toward the test. Poorly motivated assessors quickly lose interest in their work, become hasty, careless and apparently poor at discrimination. Their appearances also start to influence others leading to a general despondency in the panel. Therefore, it is important to ensure that assessors are aware of their contribution to the success of quality test. All observations and objective should be independent and be made by individuals who are confident in their judgments. Domineering assessors who seek to impose their views on others should not be selected. For same reason, mix of senior and junior judges should be avoided to exclude assessors with any particular bias (Arthey and Dennis, 1991).

2.4 Mathematical Model

In calibrating microwave power output, calorimetrical method, which is to measure the change of temperature of a known mass of water for a known period of time, is applied. The power output can be calculated by the following equation (Zheng *et al.*, 2004).

$$Q_{abs} = \frac{mC_{p}\Delta T}{t}$$
(2.9)

Absolute moisture content is the ratio of water mass in the feed to dry solid mass (Geankoplis, 2003).

$$X_A = \frac{W - W_s}{W_s} \tag{2.10}$$

Equilibrium moisture content is the minimum moisture content that can be achieved during hot air drying at fixed temperature and humidity of hot air.

Free moisture content is the moisture content above the equilibrium moisture content. The moisture content of solid can be further reduced by drying even though the drying condition remained constant (Geankoplis, 2003). The equilibrium moisture content depends on the temperature and humidity of hot air. The high temperature and low humidity of air tend to reduce the equilibrium moisture content.

$$X = X_A - X^* \tag{2.11}$$

The drying rates of vacuum microwave have been calculated according to the following equation. The drying rate calculation of hot air is different with that of microwave system. For microwave system, the drying process occurred throughout

all surface of material. Hot air drying, however, occurred on the external surface that disposed to hot air (Lin *et al.*, 1998).

$$R = \frac{\Delta X}{\Delta t} \tag{2.12}$$

CHAPTER 3

METHODOLOGY

3.1 Design of Microwave Vacuum Dehydration System

The experimental setup used for this particular microwave vacuum dehydration research was depicted in Figure 3.1. There are four key components in the design, which were vacuum chamber, heat supply of microwave, condensate unit and vacuum production unit (Woodroof and Bor, 1986). For piping and connection, polyethane tubing 7mm ID was employed to connect the vacuum chamber and condensate unit, since this material is highly microwave transparency and possesses adequate strength to withstand differential pressure. Meanwhile, LPG gas tubing 7mm ID was employed to connect among the other units, except the vacuum chamber. LPG gas tubing is not absolutely transparent to microwave and hence direct exposure to microwave irradiation should be avoided. However, the selection of this tubing can possesses adequate strength to withstand high differential pressure with the relatively low cost compared to polyethane tubing. Therefore, this type of tubing is recommended to be employed for connecting the units where positioned outside the microwave irradiation spot. Silica gel lapped on each connecting junction to ensure the entire system air-sealed. Initial test run had been conducted to investigate the strength of the construct material against the damage of microwave during irradiation.

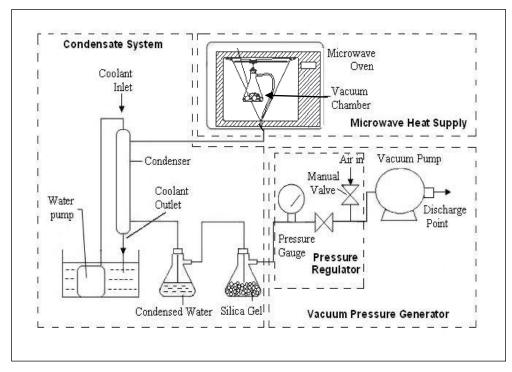


Figure 3.1: Laboratory-scale Microwave Vacuum Dehydration Rig

A vacuum flask with adequate strength to withstand the differential pressure was employed as vacuum chamber, which was then placed in a microwave chamber, as illustrated in Figure 3.1. One of the important properties about the vacuum flask is that, it must microwave transparency, which is the key to ensure microwave penetrate through it and reach experimental material during irradiation. Thus, the vacuum flask made of Pyrex glass is chosen where it does not absorb microwave energy.

A domestic microwave (SHARP Model R-958A) of rated capacity of 900W and frequency of 2.45GHz with 5 magnetron levels was used as heat supply unit. Although the microwave oven provided several power levels, there is no change in the frequency and intensity of the microwave; instead, the power levels are about the duration and cycle of magnetron on and off. Figure 3.2 showed the irradiation cycle of different power levels for this microwave oven. It is observed that the time of a complete irradiation phase for every power level mode is the same, 30 seconds. However, the period of irradiation occurs in a complete phase are: 30 second for high

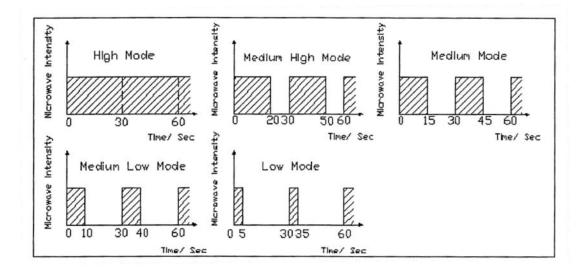


Figure 3. 2 : The radiation cycles of various power levels of microwave (SHARP Model: R-958A)

mode, 20 seconds for medium high, 15 seconds for medium, 10 seconds for medium low and 5 seconds for low mode.

The condensate unit consists of a condenser, and two conical flasks where one is an empty collection flask and the other is filled with silica gel. The condensed water was collected in the collection flask while the moist air from this flask is sucked into the silica gel filled flask to reduce its humidity. At the start of each drying experiment, the collection flask must be empty in order to reduce back pressure that may occur when the vacuum pump is switched off.

The vacuum production unit consisted of a vacuum pump, pressure regulator, and a deflection type pressure gauge. A vacuum pump was connected to the vacuum chamber to generate processing vacuum level during the experiment. The result showed that the vacuum pump used in this research was capable to generate vacuum level up to -65cmHg, which corresponded to water normal boiling point of 53°C. The pressure regulator consists of two valves that connected parallel. One of these valves was positioned in series with the vacuum pump and functioned as on-off valve, to maintain the vacuum level when the vacuum pump is paused. Another valve is

served to manipulate the vacuum level in the vacuum chamber by allow some air flowed into the chamber (for low vacuum level application, >-65cmHg). With fully close of this valve, no air flow through the valve and the vacuum level at the chamber is maximum, i.e., -65cmHg.

In order to cope the problem of non-homogeneity of microwave hating in the cavity, some journals (Kelen *et al.*, 2006) suggested that the microwave food should moved or changed its position continuously in the cavity during the microwave irradiation so that the entire surface of food is exposed evenly. This may prevent the experimental material having different rates of energy absorption where uneven heating or burning may occur. Thus, a mechanical moving tool was designed and employed, as illustrated in Figure 3.3. All construct materials in this design were non-metallic and transparent to the microwave. Note that the vacuum chamber is moving to the right hand side when the string A is drawn, vice versa. In this case, the maximum displacement was approximate 10cm.

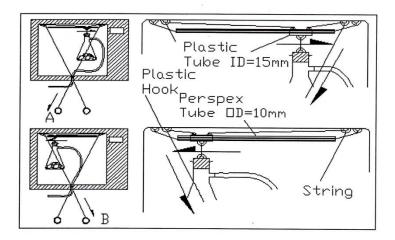


Figure 3. 3: Manual mechanical moving tool of vacuum microwave dehydration system

3.1.1 Calibration of Microwave Power

A microwave oven does not convert all electrical energy into microwave and also, not all the microwave energy is totally absorbed during the heating process. The microwave power output is somewhat different from the rated capacity that is stated in the manufacturer's literature and this may be attributed to the effects of magnetron filament and heating. Therefore, calibration is important to determine the power absorption for each power modes.

In order to determine the power absorption, 500mL distilled water at 28°C (room temperature) was heated in the microwave oven with applying the high mode. The temperature of water was recorded for every minute until the water boil. The similar procedures were repeated for medium high, medium, medium low and low mode. The power absorption was calculated, based on the amount of sensible heat required to rise temperature.

3.2 Material Preparation and Experimental Procedure

Three experimental materials are tested to investigate their drying mechanisme and the kinetics of microwave transfer in the vacuum testing rig. The selected thermolabile materials are jackfruit (*Artocarpus heterophyllus Lam.*), papaya (*Carica papaya L.*) and guava (*Psidium guajava L.*). The following sections will further discuss the preparation of these materials.

3.2.1 Jackfruit (Artocarpus heterophyllus Lam.)

Fresh jackfruit (*Artocarpus heterophyllus Lam.*) cultivar J31 obtained from a commercial farm in Pekan Nanas, Johor, Malaysia, was used for dehydration and rehydration experiments. All jackfruit was come from the same batch. The edible bulbs of jackfruit were selected manually to obtain those in good condition and without deterioration. The jackfruit bulbs were washed to remove dirt, dust, plant parts and then, allowed to drain for 10min. Then, the bulbs were cut into halves. In a rule, washing should be encouraged before peeling, cutting or pitting to reduce the losses of sugar, vitamins, minerals and other soluble elements (Woodroof and Bor, 1986).

A 100g sample was placed into a vacuum chamber. A vacuum pump was turned on to generate -65cmHg vacuum level in the chamber. Then, microwave oven was turned on and the power level was set to high mode. The sample was irradiated for one minute, followed by weighed with the electronic balance. Weight loss was recorded. The similar procedures were repeated until the targeted moisture content was reached. The experiment was repeated with employing medium high, medium, medium low and low mode.

3.2.2 Guava (Psidium guajava L.)

Fresh ripe guavas were obtained from a local supermarket and were stored at room temperature. Prior to drying, the guavas were washed, hand peeled and cut into quarters. The guavas were then pitted and the flesh was manually cut into slices with a thickness of approximately 5mm. The sample size used for each drying experiment was $50\pm1g$ of this prepared guava sample and the weight was determined using an electronic balance.

A sample amount of 50g was put into the vacuum chamber. Then, microwave oven was turned on and the power level was set to high mode. The sample was irradiated for half minute, followed by weighed with the electronic balance. Weight loss was recorded. The similar procedures were repeated until the targeted moisture content was reached. The experiment was repeated with employing medium high, medium, medium low and low mode.

3.2.3 Papaya (*Carica papaya L.*)

The unripe, green papayas were select, where presented from MARDI, Pontian, Johor and kept in cold storage at 4-5°C. Prior to dehydration, papaya were thoroughly washed to remove dirt. Slices of approximately 5 mm thickness were obtained by carefully cutting papaya horizontally with a vegetable knife. The sizes of the slices were graded to eliminate the variations in respects to exposed area. The drying experiments were carried out without any pretreatments. Then, the sliced papayas were weighted. Moisture content of the samples was determined in an oven at 107°C until constant weight. The initial moisture content of the slices was range from 92% to 93% (w.b.). A 200 g sample was place in a vacuum chamber and the vacuum pump was turn on to generate -65 cmHg vacuum levels in the chamber. Then, microwave oven was turned on and the power level was set to high mode. The sample was irradiated for half minute, followed by weighed with the electronic balance. Weight loss was recorded. The similar procedures were repeated until the targeted moisture content was reached. The experiment was repeated with employing medium high, medium, medium low and low mode.

3.3 Rehydration Test

A small amount of the dried sample (5 g for both jackfruit and papaya; 2 g for guava) was immersed in 500mL distilled water in a beaker at room temperature $28 \,^{\circ}C$ for 30 minutes. Then, the sample was put out from the beaker with using a chip basket. Surface moisture of the sample was removed by wiping the sample off with a tissue paper. The weight of rehydrated sample was recorded. The similar procedures were repeated until no significant difference in weight was tracked. This rehydration test was conducted for the dried sample produced microwave vacuum system drying. The rehydration rate, rehydration ratio and coefficient of rehydration were then evaluated according to equation (2.6) and (2.7).

In general, the rehydration test can be conducted with any water temperature and often at room temperature or $100 \,^{\circ}C$. The rehydration rate is becoming higher along with the increasing of the temperature. For example, Tein *et al.*, (1998); had reported that the rehydration periods were 5 minutes for $100 \,^{\circ}C$ rather than 90 minutes for $25 \,^{\circ}C$. However, the water at room temperature has been used in this study. High temperature tends to damage the fabric structure of jackfruit and hence reduces in rehydration potential (Tein *et al.*, 1998). Low temperature rehydration yields more representative result owing to the neglect of thermal damage of hot water.

3.4 Enzymatic Activity Analysis

The enzyme activity is commonly determined by measuring the rate of protein degradation produced by a given amount of papain. Various methods have been published using casein, gelatin, hemoglobin, milk, and meat powder. This method used is Milk Clot Unit (MCU). This assay is based on the proteolytic hydrolysis of a buffered milk substrate at 40°C. Enzymatic activity was related to the time required to clot 5 ml of substrate.

3.4.1 Reagent Preparation

3.4.1.1 Concentrated Buffer (stock reagent)

- 1. Acetic acid solution: Glacial acetic acid of 61.0 g was added into 800 ml distilled water in a 1000 ml volumetric flask. Then, dilute with distilled water until the mark.
- Sodium Hydroxide (NaOH) 1 N was added to 500 ml acetic acid solution with constant stirring to give pH of 4.5. The pH was measured by using standardized pH meter.

3.4.1.2 Dilute Buffer Solution

The concentrated buffer solution of 114 ml was mixed with distilled water to make total volume of 850 ml. This solution was prepared fresh daily to make milk substrate.

3.4.1.3 Milk Substrate

This milk substrate was stored at 4 to 8°C to be stable for one week.

- 1. Instant non fat milk of 200 g was slowly mixed with 850 ml of dilute buffer solution in a 2000 ml beaker using a magnetic stirrer.
- 2. The mixture was mixed for 30 minutes to make sure that all the powder was already mix, and no lumps remain.
- 3. The mixture was then added five drops of toluene and stirred for another 5 minutes.
- 4. The mixture with covered was leave for at least one hour to make sure any foaming had dissipated.
- 5. The mixture was then filter through the glass wool, which has been loosely packed into a funnel.
- 6. The filtrate was kept in refrigeration for 4 hours before can be used.

3.4.1.4 Enzyme Buffer

1. Beaker of 2500 ml with a stir bar was prepared on the magnetic mixer.

- 2. Distilled water of 1600 ml was added into the beaker.
- 3. Then, anhydrous sodium phosphate of 14.20 g, L-Cysteine of 12.20 g and EDTA of 28.00 g were added into the beaker.
- 4. The mixture was stirrer until dissolved.
- 5. The pH of the buffer solution was adjusted to 6.0 with 1 N NaOH (the original pH should be about 5.7, then approximately 20 ml of NaOH will be needed).
- 6. The buffer solution was transferred to a 2000 ml volumetric flask and dilute until the mark with distilled water.

3.4.2 Procedures

3.4.2.1 Milk substrate standardization

- 1. Commercial papain was accurately weight and diluted with the enzyme buffer.
- The beaker was placed on a stir plate, and the solution was stirred for at least 5 minutes.
- 3. The solution was then transferred into 100 ml volumetric flask quantitatively.
- 4. The average of three clotting times were use to determine the milk clotting factor, M.

3.4.2.2 Enzyme preparation

- An enzyme solution in buffer solution was prepared to make 2.0 ml of the final dilution gave a clotting time equal to the standard clotting time (between 2.5 to 3.5 min).
- 2. Enzyme calculation was shown in Equation 2.7.

Enzyme weight (g) = $\frac{M}{2 \times ST \times Target activity}$

(3.1)

where 2* is 2 ml of enzyme preparation injected

3.4.2.3 Enzyme evaluation

- 1. Substrate of 25 ml was pipette into a series of the screw cap tubes.
- 2. For each enzyme sample allowed at least two tubes.
- 3. Each tube was screw and allowed to equilibrate for 20 minutes in a $40^{\circ}C \pm 0.1^{\circ}C$ water bath.
- 4. At zero time, 2.0 ml of enzyme solution was pipette into the test tubes, while simultaneously started the stopwatch. The solution was mixed by inverted slowly for four times.
- 5. Then, the tube was put into the water bath and the tube was slowly rotate horizontally. The milk film that drained from the tube walls was observed.
- 6. The milk film will begin to thicken approximately 20 seconds before the end point.
- 7. The end point was the exact moment that "pebbling" begun (small beads of coagulated milk along the glass).
- 8. The stopwatch was stop and the time in minutes.

3.4.2.4 Calculation

Sample activity as shown in Equation 2.8.

$$MCU/mg = \frac{M}{T \times W}$$

where:

М	=	Milk factor
Т	=	Clotting time of sample (min)
W	=	weight of enzyme added to the substrate in 2 ml aliquot
		$(g wt. \times 2)$

(3.2)

CHAPTER 4

STUDY ON THE DRYING MECHANISM AND ENERGY TRANSFER OF JACKFRUIT AND GUAVA IN VACUUM MICROWAVE SYSTEM

4.1 Microwave Power Output Measurement

Prior to drying the testing materials using the microwave vacuum drying equipment, the microwave power density of each microwave power setting is conducted. This was carried out to obtain the actual power density since the power density may differ from the value stated by the manufacturer. This difference is usually because of magnetron heating and also aging. Apart from that, variations in power output may also be due to operation of the magnetron for a long period of time (Cui et al., 2004).

The microwave power output for each microwave setting was determined calorimetrically. Table 4.1 shows the microwave power output for each microwave power setting.

Microwave Power Setting	Power Output (W)
High	573.77
Medium High	447.78
Medium	343.10
Medium Low	228.73
Low	92.77

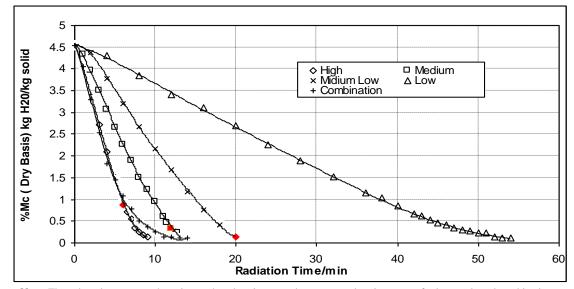
.... 4 4 0 ...

4.2 **Vacuum Microwave Drying Characteristics**

4.2.1 Jackfruit

The total drying time required to reaches the jackfruit sample to the targeted moisture content under -65cmHg vacuum pressure at high, medium, medium low and low mode are 9 min, 13 min, 21 min and 53 min respectively as shown in Figure 4.1. The drying rate increases along with the increasing of the microwave power density because higher power absorption accelerates the evaporation rate of moisture. The drying rate of microwave vacuum drying is faster than hot air drying. This occurs because of the fast mass and heat transfer within the jackfruit during microwave heating. In microwave drying, the heat is generated directly within the fruit and hence creates a large vapour pressure differential between the interior and the outer surface of material being dried. High pressure gradient increases the mass diffusivity and thereby accelerates the moisture diffusion form the interior to outer surface. Besides, high pressure gradient also causes the structure of jackfruit halves to puff or to expand and this attributes to low density product in comparison with hot air dried product and freeze dried product. With the low density, the amount of vapour gap in material structure is relatively large and this reduces the difficulty of vapour to transfer from the interior to outer surface. In the tradisional hot air drying, however,

heat is transferred from the outer surface to the interior and this occurred rather slowly. The direction of heat transfer is opposite with the direction of mass transfer. The amount of moisture transferred to and evaporated on the outer surface is much low in comparison with microwave vacuum drying. The similar results had reported by Andres *et al.*, (2003) in their studied of drying apple with the use of combined hot air-microwave dehydration.



Note: The red marks represent the point at where burning started to occur; no burning occurs for low mode and combination of the various modes.

Figure 4. 1: Dehydration curves for different power levels

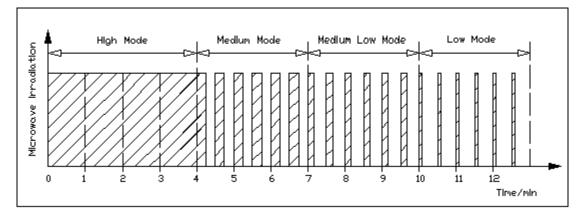
The high power level reduces the drying time; however, too high microwave power causes physical damage to the dehydrated products, such as over-heating or char. Continuous microwave irradiation results continuous rise in local temperature of material to be dried and eventually causes charring. Therefore, the proper microwave irradiation cycle or time schedule has to be determined in order to simulate effective drying, and this mainly depends on the types of material to be dried, mass load and the intensity of microwave.

4.2.1.1 Development of Proper irradiation Cycle

One distinct advantage of microwave heating is power absorption proportional to the residue moisture content (Prabhanjan et al., 1995). This could be seen in Figure 4.1, where the drying rate decreases along with the progressing of drying time. When the residue moisture content is too low, most of the microwave power lost as leakage, non productivity heating and sample charring, but not moisture evaporation. Drying of low residue moisture content material with the use of high microwave power is not effective therefore. In order to improve the effectiveness of drying and to reduce the tendency of charring, the use of different power level where depends on the residue moisture content is encouraged. The experiments had been conducted to determine at where the range of the residue moisture content was when the burning started at the use of a certain mode. The results shows that the range at high, medium, medium low and low mode are 45-50% wt, 20-25% wt, 10-15% wt and no detectable or <10% wt, respectively. Microwave irradiation with the use of a certain mode for a sample with lower residue moisture content than the corresponded range of that mode will cause burning or overheating. The drying process is not effective therefore. With the use of these ranges, the proper irradiation cycle can be determined so that improves the weakness of microwave heating such as overheating. The type of power mode to be employed and at when it being employed during drying are according to the residue moisture content of material being dried along drying.

The proper irradiation cycle of the combination of various modes had been determined, as illustrated in Figure 4.2. At the initial drying stage, the jackfruit halves with high initial moisture content are irradiated continuously. High moisture content of jackfruit at initial stage prevents them to be overheated. As the decreasing of moisture content over the time, the irradiation frequency is reduced to prevent over-burning. This cycle could be manually set with changing the power mode systematically during drying. For instant, to dry jackfruit with initial moisture content of 82wt% (d.b.), the microwave oven is switched to high mode for four minutes, then three minutes with medium mode, four minutes with medium low

mode and finally, three minutes with low mode. No time interval is necessary in between two shifted modes. The experiments had been conducted to employ this cycle and the results were rather satisfactory, no burning occurred and the total drying time required was very short, approximately 12 minutes, as illustrated in Figure 4.2. It is noted that different material requires different irradiation cycle in drying.



Note: The cycle period: 30sec; 1) High: 30sec on-0sec paused; 2) Medium: 15sec on-15sec paused; 3) Medium Low: 10sec on-20sec paused; 1) Low: 5sec on-25sec paused;

Figure 4. 2: Microwave irradiation of the combination of different power mode

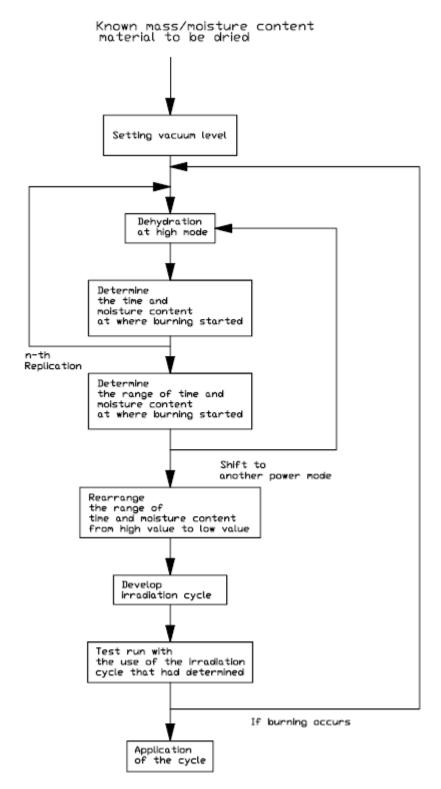


Figure 4. 3: The algorithm in determining proper irradiation cycle of microwave vacuum drying

4.2.2 Guava

Guava samples with initial moisture content of 89 % were dried n microwave vacuum to a final moisture content of 10%. The amount of vacuum pressure applied was -600mmHg. The corresponding boiling point of water is about 61°C. The MWVD experiment was conducted using only three microwave power setting which are high, medium high and medium.

4.2.2.1 Effects of Microwave Power Setting

MWVD of guava using the high microwave power setting was carried out. The power output of 573.77 W was found very high for the sample size of 50g which equivalent to the microwave power density of 11.393 kW / kg.

Therefore, in order to reduce the microwave power density to a suitable value, the load was increased. This process was done by using water as an additional load to absorb the microwave power. In this way, the sample would absorb microwave power at appropriate level and charring of the sample could be reduced.

The MWVD experiment was then continued with microwave power density of 1kW/kg. At the onset of drying, the water vapour from the sample was condensed on the inside wall of the vacuum chamber and the surface of the sample became wetter. As the drying proceeds, it was observed that dry streaks appeared on the sample. During the later stages of drying, the dry streaks expanded and turned yellow. Finally, when the targeted moisture content of ten percent was reached, the dried sample has almost turned completely yellow. Additionally, the experimental results indicated that drying of guavas occurs in the falling rate period as no constant rate period was observed during the drying process. This is similar to the finding by Maskan (2005) that the drying of bananas takes place in the falling rate period. It can be observed that the drying rate of the samples increase if compared to literature value for conventional drying techniques. This is because, of the volumetric heating mechanism of microwaves and application of vacuum which reduces the boiling point of water.

The MWVD experiment was conducted on the guava samples until the desired moisture content of ten percent was obtained. It can be observed from Figure 4.4 that at the start of the drying process the moisture loss was small. This is because during this stage, the microwave energy is required to heat the vacuum flask and also required to heat the water in the sample to the evaporation temperature (Krulis et al., 2005).

Afterwards, the moisture loss increased which indicates acceleration of the drying rate. This is probably due to the opening of the physical structure which permits fast evaporation and transport of water (Wang et al., 2004).

Figure 4.4 also shows that when the moisture content reached about 84 percent, the drying rate started to decrease. This is because at the later stages of drying, the amount of moisture in the sample is limited. As the microwave power dissipation is highly dependent on the moisture content of the material, the volumetric heating due to microwave power dissipation is therefore reduced resulting in lower moisture loss. Apart from that, a portion of the microwave power also reflects back to the magnetron (Lian et al., 1997)

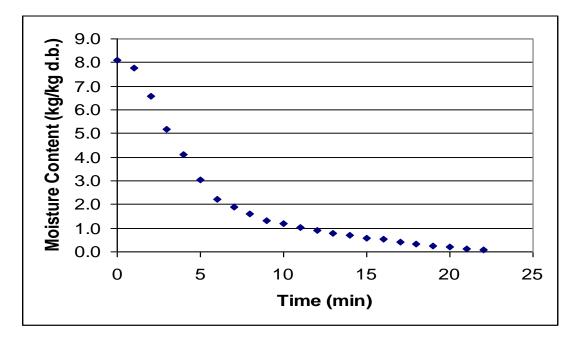


Figure 4. 4 Drying curve for guava dried using high microwave power setting with power density of 1kW/kg and vacuum pressure of -600mmHg

The guavas were also dried using different microwave power setting which are medium and also medium high setting. The microwave power density used for the drying experiments were maintained at about 1kW/kg.

Figure 4.5 shows the drying curves of guava dried using different microwave power setting with the same power density of 1kW/kg. It is illustrated in the figure that for the different power settings, the drying time is almost equal when the same power density is used. However, moisture loss is faster when high microwave power setting is used. This may be due to the fact that for high microwave power setting, the magnetron is continuously turned on therefore more power is absorbed and evaporation of moisture content is also greater than the lower power settings.

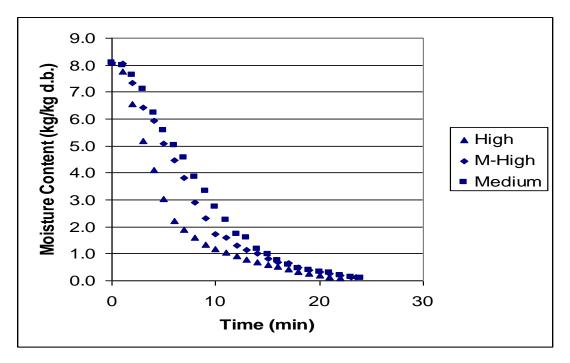


Figure 4. 5 Drying curve for guavas dried using different microwave power setting with power density of approximately 1kW/kg

4.2.2.2 Effects of Microwave Power Densities

In order to study the effect of different microwave power density on MWVD of guava, power densities of about 0.5, 1 and 1.9 kW/kg were used to dry the guavas. The microwave power setting used for the drying experiments was high power setting.

Figure 4.6 illustrates that when the power density is changed, the dehydration rate for the guava also changes. It was observed that when a high power setting was used with different power densities of 0.5, 1 and 1.9 (kW/kg) respectively, the mean drying rate of the samples increased with increment of the power density. Application of each microwave power density requires an average of 14, 21 and 31.5 minutes respectively to reach the desired moisture content of ten percent (wet basis).

The faster dehydration rate at higher power densities shows that more heat is generated in the sample. This results in larger vapour pressure differential between the centre and the surface of the product which consequently increases the drying rate (Wang et al., 2005).

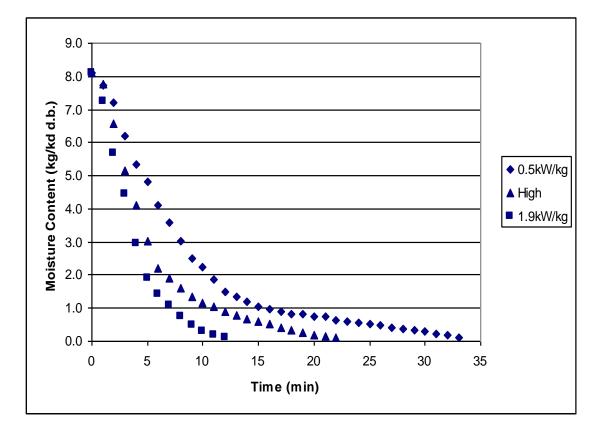


Figure 4. 6 Drying curves of microwave vacuum dried guavas dried using different power densities

Nonetheless, more severe charring of the samples occurred when higher microwave power densities were used. It was observed that for microwave power density of 1.9kW/kg, charring of the sample occurred within approximately four minutes. Meanwhile for both microwave power densities of 0.5kW/kg and 1kW/kg it takes longer than four minutes for charring to occur. Physical damages such as charring and scorching are due to the continuous rise of the local temperature when the moisture content is low. Apart from that, this also shows that there is a possible non-uniform microwave within the cavity. As a result, the quality of dried product is

more difficult to maintain when high microwave power densities are used (Hu et al., 2006).

Microwave power setting is controlled by the amount of time in which the magnetron is turned on and off. The time duration when magnetron is alternately turned on and off differ for each microwave power setting. This is actually what affects the microwave power output for each setting.

In order to investigate the effects of microwave power setting on the MWVD of guava, approximately the same amount of power density was used while the power setting was changed. The power density used was approximately 1kW/kg and this was set by changing the water load in the microwave cavity.

Meanwhile, the power settings that were used for the drying experiments were high, medium high and medium. Drying was initiated using high microwave power density for five minutes. Then medium high power setting was used to dry the samples for another five minutes until the moisture content reached about 0.5 percent. Afterwards, the medium microwave power setting was used to dry the guavas to the desired moisture content. The application of combined microwave power setting reduced the charring of the samples.

The graph in Figure 4.7 shows the moisture loss of the guavas during the MWVD using different microwave power settings. It can be seen that it takes roughly about 25 minutes for the guava sample to reach the desired moisture content. Apart from that, the moisture loss is proportional with the microwave power setting used. For instance, the lower microwave power setting caused less moisture loss from the sample.

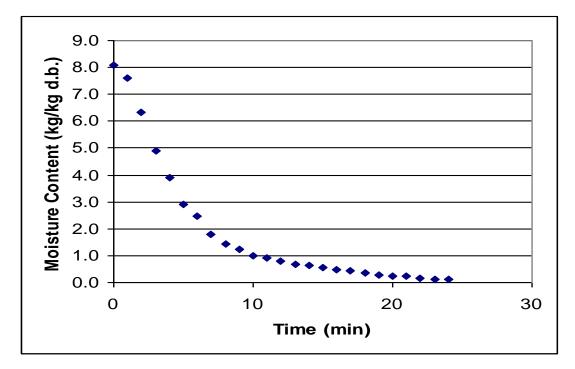


Figure 4. 7 Drying curve for the MWVD of guava using the combination of different power settings

The dried product obtained from combining the microwave power settings were less prone to charring. This is because the power setting was changed before charring could take place. Additionally, the products have a more intense yellow colour compared to samples dried using only one microwave power setting.

4.2.2.3 Combination of Microwave Power Densities

The microwave power absorption of materials is proportional to the amount of water content of material (Lian et al., 1997). Therefore, efforts were made to use different combination of microwave power densities to in each stage of the guava drying process. This can help reduce the power requirements of the MWVD process which is considered one of the limitations of this drying technology (Zhang et al., 2006). The MWVD experiments were conducted by reducing the microwave power density as the moisture content decreases. Initially, higher microwave power densities were continuously used until charring of the sample is observed. Subsequently, the microwave power density is reduced in order to get a better looking product. The algorithm for determining the suitable microwave power combination for the MWVD is presented in Figure 4.8.

The experimental results shows that the suitable combination of microwave power densities for the MWVD of guavas was four minutes microwave irradiation using 1.9kW/kg power density followed by five minutes using 1kW/kg power density and lastly, 0.5kW/kg power density was used to drying the sample to a final moisture content of ten percent. This combination of microwave power densities produced product with the least amount of charring. The average drying time required for this drying process is about 29 minutes. The drying curve for MWVD of guava using combination of microwave power densities is illustrated in Figure 4.9.

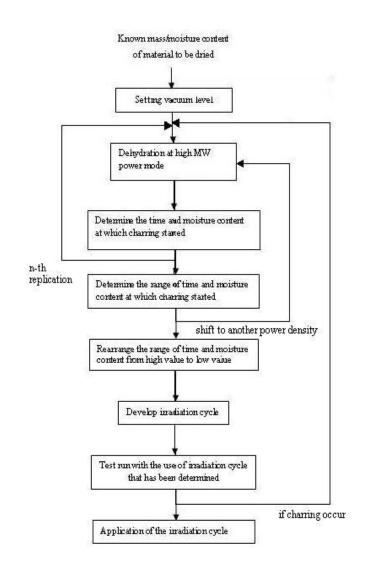


Figure 4. 8 The algorithm in determining the suitable microwave power combination for the MWVD process

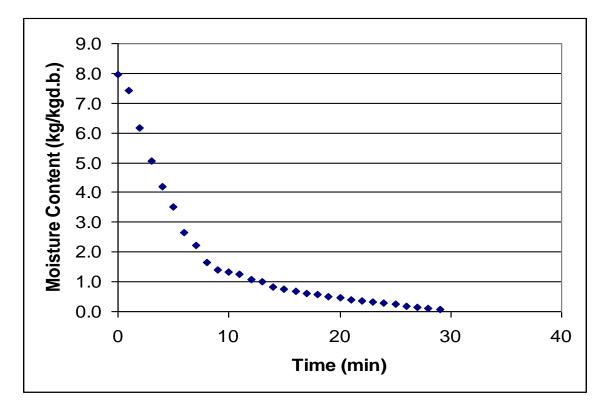


Figure 4. 9 Drying curve for MWVD of guava using combination of different power settings

4.3 Rehydration

Rehydration potential is an important parameter to be used for investigating the quality of dehydrated product, especially for those high in initial moisture content and easily undergo shrinkage during drying. In general, it is used as an indicator to measure the capability of dehydrated product to reconstitute to their initial structure when free water is added to it. Rehydration potential is quantitatively measured in term of rehydration ratio or coefficient of rehydration. Rehydration ratio RR is the mass ratio of the rehydrated sample to dehydrated sample, while coefficient of rehydration COR is a quantitative parameter which indicates the capability of dehydrated sample to reconstitute. Unity of coefficient of rehydration means absolute reconstitution of dehydrated products to its initial structure. In general, poor drying condition or wrong selection of drying method yields product with poor rehydration potential.

4.3.1 Result of Rehydrated Jackfruit

With the high penetration of microwave (\approx 4.9cm for frequency 2.45GHz), the entire portions of the sample are heated uniformly during drying. Shrinkage or case hardening which resulted by uneven heating does not occur. High vacuum level causes low boiling point of water and hence makes the dehydration process occurs at relatively low temperature. This reduces the effects of thermal degradation of the nutritive value. The texture and structure of microwave vacuum dried product are well preserved in comparison with the hot air dried product. In general, high rehydration rate made the product more competitive in commercialized owing to the desirable short rehydration time.

Table 4. 2: Kenyaration ratio and coefficient of renyaration		
Drying methods	Rehydration Ratio, RR	Coefficient of Rehydration, COR
Microwave vacuum drying	3.588	0.718

 Table 4. 2: Rehydration ratio and coefficient of rehydration

Note: Unity of COR means absolutely reconstitute to its original structure.

The residue moisture content of raw sample used= 10 ± 5 wt% (w.b.).

4.3.2 Result of Rehydrated Guava

The experimental results show that the rehydration ratio of every dried products will decrease with time. In Figure 4.10, the trend of the dried samples by different microwave power densities are quite similar, while the final rehydration ratio of each trend gives the same result. Where approximately 0,1 at the moment of about 120 mins. Meanwhile, for samples dried using different microwave power setting with the same power density of about 1kW/kg, the rehydration ratio was slightly different from each other. Figure 4.11 shows that the sample dried with high microwave power setting has the highest rehydration ratio followed by sample dried using medium high and medium microwave power setting.

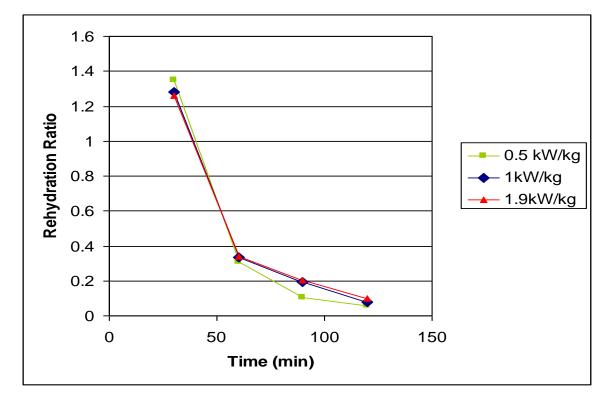


Figure 4. 10 Effect of microwave power density on the rehydration ratio of microwave vacuum dried guavas

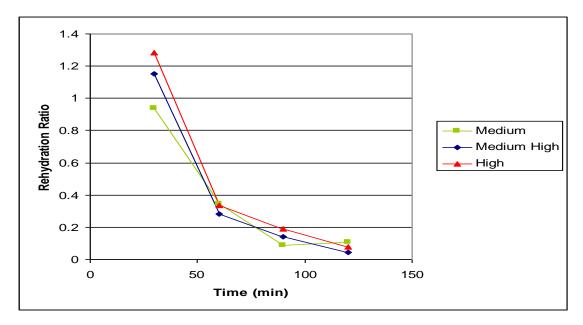


Figure 4. 11 Effect of different microwave power settings on the rehydration ratio of microwave vacuum dried guavas

Additionally, the rehydration ratio of microwave vacuum dried and conventionally dried guavas were compared. Figure 4.12 shows a higher rehydration ratio if the microwave vacuum dried sample initially compared to the conventionally dried sample. This agrees with the findings by Lin et al. (1998) with dried carrot slices. It was observed that the rehydrated microwave vacuum dried sample is lighter in colour and softer than the dried sample.

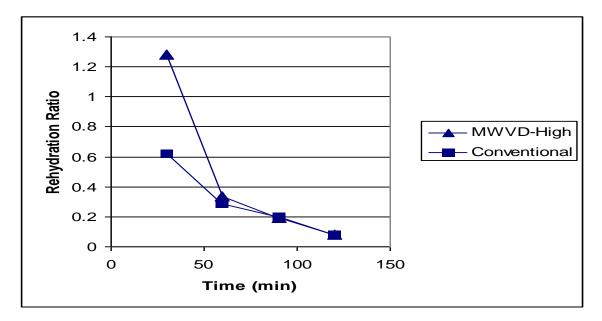


Figure 4. 12 Effect of different drying conditions on the rehydration ratio

CHAPTER 5

STUDY ON THE DRYING MECHANISM AND ENERGY TRANSFER OF PAPAYA IN VACUUM MICROWAVE SYSTEM AND THE ENZYMATIC QUALITY

5.1 Combination of Microwave Power Mode

The total drying time under 65 cmHg vacuum pressures required to reach the final moisture content was 74 min by using combination of microwave power mode and the result is illustrated in Figure 5.1. It was observed that the sample exhibited slight hot spot when drying using high microwave power mode for 4 to 5 minutes at the beginning with higher moisture content. Then, the sample was dried for 7 minutes, 10 minutes and 53 minutes, respectively using medium, medium low and low mode.

Preliminary experiments showed that using combination of microwave power mode resulted in improved appearance and drying time on the sample compared with drying using only one power mode. Drying with high microwave power had resulted in physical damages to the products e.g. scorching, over-heating or charring, when the final moisture content had achieved. In contrast, drying with low microwave power had produced better appearance of the products, however prolonged the drying time. Such physical damages might attribute to reduction in moisture content and dielectric loss since the amount of microwave energy absorbed by the material depends upon its dielectric properties and the electric field strength (Mudgett, 1990). At higher moisture content, the dielectric constant is higher as major component in fruits is water that contributes to the dielectric constant. However, as drying proceeds, the dielectric constant decreased and hence, the material absorbed fewer microwave power and heats poorly. This may lead to self-limitation of the heating as the material become relatively transparent at low moisture. Thus, as moisture decreased, lower microwave power mode was used to lessen the caused of physical damages to the products as continuous local temperature rising was avoided while moisture is removed.

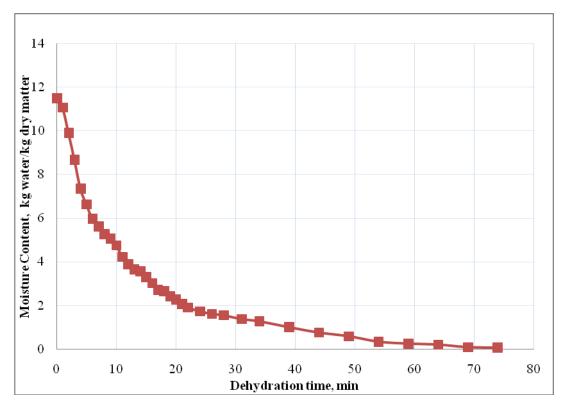


Figure 5. 1: Drying curve of microwave-vacuum dried (MVD) papaya slices (5 mm thick, combination of microwave power at P = -86.66 kPa system

pressure)

5.2 Effects of Sample Thickness

Normally in the conventional drying process, small thickness of papaya slices exhibited more rapid dehydration. Nonetheless, the same expectation was imprecise with microwave heating. Referring to the appearance of the microwave-vacuum dehydrated products obtained as illustrated in Appendix A, the product undergone severe hot spots. It was expected that the energy did not penetrate as deeply as possible in the products. If it is not, then the heating is limited to the surface and causing physical damages to the products. This had led to another important characteristic of microwave heating namely, penetration depth. As stated earlier, penetration depth is affected by dielectric properties of the materials used to heat. The dielectric properties are composed of two parts, which is the dielectric constant, ϵ' and dielectric loss, ϵ'' .

The dielectric properties of papaya were determined by predictive equations for the dielectric constant and the loss factor of the fruits at 2450 MHz by Sipahioglu and Barringer (2003). The equations were dependent on temperature (°C), moisture content (%) and wet basis ash (%). For high moisture content such as cucumber with 95.87% moisture, obtained high dielectric constant since water is a strong polar solvent in most foods and reorients in response to changes in field polarity. Appendix B shows the calculation of the approximate value of dielectric constant, ε' is 65.33 and dielectric loss, ε'' is 12.36.

Consequently, Figure 5.2 shows the effect of different thickness of the sample on drying time during microwave-vacuum drying. The sample surface area of the papaya was remaining constant at $2.0 \text{ cm} \times 2.0 \text{ cm}$ with similar mass load of 200 g of papaya. The drying rate decreased along with decreasing of papaya moisture contents. The drying times required for papaya with thickness 1.0, 2.5 and 3.0 cm thickness were 59, 50 and 70 min respectively.

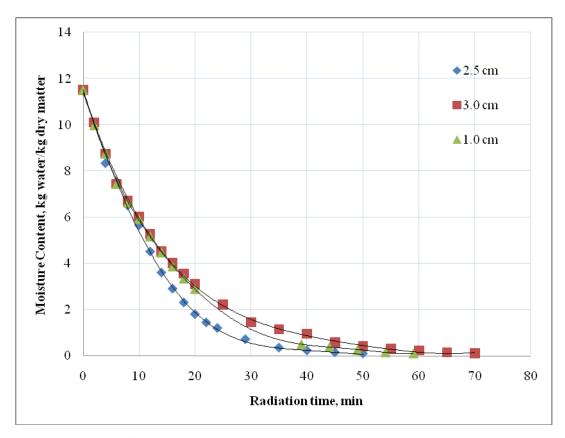


Figure 5. 2: Effect of papaya thickness on drying time during microwavevacuum drying (MVD) of papaya slices using combination of microwave power mode at -86.66 kPa system pressure.

The sample was dehydrated using combination of microwave power level. During the experiments, it was observed that the samples exhibited slight burned or hot spot after 5 to 6 minutes of radiated using the high microwave power mode at initial moisture content. Then, another 8 minutes, 6 minutes, 9 to 10 minutes and 21 to 35 minutes using medium high, medium, medium low and low microwave power mode respectively. Evidently, the optimum thickness of papaya is 2.5 cm, which exhibited higher drying rate, thus had more shorter and efficient drying time.

5.3 Rehydration Characteristic

The rehydration curves of microwave-vacuum dried papaya samples, at ambient temperature (30°C), as affected sample thickness are shown in Figure 5.3, where it further explained the effects of sample thickness on reconstitution ability. Papaya slices with thickness 1.0 cm exhibited higher rehydration ratio as well as higher rehydration rates than papaya slices with thickness of 3.0 cm. Papaya slices with thickness 2.5 cm, due to their more porous structure, had the highest rehydration ratio and rehydration rate. This indicates the optimum thickness of the papaya sample is 2.5 cm with better rehydration characteristics.

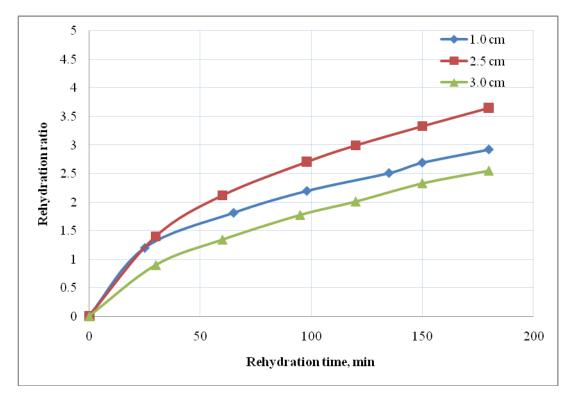


Figure 5. 3: Effect of microwave-vacuum dried papaya thickness on rehydration ratio at 30°C water temperature at -86.66 kPa system pressure.

5.4 Enzymatic activity

Enzymatic activity analysis was conducted for each dehydrated products. Each dehydrated products was grind to create larger contact surface area. Then, 5 g of the sample was diluted with 100 ml enzyme buffer. The mixture was stirred and then vacuum filtered to obtain the filtrate, which then analyzed by taking the clotting time of the substrate for 2 ml of the filtrate. For standard papain, the enzyme activity was 3.36 U/mg and the average clotting time of standard papain at concentration of 0.0436 mg/mL was 62.37 s. From the calculation using Equation 3.1, the milk clotting factor was 3.0457×10^{-4} . The average clotting time and enzyme activity for each dehydrated sample was shown in Table 5.1 by applied Equation 3.2.

Dehydration samples	Average	Enzyme activity
	clotting time(s)	(10 ⁻³ MCU/mg)
MWVD: 65 cmHg, 5mm	22.70	8.0503
MWVD: 65 cmHg, 1.0 cm	23.66	7.7237
MWVD: 65 cmHg, 2.5 cm	18.87	9.6860
MWVD: 65 cmHg, 3.0 cm	21.82	8.3750

 Table 5. 1: The effects of drying methods and papaya thickness on enzyme

 activity

CHAPTER 6

CONCLUSION AND RECOMMENDATIONS

6.1 Conclusion

A dehydration process for thermolabile materials has been successfully developed using microwave vacuum technology. Microwave is recognized as a fast heating process characterised by volumetric heating. It heats material from inside out, contrary to conventional heating which heat the surface of the material first. Incorporation of vacuum to microwave system results in numerous advantages especially in dehydration of heat sensitive materials. The creation of vacuum condition allows water to evaporate at temperature lower than normal boiling point of 100°C. This is due to the reduction of pressure below atmospheric pressure.

With the development of microwave vacuum technology, heat sensitive materials such as biological and agricultural products can be dehydrated at lower temperature thus preventing degradation of nutritional values such as vitamin C, beneficial enzyme, etc. In addition, the material's color and texture can be retained as the original.

In microwave vacuum drying of jackfruit, the qualities of the dehydrated product were rather satisfactory, as attaining the appearance greatly similar to its origin. The product was light-yellow in colour and exhibited slightly lighter in colour than its origin. Besides, the capability of product to reconstitute to its origin structure was rather high, where could be indicated by its high rehydration potential. The bulk density of product however was the lightest among the dehydrated sample because of the effects of puffing during microwave heating and the high vapour pressure gradient between the interior and the outer surface placed in vacuum.

Microwave vacuum drying (MWVD) of guavas gave fast drying time of less than 30 minutes. The dried guavas produced using this drying method are yellow in colour and is slightly puffed. The microwave drying experiments showed that the increment of microwave power densities produces higher drying rates but the sample will have higher risks of charring. The combination of microwave power densities can be used to reduce the charring of the sample. Apart from that, by combining the microwave power densities, the power requirement of the drying process may also be reduced and this can make MWVD more attractive.

Microwave-vacuum drying of papaya at vacuum pressure of 86.66kPa, with 2.5 cm thickness radiated using combination microwave power mode of high, medium high, medium, medium low and low respectively, for 6, 8, 6, 10 and 35 minutes, ensured the best drying conditions with shortest drying time and the best overall quality in perspective of reconstitution ability and enzyme retained of 0.009686 MCU/mg.

6.2 **Recommendations**

With the microwave vacuum dehydration system was developed, it is highly recommended to conduct a study on determination of optimum dehydration process such as microwave power, vacuum pressure, irradiation time and the mass load. It is essential in order to produce a high quality dehydrated product in terms of nutritional values and appearance. This will not only promote commercialization of the product but may as well encourage the adoption of microwave vacuum dehydration system in industrial level.

REFERENCES

- Andres, A., Bilbao, C. and Fito, P. (2003). "Drying Kinetics of Apple Cylinders under Combined Hot Air-Microwave Dehydration". Journal of Food Engineering. 63. 71-78.
- Arthey, D. and Dennis, C. (1991). "Vegetable Processing". New York: VCH Publishers.
- Bilbao, S.C., Andres, A., Sainz, B.C., Chiralt, A. and Fito, P. (2006). "Microwave Phenomena During Drying of Apple Cylinders". Journal of Food Engineering. 74. 160-167.
- Chen, W., Gast, K.L.B., and Smithey, S. (2000). "The effects of different freezedrying processes on the moisture content, color, and physical strength of roses and carnations". Scientia Horticulture. 84: 321-332
- Crane, J.H., Balerdi, C.F. and Campbell, R.J. (2002). "The Jackfruit (*Artocarpus heterophyllus Lam*)". University of Florida: HS-882.
- Contreras, C., Martin, M.E., Chiralt, A. and Martinez, N. (2005). "Effect of Vacuum Impregnation and Microwave Application on Structural Changes Which Occurred During Air-Drying of Apple". Journal of Food Engineering. 38. 47-477.
- Cui, Z., W., Xu, S., Y., and Sun, D., W.(2004).Microwave-vacuum drying kinetics of carrot slices. Journal of Food Engineering. 65.157-164.
- Drouzas, A.E. and Schubert, H. (1996). "Microwave Application in Vacuum Drying of Fruits". Journal of Food Engineering. 28. 203-209.

- Drouzas, A.H., Tsami, E. and Saravacos, G.D. (1999). "Microwave/ Vacuum Drying of Model Fruit Gels". Journal of Food Engineering. 39. 117-122.
- Felder, R.M. and Rousseau, R.W. (1986). "Elementary Principles of Chemical Processes". 2nd ed. New York: John Wiley& Sons. 628-629.
- Food Habits Research and Development. (1998). "Nutrient Composition of MalaysiaFoods Asean Food Habits Projek Nasional Sub-Commitee on Protein Report".Malaysia.
- Geankoplis, G.J. (2003). "Transportation Process and Separation Process Principle". 5th ed. United State: Pearson Education, Inc. 559-610.
- Giri, S.K. and Prasad, S. (2005). "Drying Kinetics and Rehydration Characteristics of Microwave-Vacuum and Convective Hot-Air Dried Mushrooms". Journal of Food Engineering. 29. 122-129.
- Gould, G.W. (1996). "New Methods of Food Preservation". UK: Blackie Academic and Professional, an Imprint of Chapman& Hall. 112-133.
- Hu, Q.G., Zhang, M., Mujumdar, A.S., Xiao, G.N. and Sun, J.C. (2006). "Drying of Edamames by Hot Air Vacuum Microwave Combination". Journal of Food Engineering. 77. 977-982.
- Kelen, A., Ress, S., Nagy, E., Pallai, V.E. and Pintye, H.K. (2005). "Practical Method for Choosing Diluent that Ensures the Best Temperature Uniformly in the Case of Pharmaceutical Microwave Vacuum Drying of a Heat Sensitive Product". Journal of Food Engineering. 62. 101-109.
- Kelen, A., Ress, S., Nagy, E. and Pintye, H.K. (2006). "Mapping of Temperature Distribution in Pharmaceutical Microwave Vacuum Drying". Powder Technology. 162. 133-137.

- Kementerian Pertanian.Malaysia. (1998). "Laporan Keluasan Tanaman Pelbagai". Malaysia.
- Kordylas, M.J. (1990). "Processing and Preservation of Tropical and Subtropical Foods". Hong Kong: Macmillan Education Ltd. 199-206.
- Leite, J.B., Mancini, M.C. and Borges, S.V. (2005). "Effect of Drying Temperature on the Quality of Dried Bananas *cv. prata* and *d,agua*". Journal of Food Technology.
- Lin, T.M., Durance, T.M. and Scaman, C.H. (1998). "Characterization of Vacuum Microwave, Air and Freeze Dried Carrot Slices". Food Research International. 31. 111-117.
- May, B.K. and Perre, P. (2002). "The Importance of Considering Exchange Surface Area Reduction to Exhibit a Constant Drying Flux Period in Foodstuffs". Journal of Food Engineering. 54. 271-282.
- Mellor, J.D. (1978). "Fundamentals of Freeze-Drying". London: Academic Press Inc. (London) Ltd. 12-14.
- McCabe, W.L., Harriott, P. and Smith, J.C. (1993). "Unit Operations of Chemical Engineering". 5th ed. New York: McGraw-Hill, inc. 767-792.
- Mujumdar, A.S. (1987). "Handbook of Industrial Drying". New York: Marcel Dekker, Inc. 327-334.
- Oetjen, G.W. and Haseley, P. (2004). "Freeze-Drying". 2nd ed. Germany: Wiley-VCH Verlag GmbH& Co. KGaA. 76-105.
- Ong, B.T., Osman, A., Voon, Y.Y., Hashim, M.D., Chew, P.M. and Kong, W.Y.
 (2006). "Chemical and Flavour Changes in Jackfruit (*Artocarpus heterophyllus Lam.*) Cultivar J3 during Ripening". Postharvest Biology and Technology. 40. 279-286.

Prabhanjan, D.G., Ramaswamy, H.S. and Raghavan, G.S.V. (1995). "Microwave-Assisted Convective Air Drying of Thin Layer Carrots". Journal of Food Engineering. 25. 283-293.

- Stewart, G.F. and Amerine, M.A. (1982). "Introduction to Food Science and Technology". 2nd ed. New York: Academic Press. 180, 188-189.
- Tsami, E., Krokida, M.K., and Drouzas, A.E. (1999). "Effect of Drying Method on the Sorption Characteristics of Model Fruit Powders". Journal of Food Engineering. 38. 381-392.
- Tooley, P. (1971). "Food and Drugs". London: John Murray. Ltd. 35-36.
- Welti-Chanes, J., Velez-Ruiz, J. and Barbosa-Canovas, G.V. (2003). "Transport Phenomena in Food Processing". New York: CRC Press LLC. 173-179.
- Woodroof, J.G. and Bor, S.L. (1986). "Commercial Fruit Processing". 2nd ed. United States of America: The Avi Publishing Co, Inc. 367-371.
- Zheng, W.C., Shi, Y.X., and Da, W.S. (2004). "Microwave-Vacuum Drying Kinetics of Carrot Slices". Journal of Food Engineering. 65. 157-164.

APPENDICES

APPENDIX A-Microwave Vacuum Dehydrated papaya

Thickness of 5mm



Thickness of 1.0 cm



Thickness of 2.5 cm



D2-4: Thickness of 3.0 cm



APPENDIX B : Calculations of Rate of Drying, Penetration Depth & Dielectric properties

Rate of Drying

Taking experimental data from hot air drying at 40°C:

$$\begin{split} \text{Moisture content (dry basis)} &= \frac{W - W_{\text{s}}}{W_{\text{s}}} \Big(\frac{\text{kg total water}}{\text{kg dry solid}} \Big) \\ &= \frac{204.38 - 18.12}{18.12} \\ &= 10.2792 \Big(\frac{\text{kg total water}}{\text{kg dry solid}} \Big) \end{split}$$

Rehydration ratio (RR) = $\frac{\text{Mass of water absorbed during rehydration}}{\text{Mass of water removed during drying}}$ = $\frac{3.06}{5.02}$ = 0.6096

Rate of drying
$$\frac{dX}{dt} = \frac{10.2792 - 11.4983}{30 - 0}$$
$$= -0.0406 \left(\frac{\text{kg total water}}{\text{kg dry solid. min}}\right)$$

vs average X =
$$\frac{11.4983 + 10.2792}{2}$$

= 10.8888

Penetration Depth & Dielectric properties

Fruits	Temperature (°C)	Moisture content	Ash content (wet
		(%)	basis)
Papaya	53	92	0.5

Dielectric constant				
Fruit	$\epsilon' = 22.12 + 0.2379T + 0.5532M - 0.0005132T^2 - 0.003866MT$			
s	$= 22.12 + 0.2379(53) + 0.5532(92) - 0.0005132(53)^2 - 0.003866(93)^2 - 0.00386(93)^2 - 0.003866(93)^2 - 0.003866(93)^2 - 0.$			
	= 65.33			
Dielectric loss factor				
Fruit s	$\label{eq:energy} \begin{split} \epsilon^{\prime\prime} &= 33.41 - 0.4415T + 0.0014T^2 - 0.1746M + 1.438A + 0.001578MT \\ & + 0.2289AT \end{split}$			
	$= 33.41 - 0.4415(53) + 0.0014(53)^2 - 0.1746(92) + 1.438(0.5) + 0.001578(92)(53) + 0.2289(0.5)(53)$			
	= 12.36			

T=temperature (°C), M=Moisture (%), A=wet basis ash (%)

For microwave frequency of 2450 MHz:

$$\tan^2 \delta = \varepsilon'' / \varepsilon' = \frac{12.36}{65.33} = 0.1892$$

$$\lambda_{o} = \frac{c}{f} = \frac{3 \times 10^{10} \, \text{cm/s}}{2450 \, \text{MHz}} = 12.24 \, \text{cm}$$

Thus,

Penetration depth, D =
$$\frac{\lambda_o \sqrt{2}}{2\pi} \left[\epsilon' \left(\sqrt{1 + \tan^2 \delta} - 1 \right) \right]^{-\frac{1}{2}}$$

= $\frac{12.24 \text{ cm } \sqrt{2}}{2\pi} \left[12.36 \left(\sqrt{1 + 0.1892} - 1 \right) \right]^{-\frac{1}{2}}$
= 2.6048 cm