

T.R.
GEBZE TECHNICAL UNIVERSITY
GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

**EFFECT OF DIFFERENT DRYING METHODS ON DRYING
CHARACTERISTICS AND QUALITY OF BEE POLLEN**

AYLA IŞIK
A THESIS SUBMITTED FOR THE DEGREE OF
DOCTOR OF PHILOSOPHY
DEPARTMENT OF CHEMICAL ENGINEERING

GEBZE
2022

T.R.
GEBZE TECHNICAL UNIVERSITY
GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

**EFFECT OF DIFFERENT DRYING
METHODS ON DRYING
CHARACTERISTICS AND QUALITY OF
BEE POLLEN**

AYLA IŞIK
**A THESIS SUBMITTED FOR THE DEGREE OF
DOCTOR OF PHILOSOPHY
DEPARTMENT OF CHEMICAL ENGINEERING**

THESIS SUPERVISOR
PROF. DR. MURAT ÖZDEMİR

GEBZE

2022



DOKTORA JÜRİ ONAY FORMU

GTÜ Fen Bilimleri Enstitüsü Yönetim Kurulu'nun 30/06/2022 tarih ve 2022/33 sayılı kararıyla oluşturulan jüri tarafından 20/07/2022 tarihinde tez savunma sınavı yapılan Ayla Işık'ın tez çalışması Kimya Mühendisliği Anabilim Dalında DOKTORA tezi olarak kabul edilmiştir.

JÜRİ

ÜYE (TEZ DANIŞMANI) : Prof. Dr. Murat Özdemir

ÜYE : Prof. Dr. Mehmet Melikoğlu

ÜYE : Prof. Dr. İbrahim Doymaz

ÜYE : Dr. Öğr. Üyesi Bengü Ergüden

ÜYE : Dr. Öğr. Üyesi Aylin Seylam Küşümler

ONAY

Gebze Teknik Üniversitesi Enstitüsü Yönetim Kurulu'nun
...../...../..... tarih ve/..... sayılı kararı.

SUMMARY

Bee pollens were hot air dried at 40, 45, 50, 55 and 60°C, infrared dried at 50, 62, 74 and 88 W, microwave dried at 180, 360, 540, 720 and 900 W and freeze dried. Effective moisture diffusivity (D_{eff}) values for the bee pollens varied from 1.90×10^{-11} to $0.04 \times 10^{-8} \text{ m}^2/\text{s}$. Activation energy (E_a) values were 42.96 kJ/mol, 71.68 W/g and 5.89 W/g for hot air, infrared and microwave dryings, respectively. Crude proteins, fats and total carbohydrates of the dried bee pollens were affected by the drying temperature, and infrared and microwave power levels, but not the freeze drying. Freeze drying was a highly effective drying method in retention of vitamin C in bee pollen. Freeze dried bee pollen had the highest solubility in water. L^* , a^* and b^* values were affected by the drying temperature, and infrared and microwave power levels during drying, but not the freeze drying. The color of the freeze dried bee pollen was found to be the closest to the color of the fresh bee pollen. Morphological change and shrinkage were minimal in freeze dried bee pollens which took the highest sensory scores. Freeze dried bee pollen retained its quality attributes better than hot air, infrared and microwave dried bee pollens. Freeze drying is more appropriate method for producing high quality dried bee pollen than the other drying methods, but it takes so long. Hot air drying at 40°C is recommended as a cost effective and faster alternative to freeze drying.

Key Words: Bee pollen, Hot air drying, Infrared drying, Microwave drying, Freeze drying, Quality.

ÖZET

Arı polenleri 40, 45, 50, 55 ve 60°C sıcaklıktaki sıcak havayla, 50, 62, 74 ve 88 Watt'da kızılötesi ışınlarla, 180, 360, 540, 720 ve 900 Watt'da mikrodalgada ve dondurularak kurutulmuştur. Arı polenlerinin efektif nem yayılımı (D_{eff}) değerleri $1,90 \times 10^{-11}$ ile $0,04 \times 10^{-8}$ m²/s arasında değişmiştir. Sıcak hava, kızılötesi ve mikrodalga kurutma için aktivasyon enerjisi (E_a) değerleri sırasıyla 42.96 kJ/mol, 71.68 W/g ve 5.89 W/g olarak bulunmuştur. Kurutulmuş arı polenlerinin içindeki ham proteinler, yağlar ve toplam karbonhidratlar kurutma sıcaklığından, kurutma sırasındaki kızılötesi ve mikrodalga güç seviyelerinden etkilenmiş, ancak dondurarak kurutmadan etkilenmemiştir. Dondurarak kurutma arı polenindeki C vitamininin muhafazasında oldukça etkili bir kurutma yöntemi olmuştur. Dondurularak kurutulmuş arı poleni suda en yüksek çözünürlük değerini vermiştir. L^* , a^* ve b^* değerleri kurutma sırasında kurutma sıcaklığı ile kızılötesi ve mikrodalga güç seviyelerinden etkilenmiş, ancak dondurarak kurutmadan etkilenmemiştir. Dondurularak kurutulmuş arı polenin renginin, taze arı polenin rengine en yakın olduğu bulunmuştur. Dondurularak kurutulmuş arı polenlerinde morfolojik değişim ve büzülme minimum düzeyde olmuş, en yüksek duyusal puanları almıştır. Dondurularak kurutulmuş arı poleni, kalite özelliklerini sıcak hava, kızılötesi ve mikrodalga ile kurutulmuş arı polenlerinden daha iyi korumuştur. Yüksek kaliteli kurutulmuş arı poleni elde etmek için dondurarak kurutmanın diğer kurutma yöntemlerine göre daha uygun olduğu, ancak çok uzun sürdüğü tespit edilmiştir. 40°C sıcaklıktaki sıcak havayla kurutma işlemi, dondurarak kurutmaya kıyasla daha uygun maliyetli ve daha hızlı bir alternatif olarak önerilebilir.

Anahtar Kelimeler: Arı poleni, Sıcak hava kurutma, Kızılötesi kurutma, Mikrodalga kurutma, Dondurarak kurutma, Kalite.

ACKNOWLEDGMENTS

Firstly, I would like to express my deep and sincere gratitude to my supervisor, Prof. Dr. Murat ÖZDEMİR, who has always been with me with his knowledge and support from the beginning to the end of my thesis. He led me with his valuable contributions and criticism throughout my Ph. D. thesis.

I wish to express my sincere appreciation to Prof. Dr. İbrahim DOYMAZ for his valuable comments and guidance. I would also like to thank to my thesis defense members, Prof. Dr. Mehmet MELİKOĞLU, Asst. Prof. Dr. Bengü ERGÜDEN and Asst. Prof. Dr. Aylin Seylam KÜŞÜMLER for their valuable contributions and constructive comments.

I deeply appreciate to my family for everything they have done for me. I would also like to extend my thanks to my parents for their love and support.

TABLE of CONTENTS

	<u>Page</u>
SUMMARY	v
ÖZET	vi
ACKNOWLEDGMENTS	vii
TABLE of CONTENTS	viii
LIST of ABBREVIATIONS and ACRONYMS	xi
LIST of FIGURES	xiii
LIST of TABLES	xv
1. INTRODUCTION	1
1.1. Hypothesis	3
1.2. Objectives	3
2. MATERIALS and METHODS	4
2.1. Sample Preparation	4
2.2. Pollen Analysis	4
2.3. Drying Experiments	4
2.3.1. Hot Air Drying	4
2.3.2. Infrared Drying.	5
2.3.3. Microwave Drying	5
2.3.4. Freeze Drying	6
2.4. Determination of Effective Moisture Diffusivity	6
2.5. Calculation of Activation Energy for Hot Air Drying	8
2.6. Calculation of Activation Energies for Infrared and Microwave Dryings	9
2.7. Determination of Protein Content	10
2.8. Determination of Fat Content	10
2.9. Determination of Ash	11
2.10. Determination of Total Carbohydrates	11
2.11. Determination of Vitamin C	11
2.12. Determination of Solubility Index	12
2.13. Color Measurements	12

2.14. Scanning Electron Microscopy (SEM) Analysis	13
2.15. Sensory Evaluation	13
2.16. Statistical Analysis	14
3. RESULTS and DISCUSSION	15
3.1. Hot Air Drying	15
3.1.1. Drying Characteristics	15
3.1.2. Effective Moisture Diffusivity and Activation Energy for Hot Air Drying	17
3.1.3. Effect of Hot Air Drying Temperature on Physicochemical Properties	18
3.1.4. Effect of Hot Air Drying on Solubility Index	22
3.1.5. Effect of Hot Air Drying on Color	22
3.1.6. Morphological Structure of Hot Air Dried Bee Pollen	23
3.1.7. Effect of Hot Air Drying on Sensory Attributes	25
3.2. Infrared Drying	26
3.2.1. Drying Characteristics	26
3.2.2. Effective Moisture Diffusivity and Activation Energy for Infrared Drying	27
3.2.3. Effect of Infrared Drying Power Level on Physicochemical Properties	29
3.2.4. Effect of Infrared Drying on Solubility Index	32
3.2.5. Effect of Infrared Drying on Color	32
3.2.6. Morphological Structure of Infrared Dried Bee Pollen	33
3.2.7. Effect of Infrared Drying on Sensory Attributes	36
3.3. Microwave Drying	36
3.3.1. Drying Characteristics	36
3.3.2. Effective Moisture Diffusivity and Activation Energy for Microwave Drying	38
3.3.3. Effect of Microwave Drying Power Level on Physicochemical Properties	40
3.3.4. Effect of Microwave Drying on Solubility Index	44
3.3.5. Effect of Microwave Drying on Color	44
3.3.6. Morphological Structure of Microwave Dried Bee Pollen	46
3.3.7. Effect of Microwave Drying on Sensory Attributes	48

3.4. Freeze Drying	49
3.4.1. Drying Characteristics	49
3.4.2. Effective Moisture Diffusivity for Freeze Drying	50
3.4.3. Effect of Freeze Drying on Physicochemical Properties	51
3.4.4. Effect of Freeze Drying on Solubility Index	53
3.4.5. Effect of Freeze Drying on Color	53
3.4.6. Morphological Structure of Freeze Dried Bee Pollen	55
3.4.7. Effect of Freeze Drying on Sensory Attributes	55
4. CONCLUSIONS	57
REFERENCES	58
BIOGRAPHY	64

LIST of ABBREVIATIONS and ACRONYMS

<u>Abbreviations and Acronyms</u>	<u>Explanations</u>
ΔE	: Total color difference
D_{eff}	: Effective moisture diffusivity
D_o	: Constant or pre-exponential factor
E_a	: Activation energy
π	: Pi number
L	: Half thickness of slab
M	: Moisture content
M_e	: Equilibrium moisture content
M_o	: Initial moisture content
M_t	: Moisture content of bee pollen at any given time
M	: Sample weight
n	: Positive integer
P	: Infrared or microwave power level
R	: Universal gas constant
T	: Drying air temperature
t	: Time
V	: Volume of DCIP used in experiment
V_o	: Volume of DCIP for blank
W	: Watt
W_d	: Weight of bee pollen sample after drying
W_r	: Weight of sample after immersion
W_s	: Weight of dry solid content of bee pollen sample
W_t	: Weight of bee pollen sample at any time
W_o	: Weight of bee pollen sample before drying
x	: Distance in the solid
AOAC	: Association of Official Analytical Chemists
DCIP	: 2,6-dichlorophenol-indophenol
H_2SO_4	: Sulfuric acid

MR : Moisture ratio
NaOH : Sodium hydroxide
SI : Solubility index

LIST of FIGURES

<u>Figure No:</u>	<u>Page</u>
1.1: Bee pollen traps and multi-flora bee pollen.	1
3.1: Variation of moisture content with drying time for hot air drying of bee pollen at different temperature.	16
3.2: Effect of drying air temperature and drying time on the moisture ratio of bee pollen samples.	16
3.3: Arrhenius-type relationship between effective moisture diffusivity and reciprocal of absolute temperature.	18
3.4: Solubility index of the bee pollen hot air dried at various temperatures. Different letters indicate significant differences ($p \leq 0.05$) among treatments.	22
3.5: Scanning electron micrographs of (a) fresh and hot air dried bee pollen samples at (b) 40°C, (c) 45°C, (d) 50°C, (e) 55°C and (f) 60°C. Scale bar is 10 μm . Magnification: 2000 $\times$.	24
3.6: Variation of moisture content with time for the infrared drying of bee pollen at different infrared powers.	26
3.7: Effect of infrared power level and drying time on the moisture ratio of bee pollen samples.	27
3.8: Arrhenius-type relationship between effective moisture diffusivity and infrared powers.	28
3.9: Solubility index of the bee pollen infrared dried at various infrared powers. Different letters indicate significant differences ($p \leq 0.05$) among treatments.	32
3.10: Scanning electron micrographs of (a) fresh and infrared dried bee pollens at different infrared powers (b) 50 W, (c) 62 W, (d) 74 W and (e) 88 W. Scale bar is 10 μm . Magnification: 2000 $\times$.	35
3.11: Variation of moisture content with time for the microwave drying of bee pollen at different microwave output powers.	37
3.12: Effect of microwave power and drying time on the moisture ratio of bee pollen samples.	38

3.13:	Variations of $\ln(MR)$ with drying time at different microwave powers.	39
3.14:	Arrhenius-type relationship between effective moisture diffusivity and microwave power.	40
3.15:	Solubility index of bee pollen microwave dried at various microwave power levels. Different letters indicate significant differences ($p \leq 0.05$) among treatments.	44
3.16:	Scanning electron micrographs of (a) fresh and microwave dried bee pollens at different microwave power levels (b) 180 W, (c) 360 W, (d) 540 W, (e) 720 W and (f) 900 W. Scale bar is 10 μm . Magnification: 2000 $\times$.	47
3.17:	Variation of moisture content with time for the freeze dried of bee pollen.	49
3.18:	Moisture ratio versus time for the freeze dried bee pollen.	50
3.19:	Variation of $\ln(MR)$ versus drying time.	50
3.20:	Solubility index of freeze dried bee pollen. Different letters indicate significant differences ($p \leq 0.05$) among treatments.	53
3.21:	Scanning electron micrographs of (a) fresh and (b) freeze dried bee pollens. Scale bar is 10 μm . Magnification: 2000 $\times$.	55

LIST of TABLES

<u>Table No:</u>	<u>Page</u>
3.1: Effective moisture diffusivity values obtained for bee pollen samples dried at different drying temperatures.	17
3.2: Physicochemical properties of fresh and hot air dried bee pollens.	21
3.3: Color parameters of fresh and hot air dried bee pollen samples.	23
3.4: Sensory attributes of fresh and hot air dried bee pollen samples.	25
3.5: Effective moisture diffusivity values obtained for bee pollen samples dried at different infrared powers.	28
3.6: Physicochemical composition of fresh and infrared dried bee pollens.	31
3.7: Color parameters of fresh and infrared dried bee pollen samples.	33
3.8: Effect of infrared power levels on the sensory scores of the infrared dried bee pollen samples.	36
3.9: Effective moisture diffusivity values obtained for bee pollen samples microwave dried at different microwave powers.	39
3.10: Physicochemical composition of fresh and microwave dried bee pollen.	43
3.11: Color parameters of fresh and microwave dried bee pollen samples.	45
3.12: Effect of microwave power levels on the sensory scores of the microwave dried bee pollen samples.	48
3.13: Physicochemical properties of fresh and freeze dried bee pollens.	52
3.14: Color parameters of fresh and freeze dried bee pollen samples.	54
3.15: Effect of freeze drying on the sensory scores of freeze dried bee pollen samples.	56

1. INTRODUCTION

Bee pollen is made by worker bees as the result of the agglutination of flower pollen, collected at the entrance of the hive, and contains nectar and honeybee salivary substances [Campos et al., 2008]. The pollen grains (Figure 1.1) come in a wide variety of shapes, sizes, color and surface [Khalifa et al., 2021]. During collecting trips, bees pack pollen into pollen baskets on their hind legs, and it is stored inside the hive separately from the nectar cells [Almeida-Muradian et al., 2005], [Khalifa et al., 2021].



Figure 1.1. Bee pollen traps and multi-flora bee pollen.

Bee pollen is an important apicultural product containing proteins, carbohydrates, lipids, minerals and fibers [Dias et al., 2016], [Canale et al., 2016], [Borel et al., 2020], [Thakur and Nanda, 2020a,b], [Węglińska et al., 2020]. Depending on different species of bee pollen, geographical region and seasonal conditions, protein content may range from 10 to 40%, carbohydrate content from 13 to 55% and lipid content from 1 to 10% [Estevinho et al., 2012], [Li et al., 2018], [Kostić et al., 2020], [Thakur and Nanda, 2020b], [Węglińska et al., 2020]. Due to its rich nutritional value, bee pollen has been used for medical, therapeutic and nutritional purposes [Denisow and Denisow-Pietrzyk, 2016], [Li et al., 2018], [Borel et al., 2020], [Kostić et al., 2020], [Thakur and Nanda, 2020a,b]. Bee pollen with high moisture content (20-30 g water/100 g product in wet basis or 25-42.9 g water/100 g dry solid in dry basis) is perishable after a short period of time from the harvest because it is highly susceptible to microbial attacks [De-Melo et al., 2016]. Therefore, the moisture content of the bee pollen is regarded to be between 6 and 10

g water/100 g product or between 6.4 and 11.1 g water/100 g dry solid to obtain a shelf stable product [Bogdanov, 2004], [Campos et al., 2008]. In order to obtain a shelf stable product, dried bee pollen should have no more than 10% moisture to comply with the GB/T 19,330–2003 standard [Schulte et al., 2008] and no more than 8% moisture to comply with the NY 5137–2002 standard [Bonvehí and Jordà, 1997].

Drying is the most commonly used and inexpensive method to prevent rapid fermentation and microbial spoilage, and prolong the shelf life of food products by eliminating the use of additives [Reis et al., 2013], [Darvishi, 2017], [Doymaz, 2018]. Sun drying is the most widely used traditional method for the drying of the bee pollen, but it has some disadvantages such as long drying times, high risk of microbial contamination, high susceptibility to insect infestation and contamination with foreign materials, requirement of large area for drying, high man power costs and almost no control of drying conditions. On the other hand, hot air drying has reasonably shorter process time, lower risk of microbial contamination, more effective sanitary conditions and better control of drying conditions than the sun drying. Therefore, hot air drying has been used in many industrial drying applications including dehydration of food and agricultural products.

Infrared drying is a very effective drying method employed for the drying of agricultural products. The advantages of infrared energy in food dehydration are decreased drying time, high quality food products, high energy efficiency and uniform temperature in the dried product [Sadin et al., 2014], [Doymaz, 2015]. Infrared drying is used in the drying of several agricultural products including tomatoes [Doymaz, 2012], [Sadin et al., 2014], carrot [Doymaz, 2015], strawberry [Adak et al., 2017] and red pepper [Cao et al., 2016].

Microwave drying is an emerging method of drying, and it has been gaining more popularity in food processing in recent years [Chandrasekaran et al., 2013], [Soni et al., 2020]. Microwave drying has several advantages as compared to traditional convective drying and offers opportunities to shorten drying time, retain nutritional value and improve the final quality of dried products [Wojdyło et al., 2016], [Darvishi, 2017], [Shewale et al., 2019]. Microwave drying is commercially used particularly for drying of snacks, spices, pasta, instant noodles, rice and other similar ingredients [Phukasmas and Songsermpong, 2019], [Soni et al., 2020].

Freeze drying produces high quality products characterized by very low moisture content, good sensory and nutritional properties. However, freeze drying is

comparatively more expensive and takes longer times than other drying methods. Low drying rates lead to relatively low yields, and it has high capital and energy costs mainly due to cooling and vacuum systems. High energy consumption during freeze drying is the main obstacle limiting its wider application [Wojdyło et al., 2016], [Ratti, 2001].

Bee pollen is gaining growing attention as a potential source of energy and proteins for human consumption in the USA, Europe and Asia [Thakur and Nanda, 2018], [Kostić et al., 2020]. Knowledge on the drying kinetics of food and agricultural products is essential to investigate the drying behavior and understand the drying mechanism for a special product better, design a new drying system or improve the existing one, optimize the drying conditions and control the drying operation. Although the effect of drying on the quality and physicochemical properties of many biological products has been investigated, the information on drying characteristics of bee pollen is scarce. In addition, study on effective moisture diffusivity and activation energy of bee pollen has not yet been reported. Determination of the effect of hot air drying temperature, infrared power levels, microwave powers and freeze drying conditions on the drying time, bee pollen quality, physicochemical properties, morphological structure and sensory characteristics of bee pollen is important to obtain bee pollen with high quality characteristics.

1.1. Hypothesis

Dried bee pollen with high quality characteristics can be obtained using an appropriate drying method.

1.2. Objectives

The main objectives of this study were to (1) study drying kinetics of bee pollen during different drying processes, (2) compute effective moisture diffusivities and activation energies, and (3) investigate the effect of different drying methods on quality, physicochemical properties, morphological structure and sensory characteristics of bee pollen.

2. MATERIALS and METHODS

2.1. Sample Preparation

Fresh bee pollen used in this study was obtained from a beekeeper in the Kemaliye-Erzincan region of Turkey, located at 39.74 latitude and 39.49 longitude, and situated at 1195 m above sea level. Fresh bee pollens were transferred to the laboratory under chilled conditions within the same day they were harvested and kept in a refrigerator (+ 4 °C) prior to the drying experiments. Moisture contents of fresh and dried bee pollens were measured by hot air oven method at 105°C [AOAC, 2005].

2.2. Pollen Analysis

Pollen analysis was done based on the method described by Bayram (2019) where pollen types were classified as dominant pollen (> 45%), secondary pollen (16–45%), important minor pollen (3–15%) and minor pollen (< 3%). Approximately 3 g of whole bee pollen pellets were examined under a microscope (Nikon Eclipse LV 150L, Nikon Corp., Tokyo, Japan) at 500× magnifications for microscopic identification of bee pollen pellets. Reference collection slides obtained from the Institute for Apicultural Research and Development were used for the recognition of bee pollen and determination of botanical origin.

2.3. Drying Experiments

2.3.1. Hot Air Drying

Hot air drying trials were carried out at five different temperatures (40°C, 45°C, 50°C, 55°C and 60°C) at a constant air velocity of 2 m/s in a cabinet dryer (APV & Pasilac Ltd., Carlisle, Cumbria, UK) as previously described by Doymaz (2004). Drying air temperatures were measured with a digital temperature controller unit integrated with the cabinet dryer operating with an accuracy of $\pm 0.5^{\circ}\text{C}$. Air velocity was measured with an anemometer (AM-4201, Lutron, Taipei, Taiwan) with

a sensitivity of ± 0.1 m/s. Prior to the drying trials, the convective hot air dryer had been operated empty for 30 min so that a steady-state condition would have been attained within the dryer. The bee pollen samples ($25 \text{ g} \pm 0.5 \text{ g}$) were placed uniformly in stainless steel drying trays in a single layer, having a thickness of about 0.004 m. The trays were taken out at 10 ± 0.1 min intervals, and the moisture loss of the samples was determined using a digital balance (Ohaus Scout[®] Pro, Ohaus Corp., NJ, USA) with an accuracy of ± 0.01 g. All the trials were conducted in triplicate until the final moisture content of bee pollens reached to 0.088 ± 0.002 g water/g dry solid. The dried products were packed under vacuum with an aluminum layer containing a composite film barrier to oxygen and water vapor.

2.3.2. Infrared Drying

A moisture analyzer with a 250 W halogen lamp (Snijders moisture balance, Snijders b.v., Tilburg, Holland) was used for infrared drying experiments. Prior to the drying trials, the samples weighing approximately 25 ± 0.5 g were separated evenly and homogeneously over the pan, having a thickness of about 0.004 m. The drying experiments were performed at infrared power levels between 50 and 88 W. The infrared power level was set in control unit of the equipment, and it was measured using an energy meter (Peak Tech 9035, Ahrensburg, Germany). During the drying trials, the pan was taken out at 10 minutes' intervals, and the weight loss was recorded using a digital balance (Ohaus Scout[®] Pro, Ohaus Corp., NJ, USA) with an accuracy of ± 0.01 g. All the trials were conducted in triplicate until the final moisture content of bee pollens reached to 0.081 ± 0.003 g water/g dry solid. The dried products were packed under vacuum with a composite film containing an aluminum layer barrier to oxygen and water vapor.

2.3.3. Microwave Drying

A domestic digital microwave oven (Arcelik MD 599, Turkey) with technical features of 230 V and 50 Hz operating at a frequency of 2450 MHz was used. The dimensions of the microwave cavity were 505 mm $\times$ 574 mm $\times$ 376 mm. in size. The microwave oven had the capability of operating at microwave powers between 180 and 900 W. Microwave output powers and processing time were adjusted with the

aid of a digital controlling unit located on the microwave oven. Microwave drying trials were carried out at 180, 360, 540, 720 and 900 W microwave powers. Samples of whole bee pollen pellets (10 ± 0.5 g) were uniformly distributed into the Petri dishes in the form of a thin layer with a thickness of about 4 mm and placed on the center of the rotating glass of the microwave oven. Petri dishes were taken out at 30 s intervals from the microwave oven, and the weight loss of the samples was determined using a digital balance (Ohaus Scout® Pro, Ohaus Corp., NJ, USA) with an accuracy of ± 0.01 g. Surface temperatures, including the initial temperature, of bee pollen samples were measured with an infrared thermometer (Fluke 566, Fluke Corp., Everett, WA, USA) immediately when each sample was withdrawn from the microwave oven. The samples were dried until the final moisture content of bee pollens reached 0.077 ± 0.015 g water/g dry solid for the dried bee pollens to comply with GB/T 19,330–2003 [Schulte et al., 2008] and NY 5137–2002 standards [Bonvehí and Jordà, 1997]. All the microwave drying trials were conducted in triplicate. The microwave dried bee pollens were packed under vacuum with an aluminum layer containing a composite film barrier to oxygen and water vapor.

2.3.4. Freeze Drying

Freeze drying experiments were performed in a freeze dryer (Virtis Ultra 25 Super XL, USA). A sample of bee pollen pellets (15 ± 0.5 g) was placed on the tray of the freeze dryer at -30°C for 3 hours for complete freezing. Drying was performed at 20°C by setting the drying chamber pressure to 50 Pa and keeping it constant during the drying. Condenser temperature was set to -70°C . The drying was continued until the weight of the bee pollen samples reached to a level corresponding to moisture content of 0.088 ± 0.002 g water/g dry solid. Drying trials were performed triplicates. The freeze dried products were packed under vacuum with a composite film containing an aluminum layer barrier to oxygen and water vapor immediately after the freeze drying.

2.4. Determination of Effective Moisture Diffusivity

Moisture contents of the bee pollen at any given time during drying were calculated according to Eq. (2.1):

$$M_t = \frac{W_t - W_s}{W_s} \quad (2.1)$$

where M_t is the moisture content at any given time (g water/g dry solid), W_t is the weight of sample at any time (g) and W_s is the weight of dry solid content of sample (g).

Drying curves were constructed using moisture ratio (MR) values (dimensionless) calculated from Eq. (2.2) given below:

$$MR = \frac{M_t - M_e}{M_0 - M_e} \quad (2.2)$$

where M_0 is the initial moisture content (g water/g dry solid), M_t is the moisture content at any given time (g water/g dry solid) and M_e is the equilibrium moisture content (g water/g dry solid). M_e is significantly much lower than M_t and M_0 for long drying times so it can be negligible [Baomeng et al., 2014], [Ojha et al., 2017] and Eq. (2.2) can be simplified to:

$$MR = \frac{M_t}{M_0} \quad (2.3)$$

Fick's second law of diffusion equation (Eq. 2.4), symbolized as a mass diffusion equation for drying agricultural products in a falling rate period, was used to calculate the effective moisture diffusivity (D_{eff}):

$$\frac{\partial M}{\partial t} = (D_{eff}) \frac{\partial^2 M}{\partial x^2} \quad (2.4)$$

where M is the moisture content (g water/g dry solid), t is the time (s), x is the distance in the solid (m), and D_{eff} is the effective moisture diffusivity (m^2/s).

The solution of Eq. (2.4) for an infinite slab is given in Eq. (2.5) by Crank (1975) assuming that (i): initial moisture is uniformly distributed throughout the sample, (ii): moisture migration occurs by diffusion only in one dimension, (iii): external resistance and shrinkage are negligible and (iv): temperature and diffusivity are constant. The appropriate initial and boundary conditions are given below:

$$t = 0, \quad 0 \leq x \leq L, \quad M = M_0$$

$$t > 0, \quad x = 0, \quad \frac{\partial M}{\partial t} = 0$$

$$t > 0, \quad x = L, \quad M = M_e$$

$$MR = \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left(-\frac{(2n+1)^2 \pi^2 D_{eff} t}{4L^2}\right) \quad (2.5)$$

where L is the half thickness of slab (m), and n is the positive integer.

The linear solution of Eq. (2.5), which is the simple approach to the series equation, by considering only the first term of the series is significant, yields the following equation:

$$MR = \frac{8}{\pi^2} \exp\left(-\frac{\pi^2 D_{eff} t}{4L^2}\right) \quad (2.6)$$

When the natural logarithm of both sides of the Eq. (2.6) is taken, it simplifies to:

$$\ln MR = \ln \frac{8}{\pi^2} - \frac{\pi^2 D_{eff} t}{4L^2} \quad (2.7)$$

From Eq. (2.7), a plot of $\ln MR$ versus time gives a straight line with a slope as given in Eq. (2.8) from which D_{eff} can be determined.

$$\text{Slope} = \frac{\pi^2 D_{eff}}{4L^2} \quad (2.8)$$

2.5. Calculation of Activation Energy for Hot Air Drying

Arrhenius type equation was used to illustrate the relationship between the effective diffusivity and temperature for the calculation of the activation energy. The activation energy is calculated for hot air drying using Eq. (2.9):

$$D_{\text{eff}} = D_0 \exp\left(\frac{-E_a}{R(T + 273.15)}\right) \quad (2.9)$$

where D_0 is a constant or pre-exponential factor (m^2/s), E_a is the activation energy (kJ/mol), R is the universal gas constant (8.3143 J/mol K) and T is the drying air temperature ($^{\circ}\text{C}$).

The natural logarithm of the linear form of Eq. (2.9) is given in Eq. (2.10):

$$\ln(D_{\text{eff}}) = \ln(D_0) - \frac{E_a}{R(T + 273.15)} \quad (2.10)$$

The slope ($-E_a/R$) of the graph of $\ln(D_{\text{eff}})$ versus $1/(T + 273.15)$ is used to calculate E_a .

2.6. Calculation of Activation Energies for Infrared and Microwave Dryings

An Arrhenius type equation was used in a modified form to illustrate the relationship between the effective diffusivity and the power level and sample weight (m/P) instead of the temperature for calculation of the activation energy because temperature is not a directly measurable quantity in the standard infrared and microwave drying processes in this study. So, the activation energy was calculated using Eq. (2.11) (Dadali et al., 2007; Demiray et al., 2017; Kouhila et al., 2020).

$$D_{\text{eff}} = D_0 \exp\left(-\frac{E_a m}{P}\right) \quad (2.11)$$

where D_{eff} is the effective moisture diffusivity (m^2/s), D_0 is a constant or pre-exponential factor (m^2/s), E_a is the energy of activation (W/g), P is the infrared or microwave power level (W) and m is the sample weight (g).

The natural logarithm of the linear form of Eq. (2.11) is given in Eq. (2.12):

$$\ln(D_{\text{eff}}) = \ln(D_0) - \frac{E_a m}{P} \quad (2.12)$$

E_a is calculated from the slope of the graph of $\ln(D_{eff})$ versus m/P , and D_0 is calculated from the intercept.

2.7. Determination of Protein Content

Fresh and dried bee pollen samples were analyzed for crude protein content according to the Kjeldahl's method as described in AOAC (2005). One gram of bee pollen sample was taken into the combustion tube. Twenty-five ml of 98% concentrated sulfuric acid (H_2SO_4) and combustion mixture (7 grams of potassium sulfate and 0.8 grams of copper sulfate) were added into the combustion tube as catalyst, combusted at 350-400°C. The combustion was carried out through digestion unit till transparent residue was obtained. After cooling the residue to room temperature, 100 ml of deionized water was added. The mixture was neutralized with 125 ml of 40% NaOH solution in order to release gaseous ammonia. The neutralized solution was distilled through Kjeldahl's distillation apparatus. The ammonia liberated was trapped in 20 ml of 4% boric acid solution containing indicators (methyl red and ethylene blue). The amount of ammonia collected was titrated against 0.1 N H_2SO_4 to a purple end point. A blank determination was done following similar procedure, without the test sample. Factor of 6.25 was used to convert total nitrogen to proteins [AOAC, 2005]. The percent protein is calculated according to the Eq. (2.13):

$$\text{Crude protein (\%)} = \text{Nitrogen (\%)} \times 6.25 \quad (2.13)$$

2.8. Determination of Fat Content

Fat contents of fresh and dried bee pollen samples were determined using Soxhlet extractor with hexane as solvent according to the procedure given in AOAC (2005). Before the extraction, an empty extraction flask was heated, dried, cooled and weighed. Five grams of sample was weighed into an extraction thimble and extraction was carried out in the Soxhlet apparatus with hexane for 4 hours. The flask containing oil with hexane was placed in a hot air oven to evaporate hexane, cooled in a desiccator and weighed. The difference in weight between the empty flask and

the residual oil gave the fat content, and it was given in terms of percent fat [AOAC, 2005].

2.9. Determination of Ash

Ash content was determined by burning the fresh and dried bee pollen samples in a furnace up to constant weight according to AOAC (2005). Bee pollen samples (2 g) were weighed into a previously heated, dried, cooled and weighed porcelain crucibles which were placed in a furnace at 600°C, and held until they turned to a completely grey color which took nearly for 4 hours. Crucibles were transferred to a desiccator, cooled and weighed. The difference in weight between the empty crucible and crucible with the ash residue was expressed as a percent ash content [AOAC, 2005].

2.10. Determination of Total Carbohydrates

Total carbohydrates are obtained as suggested by Estevinho et al. (2012) from Eq. (2.14):

$$\text{Total carbohydrates (g/100 g)} = 100 - (\text{ash} + \text{proteins} + \text{fats}) \quad (2.14)$$

2.11. Determination of Vitamin C

Vitamin C contents of fresh and dried bee pollen samples were determined based on AOAC (2005) modified by Jiang et al. (2014). Bee pollen samples (2 g) were weighed and smashed with 2% (w/w) oxalic acid. Smashed samples were transferred to a volumetric flask and diluted to 100 ml with 1% (w/w) oxalic acid and filtered through filter papers #1240 (14-18 µm) and #1244 (2-4 µm) (Filter-Lab, Filtros Anioia, S.A., Barcelona, Spain). Following filtration, 10 ml of the filtrate was titrated with 2,6-dichlorophenol-indophenol (DCIP) until the solution became pink, which can last for 15 s without color fading. Vitamin C contents of the samples are given by Eq. (2.15).

$$\text{Vitamin C (mg/100 g)} = \frac{(V - V_0)T}{m} \times 100 \quad (2.15)$$

where T is the amount that every milliliter of DCIP can titrate the standard ascorbic acid solution (0.02 mg/ml), V is the volume of DCIP used in this experiment, V_0 is the volume of DCIP for blank and m is the mass of sample in the solution [AOAC, 2005], [Jiang et al., 2014].

2.12. Determination of Solubility Index

Bee pollen samples (5 g) were put on a metal strainer and immersed into 100 ml of deionized water at 20°C for 10 s. The samples were taken and drained for 5 min on an inclined perforated surface, gently blotted with a paper towel and weighed. The solubility index is determined from Eq. (2.16) as given by Barajas et al. (2012):

$$SI = \frac{W_d - W_r}{W_o - W_d} \times 100 \quad (2.16)$$

where SI is the solubility index (dried pollen that can dissolve in water) of dried bee pollen, W_r is the weight of sample after immersion (g), W_d is the weight of sample after drying (g) and W_o is the weight of sample before drying (g).

2.13. Color Measurements

The instrumental color parameters of bee pollen samples were determined using a Konica Minolta CR-400 (Konica Minolta, Sensing Inc., Osaka, Japan) chroma meter equipped with a D_{65} illuminant and operating with CIE $L^*a^*b^*$ (L^* : 0 to 100, a^* : –green to +red and b^* : –blue to +yellow) color space. Calibration was performed with the white color calibration tile ($Y = 86.6$, $x = 0.3188$ and $y = 0.3364$) prior to the color measurements. Ten separate measurements were randomly taken from the surface of the bee pollen samples inside the quartz cell, and the results were given as the average of ten separate measurements. The color change of bee pollen samples as affected by drying air temperature was characterized by the total color difference (ΔE) given in Eq. (2.17) [Rayaguru et al., 2011], [Cao et al., 2016]:

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2} \quad (2.17)$$

$$\Delta L = L^* - L_0^*, \Delta a = a^* - a_0^* \text{ and } \Delta b = b^* - b_0^* \quad (2.18)$$

where L_0^* , a_0^* and b_0^* were color parameters of fresh bee pollen, and L^* , a^* and b^* were the color parameters of the dried bee pollen samples.

2.14. Scanning Electron Microscopy (SEM) Analysis

The structure and morphology of fresh and dried bee pollen samples were analyzed using a scanning electron microscope (Philips XL30 FEG, Oregon, USA) operating at a voltage of 5 kV. The bee pollen samples were mounted on SEM stubs, and sputter coated with a thin layer of gold using a sputter coater (Quorum SC7620, Quorum Technologies, East Sussex, UK).

2.15. Sensory Evaluation

Acceptance tests were carried out with a panel of thirty panelists aged between 20 and 40 years old to measure the degree of liking or disliking using a hedonic rating scale. A verbal hedonic scale of 9 points (9 = liked very much, 5 = neither like nor dislike, 1 = disliked very much) was used to quantify affective dimension of the consumer perception regarding the dried bee pollen samples [Meilgaard et al., 2016]. The panelists evaluated the sensory attributes including visual appearance, color, odor and overall acceptance in individually partitioned booths in an environmentally controlled room (23 ± 2 °C and $50 \pm 5\%$ RH) under white fluorescent light. Approximately 5 g of samples were placed into odor-free, disposable, white plastic plates which were labeled with five random digits and presented to the panelists in a randomized order [Meilgaard et al., 2016]. Each sample was presented to each panelist along with the appropriate questionnaire, one at a time, with a 3 min wait between the samples. Scores higher than 5 were considered as acceptable, indicating that a score below this value for any of the attributes evaluated was deemed that potential consumers have negative perception about the product.

2.16. Statistical Analysis

At least triplicate measurements were taken to increase precision or decrease uncertainty in the measurements and to better estimate the uncertainty in the measurements by checking how reproducible the measurements are. Results were reported as mean $\pm$ standard deviation (uncertainty). One-way analysis of variance (ANOVA) followed by post hoc analysis using Tukey's multiple range test with a significance level (α) of 0.05 was performed to determine statistical differences between the means of the results for the crude proteins, fats, ash, total carbohydrates, vitamin C, solubility index, color and sensory attributes for the bee pollen samples. The differences between the means are regarded as statistically significant if the p-value is less than or equal to 0.05. All statistical analyses were performed using Minitab 16.1 software (Minitab Inc., PA, USA).

3. RESULTS and DISCUSSION

3.1. Hot Air Drying

The results of hot air drying experiments were examined from different aspects and presented under three heading.

3.1.1. Drying Characteristics

Bee pollen samples having an initial moisture content of 0.278 ± 0.003 g water/g dry solid were dried to a final moisture content of 0.088 ± 0.002 g water/g dry solid. Drying curves representing the variation of moisture content and moisture ratio (MR) with the drying time at different temperatures are shown in Figures 3.1 and 3.2, respectively. The moisture content of bee pollens decreased continuously with the drying time. As expected, the MR decreased exponentially with the drying time. The drying time at 40°C was 210 min while the drying time was reduced to 80 min when the temperature was increased to 60°C. Drying air temperature had an important effect on the drying time. The drying time was shortened by 62% when the temperature was increased from 40 to 60°C. This is due to the fact that the rate of mass transfer was higher at higher temperatures. Barajas et al. (2012) investigated the effect of temperature on the drying of bee pollens harvested from two zones of Colombia using a tray dryer with hot air at 35 and 45°C without air recirculation. For the bee pollens harvested from two different zones of Colombia, drying times at 35°C changed between 210 and 297 min whereas drying times at 45°C ranged from 156 to 198 min. In this study, drying time at 45°C was 170 min which is similar to the results reported by Barajas et al. (2012) for the drying of bee pollens at 45°C.

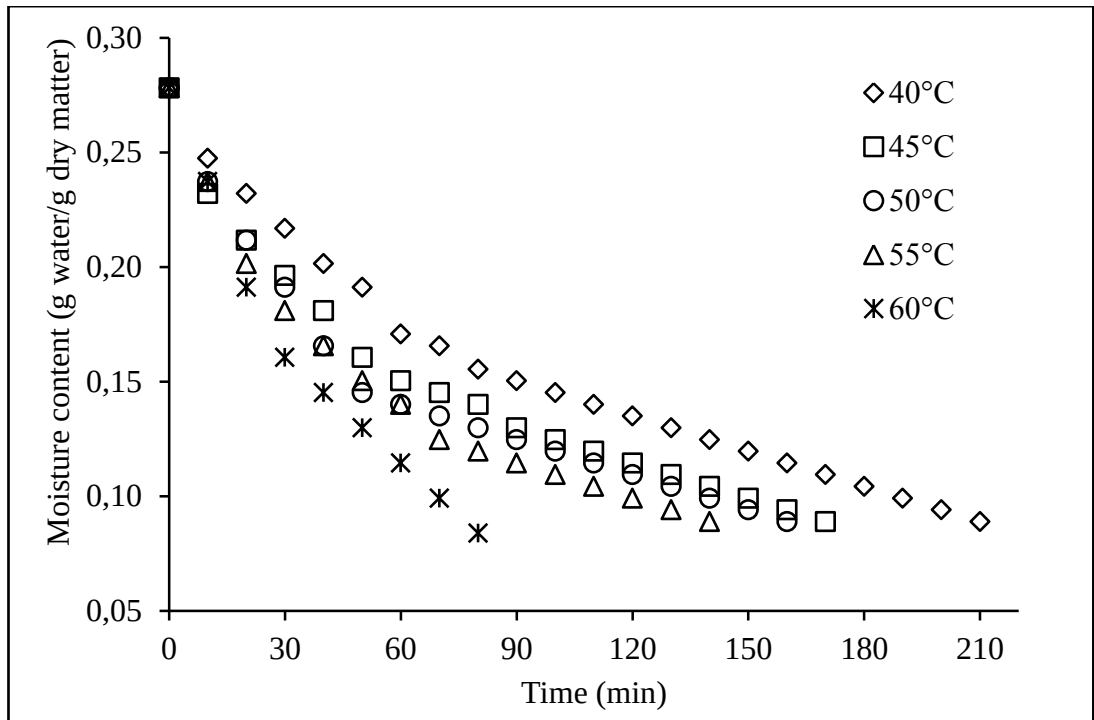


Figure 3.1: Variation of moisture content with drying time for hot air drying of bee pollen at different temperatures.

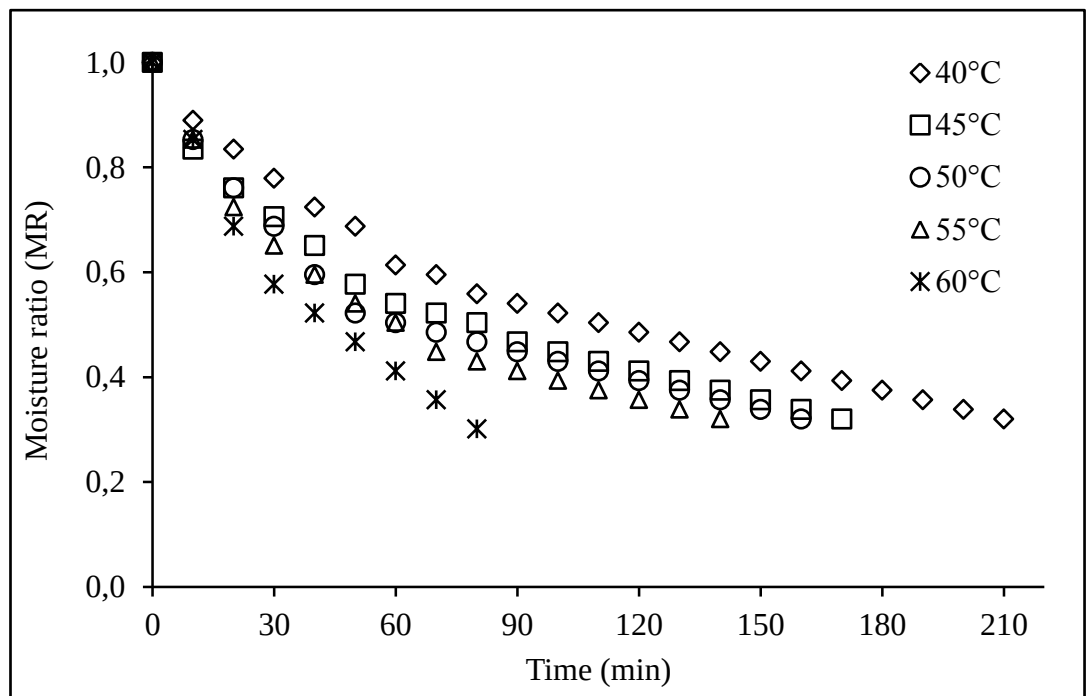


Figure 3.2: Effect of drying air temperature and drying time on the moisture ratio of bee pollen samples.

3.1.2. Effective Moisture Diffusivity and Activation Energy for Hot Air Drying

The D_{eff} values at different drying temperatures, calculated from Eq. (2.8), were found to be 1.38×10^{-10} , 1.57×10^{-10} , 1.97×10^{-10} , 2.24×10^{-10} and 4.00×10^{-10} m^2/s at 40, 45, 50, 55 and 60°C , respectively (Table 3.1). As the drying temperature was increased from 40 to 60°C , there was almost 3-fold increase in D_{eff} . This might be explained by the increased heating energy, which would increase the activity of the water molecules leading to higher moisture diffusivity when the samples were dried at higher temperatures. The D_{eff} values were within the range of 10^{-11} to 10^{-9} m^2/s for drying of food materials [Madamba et al., 1996].

Table 3.1: Effective moisture diffusivity values obtained for bee pollen samples dried at different drying temperatures.

Temperature ($^\circ\text{C}$)	Slope	D_{eff} (m^2/s)	R^2
40	0.0051	1.38×10^{-10}	0.9998
45	0.0058	1.57×10^{-10}	0.9993
50	0.0073	1.97×10^{-10}	0.9979
55	0.0083	2.24×10^{-10}	0.9981
60	0.0148	4.00×10^{-10}	0.9995

The E_a is the measure of the energy needed to initiate the moisture diffusion from the internal regions of the food material, and it also indicates the sensitivity of D_{eff} to drying temperature. The E_a was calculated by plotting $\ln(D_{\text{eff}})$ versus the reciprocal of the temperature $1/(T + 273.15)$ as shown in Figure 3.3. Results showed a linear relationship due to Arrhenius type dependence. The E_a was found to be 42.96 kJ/mol which lies within the general range of 12.7-110 kJ/mol given for food materials [Aghbashlo et al., 2008].

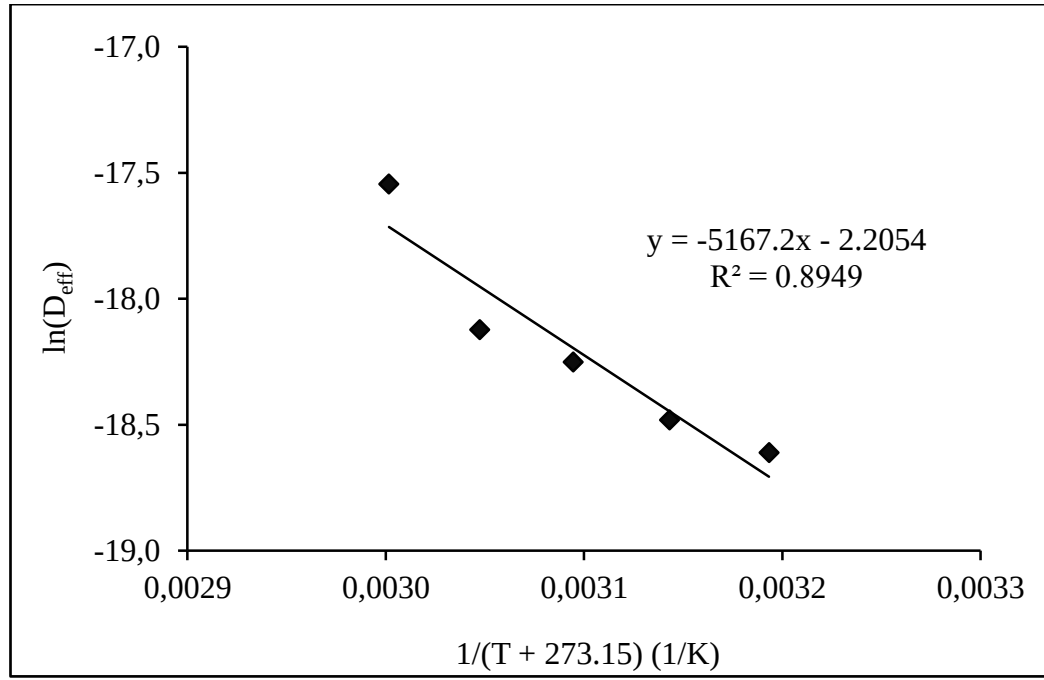


Figure 3.3: Arrhenius-type relationship between effective moisture diffusivity and reciprocal of absolute temperature.

The temperature dependence of the D_{eff} of the bee pollen during hot air drying was determined and is given in Eq. (3.1):

$$D_{\text{eff}} = 1.10 \times 10^{-1} \exp\left(-\frac{5167.2}{(T + 273.15)}\right) \quad (R^2 = 0.8949) \quad (3.1)$$

3.1.3. Effect of Hot Air Drying Temperature on Physicochemical Properties

The change in the physicochemical properties of bee pollen dried at different air temperatures is given in Table 3.2. The crude protein of fresh bee pollen was 30.36 ± 0.63 g/100 g while the crude protein content of the dried bee pollen samples varied from 12.43 ± 0.07 to 20.42 ± 0.31 g/100 g. Almeida-Muradian et al. (2005) reported protein content values of 21 g/100 g for the bee pollen pellets collected in the south region of Brazil, and Salamanca Grosso et al. (2008) found protein content of 17 g/100 g for the dried pollen from the Boyacá region of Colombia. These protein content values for the dried bee pollens were close to the protein content results of this study. The crude protein content decreased with increasing the drying air temperature. The loss in crude protein may be linked to the denaturation of

proteins as the air temperature increases. The crude protein contents of bee pollens dried at 40, 45, 50, 55 and 60°C were significantly different ($p \leq 0.05$) from that of the fresh bee pollen. The crude proteins of the bee pollens dried at 50, 55 and 60°C were not statistically different ($p \leq 0.05$).

The fat content of the bee pollens varied from 5.50 ± 0.04 to 7.22 ± 0.02 g/100 g. The fat content of the fresh bee pollen was statistically different ($p \leq 0.05$) than those of the dried bee pollen samples except the bee pollen sample dried at 40°C. Fat content values for the dried bee pollens in this study were similar to those reported by Almeida-Muradian et al. (2005) who found a fat content of 5-9 g/100 g for the dried bee pollen pellets collected in the south region of Brazil.

Ash content is an account of the inorganic matter found in bee pollen. The drying air temperature did not affect the ash content of the bee pollen. This result is consistent with the results of Barajas et al. (2012) who also found that ash content of the bee pollen was not affected by the drying air temperature. The ash contents of hot air dried bee pollen samples found in this study were similar to those reported by Almeida-Muradian et al. (2005), which ranged from 1.6-3.2 g/100 g and Barajas et al. (2012) which ranged from 1.69-3.19 g/100 g.

Carbohydrates constitute the biggest fraction of the total weight of the bee pollen [Villanueva et al., 2002]. Statistically significant differences ($p \leq 0.05$) were observed between the total carbohydrates of fresh and dried bee pollen samples. Total carbohydrates in dried bee pollens in this study ranged from 71.81 ± 0.31 to 78.70 ± 0.07 g/100 g while the carbohydrate content in dried Brazilian pollen samples was found to be 52.10 g/100 g [Carpes et al., 2009], and the carbohydrate content in Portuguese bee pollen samples changed between 60.82 and 70.76 g/100 g [Estevinho et al., 2012]. Our results were superior to the ones reported by Carpes et al. (2009), and Estevinho et al. (2012) for Brazilian and Portuguese bee pollen samples, respectively.

Vitamin C contents of dried bee pollen samples decreased with increasing the drying air temperature. There is a significant difference ($p \leq 0.05$) between the vitamin C contents of dried and fresh bee pollens. Among the dried bee pollen samples, the bee pollen dried at 40°C retained almost 76% of its vitamin C as compared with that of fresh one. As temperature increased from 40 to 60°C, the loss in vitamin C content of the bee pollen samples increased from 24% to 69% (approximately 2.9-fold). Vitamin C is a very thermosensitive compound, and an

increase in the drying air temperature and the drying time speeds up the oxidation of vitamin C, which results in loss in vitamin C content of the dried bee pollen. Our results for vitamin C were in good agreement with the results reported by Barajas et al. (2012) who found that vitamin C contents of two different dried Colombian bee pollens decreased progressively with increasing the drying air temperature. Campos et al. (2010) reported that after drying of bee pollen between 32 and 42°C, the vitamin C contents decreased by an average of 31% which is comparable with the decrease in the vitamin C content (24%) of hot air dried bee pollens at 40°C in this study.

Table 3.2: Physicochemical properties of fresh and hot air dried bee pollens¹.

	Fresh	40°C	45°C	50°C	55°C	60°C
Crude proteins (g/100 g)	30.36 ± 0.63 ^a	20.42 ± 0.31 ^b	15.56 ± 0.35 ^c	13.05 ± 0.11 ^d	12.43 ± 0.07 ^d	13.28 ± 0.27 ^d
Fats (g/100 g)	5.50 ± 0.04 ^a	5.60 ± 0.01 ^a	5.86 ± 0.03 ^b	6.09 ± 0.05 ^c	7.04 ± 0.07 ^d	7.22 ± 0.02 ^e
Ash (g/100 g)	2.18 ± 0.02 ^a	2.17 ± 0.01 ^a	2.15 ± 0.01 ^a	2.16 ± 0.02 ^a	2.15 ± 0.02 ^a	2.14 ± 0.01 ^a
Total carbohydrates (g/100 g)	61.96 ± 0.59 ^a	71.81 ± 0.31 ^b	76.16 ± 0.34 ^c	78.70 ± 0.07 ^e	78.38 ± 0.10 ^{de}	77.36 ± 0.29 ^{cd}
Vitamin C (mg/100 g)	38.48 ± 0.69 ^a	29.11 ± 0.13 ^b	24.94 ± 0.07 ^c	16.57 ± 0.32 ^d	13.99 ± 0.12 ^e	12.11 ± 0.19 ^e

¹Values are expressed in mean ± standard deviation, and values with the same letter within the same row are not statistically significant at p > 0.05.

3.1.4. Effect of Hot Air Drying on Solubility Index

The solubility index of hot air dried bee pollen samples after 50 s of dissolution was between 6.9 and 8.8 (Figure 3.4). These values revealed that the dried pollen had a high solubility index because the bee pollen samples had dissolution times around 50 s. Barajas et al. (2012) reported that after 50 s of dissolution, the solubility index values of two different Colombian bee pollens dried at 45°C were 3.5 and 5.5 which were lower than the solubility index value of bee pollen dried at 45°C in this study. This means that the dried bee pollen samples in this study are more easily soluble in water than the dried Colombian bee pollen samples.

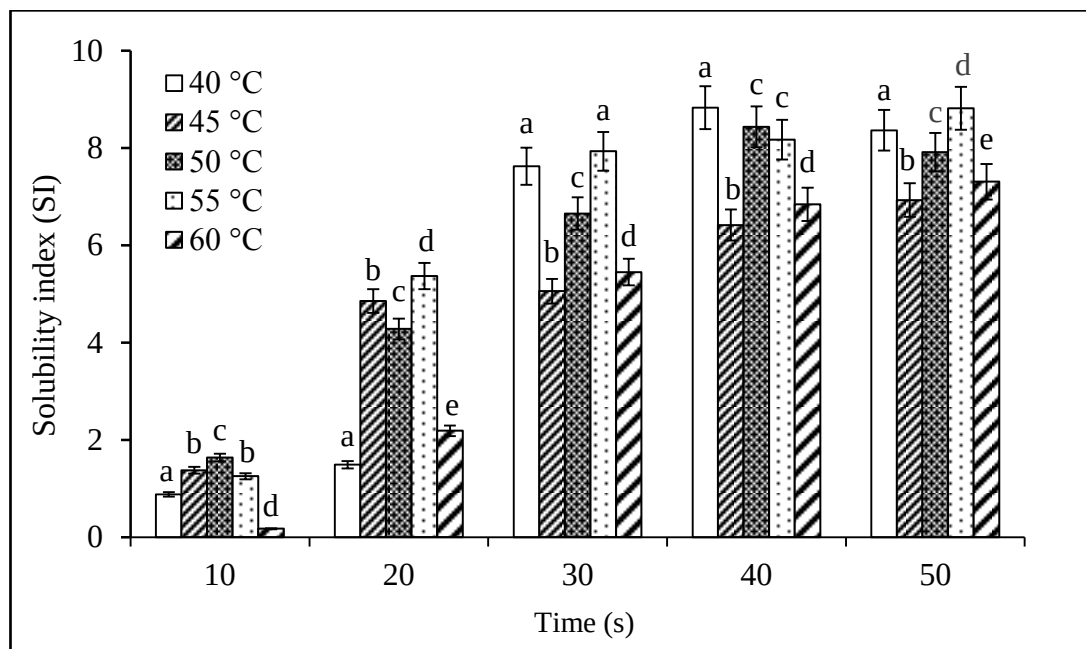


Figure 3.4: Solubility index of the bee pollen hot air dried at various temperatures. Different letters indicate significant differences ($p \leq 0.05$) among treatments.

3.1.5. Effect of Hot Air Drying on Color

All the dried bee pollen samples showed lower L^* and b^* values than the fresh bee pollen while a^* values of the dried bee pollen samples were slightly higher than those of the fresh bee pollen, but the difference was not statistically significant ($p > 0.05$) (Table 3.3). The parameter L^* indicates the darkness or lightness of the color and, in general, the dried bee pollen samples tend to have L^* values higher than 50. The L^* values of the bee pollen samples dried at 40 and 45°C did not differ

significantly ($p > 0.05$) from the L^* value of the fresh bee pollen. Among the dried bee pollen samples, the bee pollen dried at 40°C exhibited the highest L^* value while the bee pollen dried at 60°C showed the lowest L^* value. The decrease in b^* value is interpreted as a decrease in yellow color. The b^* values of the dried bee pollens decreased with increasing the drying temperature. Compared with the b^* value of the fresh bee pollen, there was no statistically significant difference ($p > 0.05$) between the b^* values of the bee pollen samples dried at 40 and 45°C.

The statistical analysis of ΔE values of the dried bee pollen samples indicated that ΔE was the lowest for the bee pollen dried at 40°C while the highest ΔE was attained for the bee pollen dried at 60°C in which higher drying temperatures led to larger ΔE values. As lower ΔE values are favorable, the color of the bee pollen dried at 40°C is the closest to the color of the fresh bee pollen.

Table 3.3: Color parameters of fresh and hot air dried bee pollen samples¹.

Temperature (°C)	L^*	a^*	b^*	ΔE
Fresh bee pollen	56.50 ± 0.84^a	10.78 ± 0.38^a	64.23 ± 2.22^a	—
40	55.23 ± 1.63^{ab}	10.86 ± 1.00^a	63.19 ± 1.84^{ab}	2.65 ± 1.42^a
45	55.00 ± 1.44^{ab}	11.32 ± 0.55^a	61.38 ± 2.25^{abc}	3.55 ± 2.84^a
50	53.07 ± 0.79^{bc}	11.33 ± 0.82^a	59.52 ± 1.26^{bc}	5.99 ± 2.16^{ab}
55	52.34 ± 1.42^c	11.52 ± 0.95^a	59.49 ± 2.07^{bc}	6.66 ± 3.30^{ab}
60	50.57 ± 0.47^c	11.82 ± 1.00^a	57.56 ± 0.77^c	9.19 ± 2.11^b

¹Values are expressed in mean $\pm$ standard deviation, and values with the same letter within the same column are not statistically significant at $p > 0.05$.

3.1.6. Morphological Structure of Hot Air Dried Bee Pollen

Fresh bee pollen grains varied in size from 28 to 35 μm and were bilaterally symmetrical and mostly had an elliptical shape with a longitudinal crevice (Figure 3.5a). The surface of the fresh bee pollen grains is mostly smooth as compared with those of the dried bee pollens. The morphological changes on the surface of bee pollen grains increased with increasing the drying temperature (Figures 3.5b-3.5f). This was due to the fact that more water evaporated as the drying temperature

increased, which resulted in increased morphological changes on the surface of the bee pollen grains.

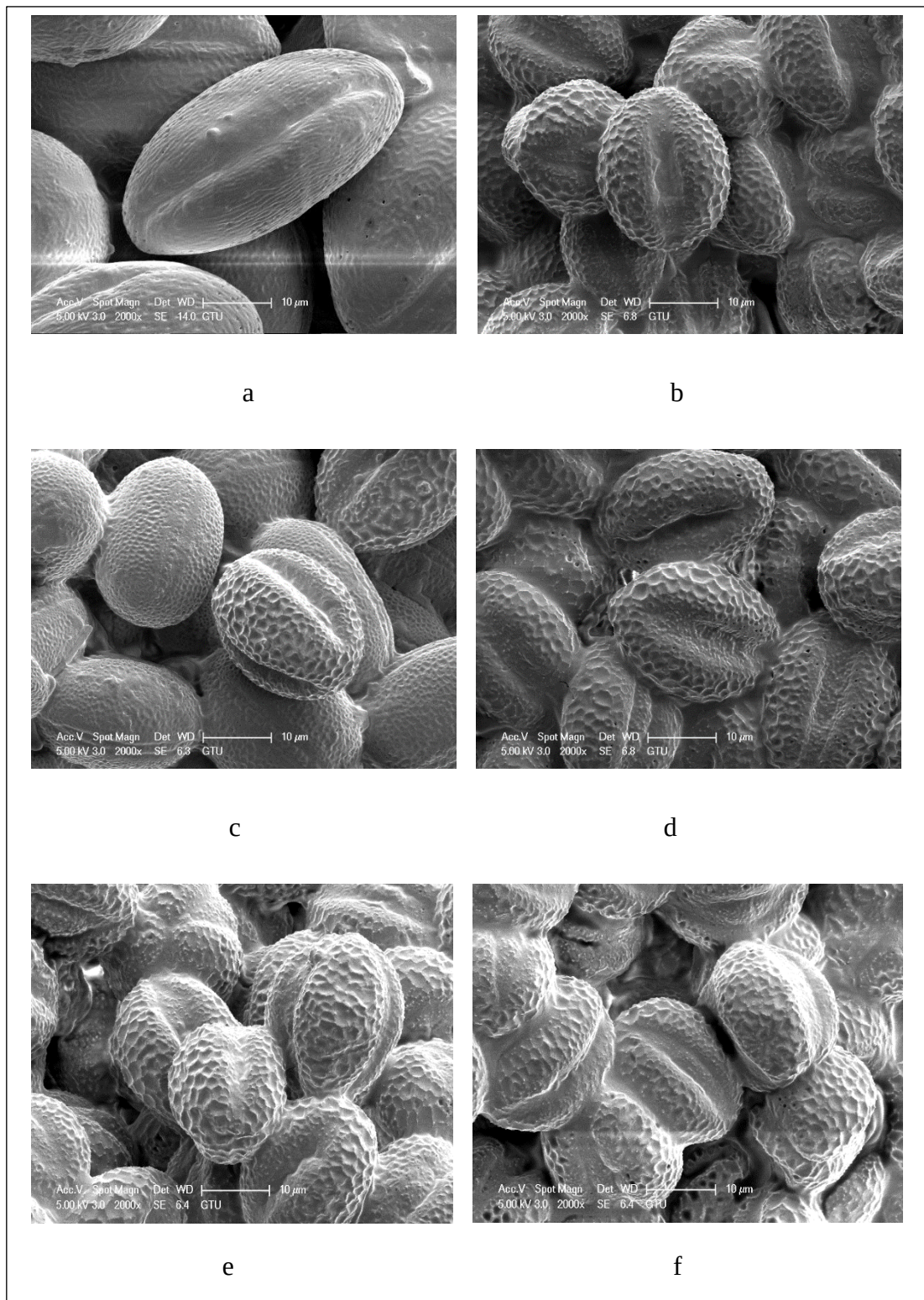


Figure 3.5: Scanning electron micrographs of (a) fresh, and hot air dried bee pollen samples at (b) 40°C, (c) 45°C, (d) 50°C, (e) 55°C and (f) 60°C. Scale bar is 10 µm. Magnification: 2000 ×.

3.1.7. Effect of Hot Air Drying on Sensory Attributes

Sensory attributes of a food product determine its acceptability by the consumers. Therefore, sensory attributes such as visual appearance, color, odor and overall acceptance of the bee pollen samples dried at different temperatures were scored by the panelists. Mean sensory scores related to sensory attributes are given in Table 3.4. The visual appearance, color, odor, and overall acceptance scores decreased with increasing the drying temperature. The bee pollen sample dried at 40°C took the highest sensory scores from the panelists while the bee pollen dried at 60°C took the lowest scores. A significant difference ($p \leq 0.05$) was observed between the sensory attributes of bee pollen dried at 40°C and those of dried at 60°C. On the other hand, there was no significant difference ($p > 0.05$) between the visual appearance, color, odor and overall acceptability scores of bee pollen dried at 40°C and those of the fresh bee pollen. The evaluation of the sensory attributes of the dried bee pollen samples showed that the bee pollen dried at 40°C retained its quality attributes better than the bee pollen samples dried at 45, 50, 55 and 60°C.

Table 3.4: Sensory attributes of fresh and hot air dried bee pollen samples¹.

Temperature (°C)	Visual appearance	Color	Odor	Overall acceptance
Fresh bee pollen	8.74 ± 0.15 ^a	8.71 ± 0.18 ^a	8.77 ± 0.12 ^a	8.72 ± 0.13 ^a
40	8.00 ± 0.39 ^{ab}	8.10 ± 0.37 ^{ab}	7.95 ± 0.27 ^{ab}	8.12 ± 0.28 ^{ab}
45	7.50 ± 1.10 ^{bc}	7.50 ± 0.71 ^{bc}	7.60 ± 0.62 ^b	7.58 ± 0.36 ^b
50	6.90 ± 0.94 ^c	6.85 ± 0.63 ^c	7.10 ± 1.22 ^{bc}	6.83 ± 0.62 ^c
55	5.80 ± 0.60 ^d	5.85 ± 0.55 ^d	6.50 ± 0.92 ^c	6.05 ± 0.45 ^d
60	4.50 ± 0.50 ^e	4.60 ± 1.26 ^e	4.90 ± 0.83 ^d	4.67 ± 0.66 ^e

¹Values are expressed in mean ± standard deviation, and values with the same letter within the same column are not statistically significant at $p > 0.05$.

3.2. Infrared Drying

3.2.1. Drying Characteristics

Bee pollen samples having an initial moisture content of 0.278 ± 0.003 g water/g dry solid were dried to a final moisture content of 0.081 ± 0.003 g water/g dry solid. Drying curves representing the variation of moisture content and the variation of moisture ratio (MR) with time at different infrared power levels are shown in Figures 3.6 and 3.7, respectively. The results showed that drying time decreased greatly when the infrared power increased. Drying of the bee pollen samples at the infrared power levels of 50, 62, 74 and 88 W to the final moisture content took 170, 130, 80 and 50 min, respectively. The drying time was shortened by 70.6% when the infrared power was increased from 50 W to 88 W. This is because the rate of mass transfer is higher at higher power levels. Similar results have also been observed for the drying of some agricultural products [Doymaz, 2015], [Olanipekun et al., 2015], [Cao et al., 2016].

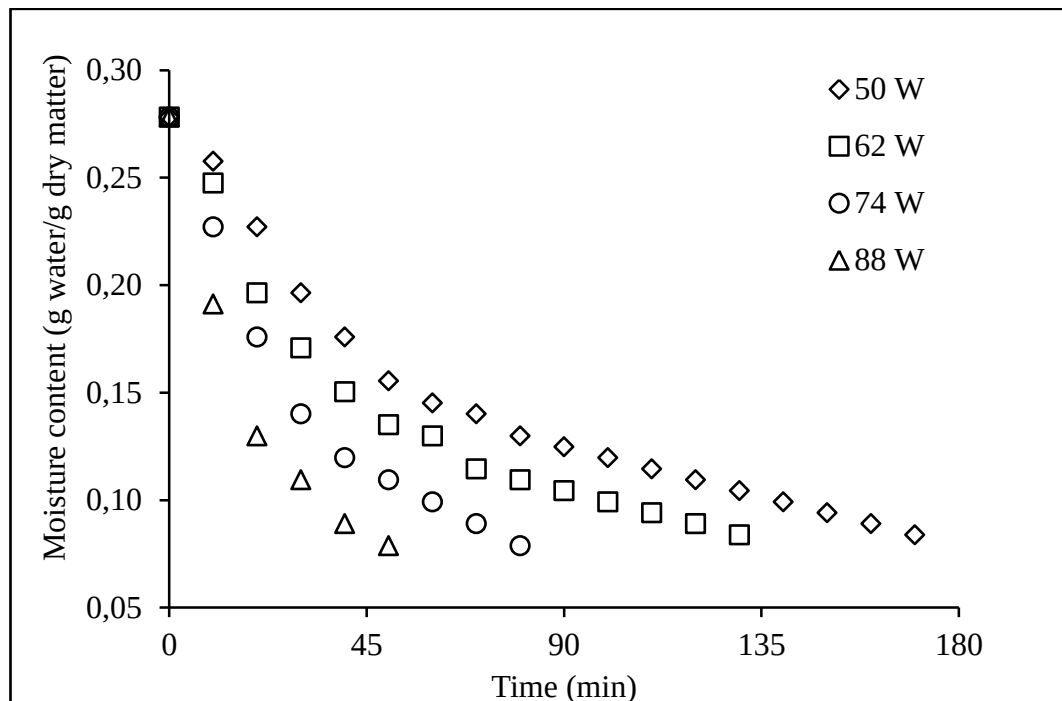


Figure 3.6: Variation of moisture content with time for the infrared drying of bee pollen at different infrared powers.

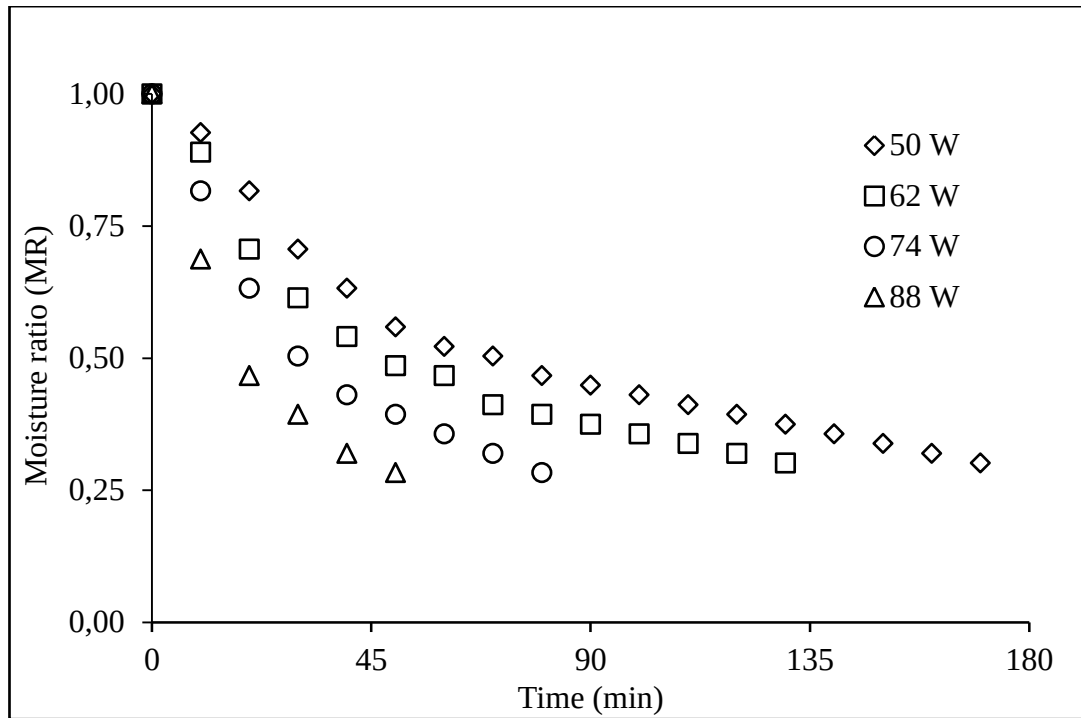


Figure 3.7: Effect of infrared power level and drying time on the moisture ratio of bee pollen samples.

3.2.2. Effective Moisture Diffusivity and Activation Energy for Infrared Drying

The D_{eff} values at different infrared powers were calculated from Eq. (2.8) and ranged from 1.92×10^{-10} to $6.81 \times 10^{-10} \text{ m}^2/\text{s}$ (Table 3.5). The D_{eff} increased as the infrared power increased. This might be explained by the increased heating energy, which would increase the activity of the water molecules leading to higher moisture diffusivity when the samples were dried at higher infrared power levels. The D_{eff} values were within the range of 10^{-11} to $10^{-9} \text{ m}^2/\text{s}$ for drying of food materials [Madamba et al., 1996].

Table 3.5: Effective moisture diffusivity values obtained for bee pollen samples dried at different infrared powers.

Infrared Power (W)	Slope	R ²	D _{eff} (m ² /s)
50	0.0071	0.9992	1.92×10^{-10}
62	0.0093	0.9993	2.51×10^{-10}
74	0.0160	0.9993	4.32×10^{-10}
88	0.0252	0.9999	6.81×10^{-10}

The E_a was calculated from Eq. (2.11) by plotting $\ln(D_{eff})$ versus m/P from which the slope of the line gives E_a and the intercept equals to $\ln(D_0)$ as given in Figure 3.8. Results showed a linear relationship due to Arrhenius type dependence.

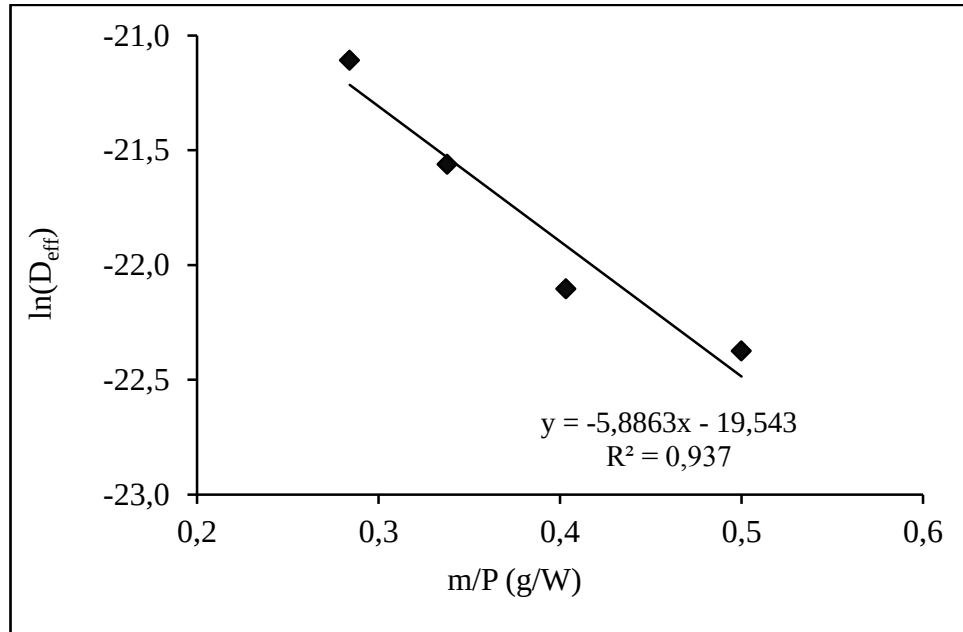


Figure 3.8: Arrhenius-type relationship between effective moisture diffusivity and infrared power.

The effect of sample weight/power level on D_{eff} of the bee pollen is determined as:

$$D_{eff} = 3.26 \times 10^{-9} \exp\left(-\frac{5.8863 \times m}{P}\right) \quad (R^2 = 0.9370) \quad (3.2)$$

The estimated values of D_0 and E_a were found to be 3.26×10^{-9} m²/s and 5.8863 W/g, respectively.

3.2.3. Effect of Infrared Drying Power Level on Physicochemical Properties

The change in the physicochemical properties of bee pollen dried at different infrared power levels is given in Table 3.6. The crude protein of fresh bee pollen was 30.36 ± 0.63 g/100 g while the crude protein content of the infrared dried bee pollen samples varied from 17.44 ± 0.50 to 24.38 ± 0.35 g/100 g. The crude protein content of bee pollens dried at 50 W, 62 W, 74 W and 88 W infrared powers were significantly different ($p \leq 0.05$) from that of the fresh bee pollen. The crude protein content decreased with increasing the infrared power level. The most significant decline in the amount of protein was observed at 88 W. The loss in crude protein may be linked to the denaturation of proteins as the infrared power level increases.

The fat content of fresh bee pollen was 5.50 ± 0.04 g/100 g. The fat content of bee pollens dried at different infrared powers varied from 5.99 ± 0.17 to 7.50 ± 0.14 g/100 g. The fat content of the fresh bee pollen was statistically different ($p \leq 0.05$) than those of the infrared dried bee pollen samples except the bee pollen sample infrared dried at 50 W. Fat content values for the infrared dried bee pollens in this study were similar to those reported by Almeida-Muradian et al. (2005) who found a fat content of 5-9 g/100 g for the dried bee pollen pellets collected in the south region of Brazil.

Ash content is an account of the inorganic matter found in bee pollen. The infrared power level did not affect the ash content of the bee pollen. This result is consistent with the results of Almeida-Muradian et al. (2005) and Barajas et al. (2012).

Carbohydrates constitute the biggest fraction of the total weight of the bee pollen [Villanueva et al., 2002]. Statistically significant differences ($p \leq 0.05$) were observed between the total carbohydrates of fresh and dried bee pollen samples. A high infrared power allows faster heating of the bee pollen samples and shortens the time needed to reach dryness, which may reduce metabolic losses of carbohydrates [Pelletier et al., 2010]. Total carbohydrates in infrared dried bee pollens in this study ranged from 67.45 ± 0.34 to 72.89 ± 0.60 g/100 g while the carbohydrate content in dried Brazilian pollen samples was found to be 52.10 g/100 g [Carpes et al., 2009] and the carbohydrate content in dried Portuguese bee pollen samples changed between 60.82 and 70.76 g/100 g [Estevinho et al., 2012].

Vitamin C contents of infrared dried bee pollen samples decreased with increasing the infrared power. There is a significant difference ($p \leq 0.05$) between the vitamin C contents of infrared dried and fresh bee pollens. Among the infrared dried bee pollen samples, the bee pollen infrared dried at 50 W retained almost 96% of its vitamin C as compared with that of fresh one. As the infrared power was increased from 50 W to 88 W the loss in vitamin C content of the bee pollen samples increased from 4% to 47%. Vitamin C is a very thermosensitive compound, and an increase in the infrared power speeds up the oxidation of vitamin C resulting in loss in vitamin C content of the dried bee pollen.

Table 3.6: Physicochemical composition of fresh and infrared dried bee pollens.¹

	Fresh	50 W	62 W	74 W	88 W
Crude proteins (g/100 g)	30.36 ± 0.63 ^a	24.38 ± 0.35 ^b	22.85 ± 0.18 ^b	20.36 ± 0.53 ^c	17.44 ± 0.50 ^d
Fats (g/100 g)	5.50 ± 0.04 ^a	5.99 ± 0.17 ^{ab}	6.07 ± 0.26 ^b	6.31 ± 0.03 ^b	7.50 ± 0.14 ^c
Ash (g/100 g)	2.18 ± 0.02 ^a	2.18 ± 0.01 ^a	2.16 ± 0.02 ^a	2.20 ± 0.01 ^a	2.17 ± 0.02 ^a
Total carbohydrates (g/100 g)	61.96 ± 0.59 ^a	67.45 ± 0.34 ^b	68.92 ± 0.24 ^b	71.13 ± 0.55 ^c	72.89 ± 0.60 ^d
Vitamin C (mg/100 g)	38.48 ± 0.69 ^a	37.05 ± 0.36 ^a	34.04 ± 0.17 ^b	27.02 ± 0.37 ^c	20.26 ± 0.41 ^d

¹Values are expressed as mean ± standard deviation; values with different letters within the same row are statistically significant at $p \leq 0.05$.

3.2.4. Effect of Infrared Drying on Solubility Index

The solubility index of infrared dried bee pollen samples after 50 s of dissolution was between 8 and 8.5 (Figure 3.9). These values revealed that the infrared dried bee pollen had a high solubility index because the bee pollen samples had dissolution times around 50 s. Barajas et al. (2012) reported that after 50 s of dissolution, the solubility index values of two different hot air dried Colombian bee pollens were 3.5 and 5.5, respectively, which were lower than the solubility index values of infrared dried bee pollen samples in this study. This means that the infrared dried bee pollen samples are more easily soluble in water than the hot air dried Colombian bee pollen samples.

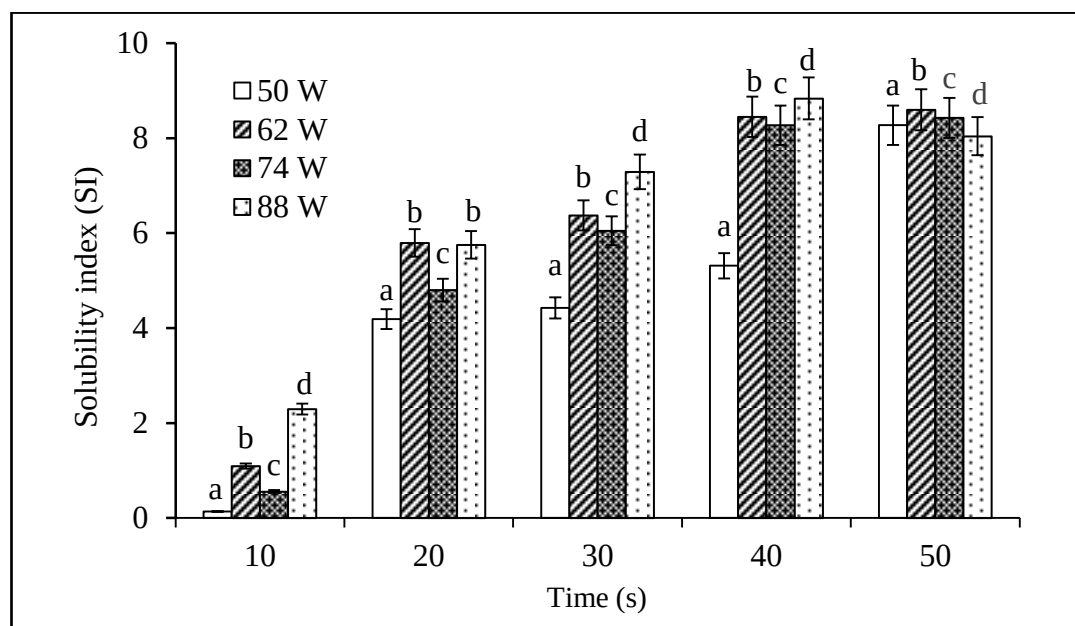


Figure 3.9: Solubility index of the bee pollen infrared dried at various infrared powers. Different letters indicate significant differences ($p \leq 0.05$) among treatments.

3.2.5. Effect of Infrared Drying on Color

Color values of the bee pollen samples dried at four different infrared powers are shown in Table 3.7. All the infrared dried bee pollen samples showed lower L^* and b^* values than the fresh bee pollen while a^* values of the infrared dried bee pollen samples were slightly higher than that of the fresh bee pollen, but the dif-

ference was not statistically significant ($p > 0.05$). The increase in a^* value and decrease in L^* value could result from non-enzymatic browning (Maillard reaction) during the drying process [Mireles-Arriaga et al., 2016], [Doymaz, 2018]. The parameter L^* indicates the darkness or lightness of the color, and L^* value decreased from 52.72 to 41.95 when the infrared power was increased from 50 W to 88 W, respectively. The decrease in b^* value is interpreted as a decrease in yellow color. The b^* values of the infrared dried bee pollens decreased with increasing the infrared power. Compared with the b^* value of the fresh bee pollen, there was no statistically significant difference ($p > 0.05$) between the b^* values of the bee pollen sample infrared dried at 50 W and 62 W. The analysis of ΔE values of the infrared dried bee pollen samples indicated that ΔE was the lowest for the bee pollen infrared dried at 50 W while the highest ΔE was attained for the bee pollen dried at 88 W in which higher infrared power levels led to larger ΔE values. As lower ΔE values are favorable, the color of the bee pollen infrared dried at 50 W is the closest to the color of the fresh bee pollen.

Table 3.7: Color parameters of fresh and infrared dried bee pollen samples¹.

Infrared Power (W)	L^*	a^*	b^*	ΔE
Fresh bee pollen	56.50 ± 0.84^a	10.78 ± 0.38^a	64.23 ± 2.22^a	—
50	52.72 ± 0.39^b	11.78 ± 0.78^{ab}	59.54 ± 0.48^b	6.27 ± 2.48^a
62	51.42 ± 1.10^b	12.77 ± 0.68^b	58.47 ± 0.75^b	8.04 ± 3.06^a
74	45.64 ± 1.60^c	12.44 ± 1.09^b	48.29 ± 1.78^c	18.6 ± 4.09^b
88	41.95 ± 1.43^d	12.75 ± 0.62^b	45.27 ± 2.76^d	24.03 ± 5.08^b

¹Values are expressed in mean $\pm$ standard deviation, and values with the same letter within the same column are not statistically significant at $p > 0.05$.

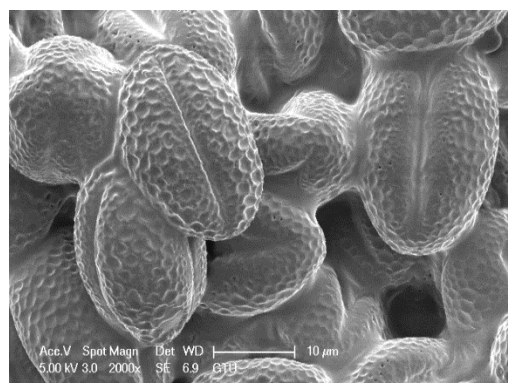
3.2.6. Morphological Structure of Infrared Dried Bee Pollen

Fresh bee pollen grains varied in size from 28 μm to 35 μm and were bilaterally symmetrical and mostly had an elliptical shape with a longitudinal crevice (Figure 3.10a). The surface of the fresh bee pollen grains is mostly smooth as

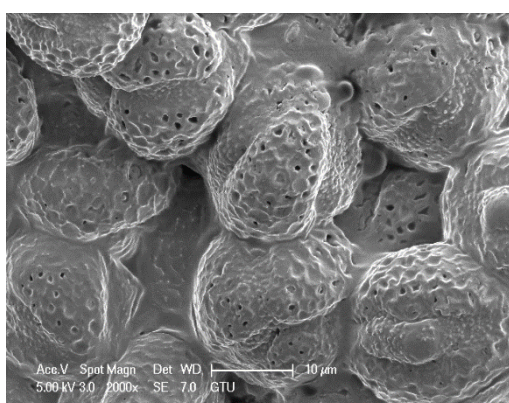
compared with those of the infrared dried bee pollens. The morphological changes on the surface of bee pollen grains increased with increasing the infrared power (Figures 3.10b-3.10e). This was due to the fact that more water was evaporated as the infrared power level increased that resulted in increased morphological changes on the surface of the bee pollen grains.



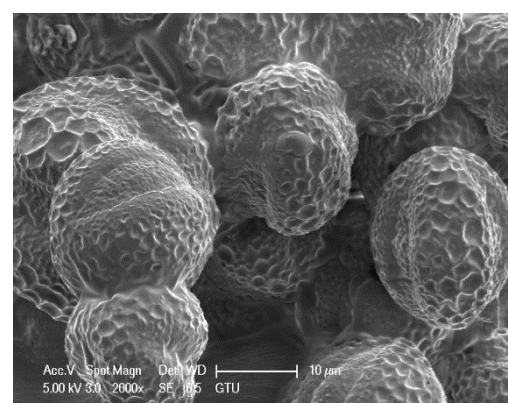
a



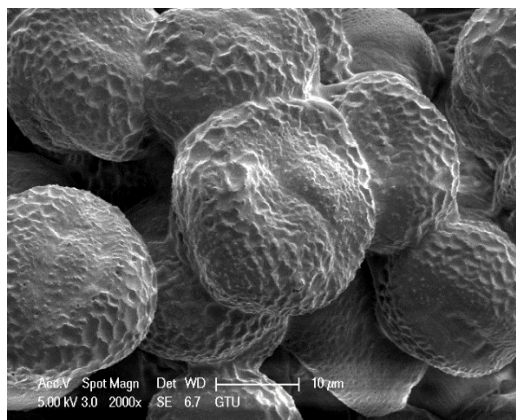
b



c



d



e

Figure 3.10: Scanning electron micrographs of (a) fresh and infrared dried bee pollens at different infrared powers (b) 50 W, (c) 62 W, (d) 74 W and (e) 88 W. Scale bar is 10 μ m. Magnification: 2000 $\times$.

3.2.7. Effect of Infrared Drying on Sensory Attributes

Sensory attributes of a food product determine its acceptability by the consumers. Therefore, sensory attributes such as visual appearance, color, odor and overall acceptance of the bee pollen samples dried at different infrared powers were scored by the panelists. Mean sensory scores related to sensory attributes are given in Table 3.8. The visual appearance, color, odor and overall acceptance scores decreased with increasing the infrared power level. The sensory attributes of bee pollen dried at 50, 62, 74 and 88 W were significantly different ($p \leq 0.05$) from that of the fresh bee pollen. The bee pollen sample dried at 50 W took the highest sensory scores from the panelists while the bee pollen dried at 88 W took the lowest scores. The reason might be less browning reaction that occurred at 50 W. In other words, the evaluation of the sensory attributes of the infrared dried bee pollen samples showed that the bee pollen infrared dried at 50 W retained its quality attributes better than the bee pollen samples infrared dried at 62, 74 and 88 W.

Table 3.8: Effect of infrared power levels on the sensory scores of the infrared dried bee pollen samples.¹

Infrared Power (W)	Visual appearance	Color	Odor	Overall acceptance
Fresh bee pollen	8.74 ± 0.15^a	8.71 ± 0.18^a	8.77 ± 0.12^a	8.72 ± 0.13^a
50	7.70 ± 0.60^b	7.50 ± 0.50^b	7.25 ± 0.75^b	7.43 ± 0.37^b
62	7.00 ± 0.63^{bc}	7.15 ± 0.81^{bc}	7.00 ± 0.77^b	7.05 ± 0.36^b
74	6.70 ± 0.78^c	6.45 ± 0.82^c	5.40 ± 0.49^c	6.18 ± 0.33^c
88	5.10 ± 0.54^d	5.25 ± 0.75^d	5.10 ± 0.54^c	5.15 ± 0.35^d

¹Values are expressed in mean $\pm$ standard deviation, and values with the same letter within the same column are not statistically significant at $p > 0.05$.

3.3. Microwave Drying

3.3.1. Drying Characteristics

Bee pollen samples having an initial moisture content of 0.278 ± 0.003 g water/g dry solid were dried to a final moisture content of 0.077 ± 0.015 g water/g

dry solid. Drying curves representing the variation of moisture content and the variation of moisture ratio (MR) with time at different microwave powers are shown in Figures 3.11 and 3.12, respectively. Results revealed that the drying time of bee pollen samples was reduced as the microwave output power was increased from 180 to 900 W. Drying of the bee pollen samples at the microwave power levels of 180, 360, 540, 720 and 900 W to the final moisture content took 2400, 480, 300, 210 and 150 seconds, respectively. The drying time was shortened by 94% when the microwave power was increased from 180 to 900 W. Increasing the microwave power decreased the drying time for the bee pollen samples. Similar results were also observed for the microwave drying of apple pomace [Wang et al., 2007], tomato pomace [Al-Harashseh et al., 2009] and *Gundelia tournefortii* [Evin, 2012] where increasing the microwave power reduced the time for microwave drying. This is because the rate of mass transfer is higher at higher microwave power levels.

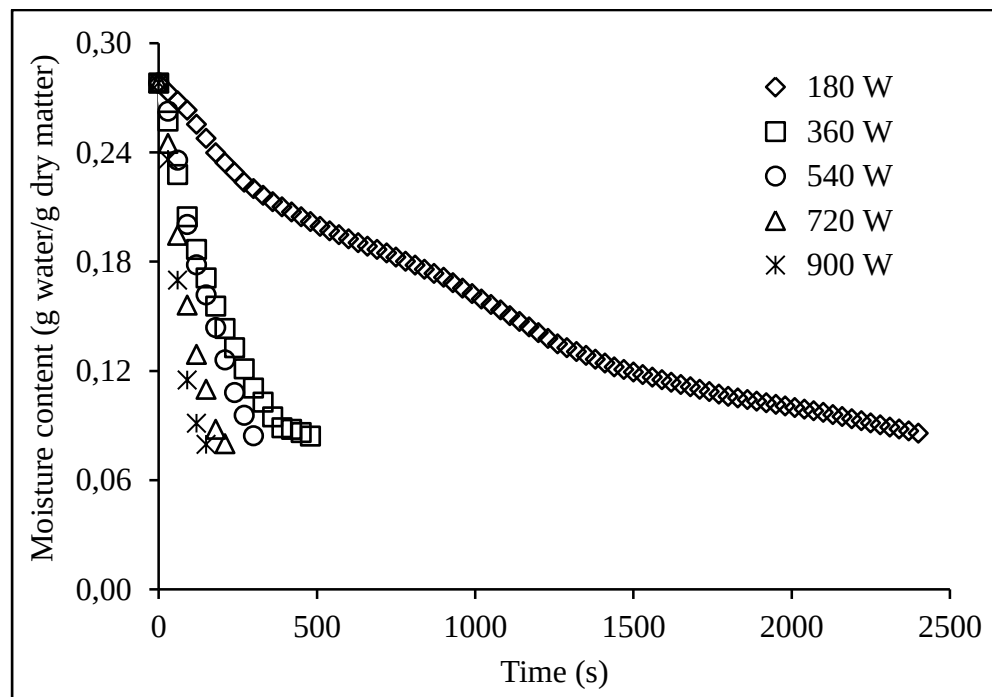


Figure 3.11: Variation of moisture content with time for the microwave drying of bee pollen at different microwave output powers.

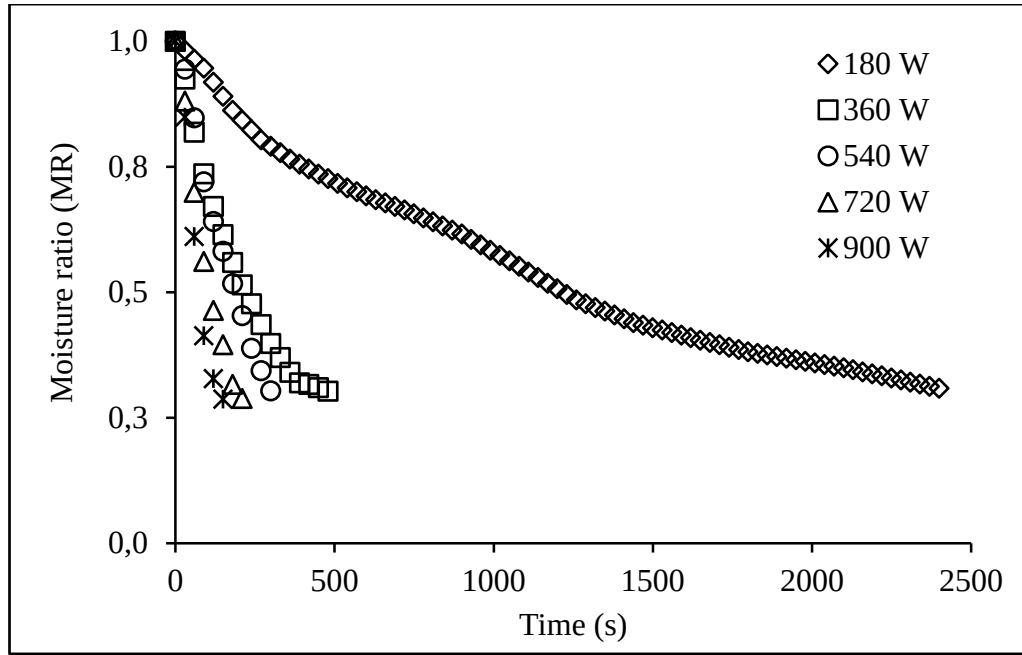


Figure 3.12: Effect of microwave power and drying time on the moisture ratio of bee pollen samples.

3.3.2. Effective Moisture Diffusivity and Activation Energy for Microwave Drying

To calculate effective moisture diffusivity values, the natural logarithm of the moisture ratio values was plotted against drying time based on the experimental data obtained for the microwave power outputs of 180-900 W as shown in Figure 3.13. D_{eff} values were calculated using Eq. (2.8) from the slope of each line. D_{eff} values at different microwave powers with the corresponding coefficient of determination (R^2) values are presented in Table 3.9. In this study, the range of D_{eff} values for the bee pollen samples varied from 0.04×10^{-8} to $1.14 \times 10^{-8} \text{ m}^2/\text{s}$. These values were within the general range of 10^{-12} to $10^{-6} \text{ m}^2/\text{s}$ for food materials [Panagiotou et al., 2004].

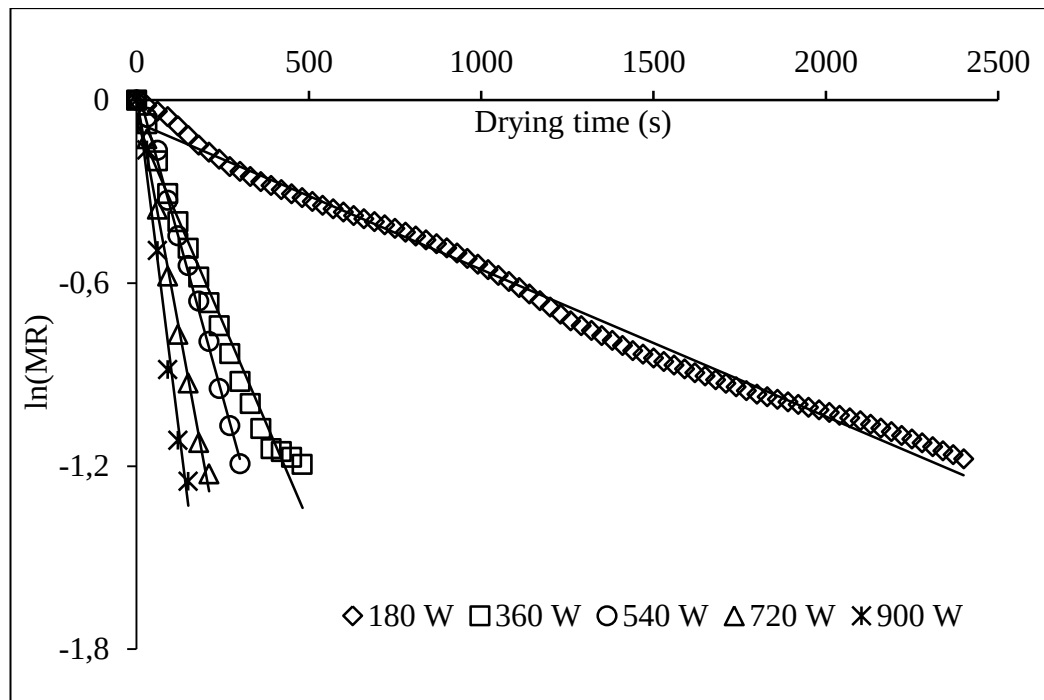


Figure 3.13. Variation of $\ln(MR)$ with drying time at different microwave powers.

Table 3.9: Effective moisture diffusivity values obtained for bee pollen samples microwave dried at different microwave powers.

Microwave Power (W)	Slope	D_{eff} (m^2/s)	R^2
180	0.0004	0.04×10^{-8}	0.9999
360	0.0029	0.26×10^{-8}	0.9994
540	0.0043	0.39×10^{-8}	0.9995
720	0.0068	0.62×10^{-8}	0.9994
900	0.0125	1.14×10^{-8}	0.9991

The natural logarithm of D_{eff} values was plotted against sample weight/microwave power values (Figure 3.14). The slope of the line gave E_a value which is the measure of the energy needed to initiate the moisture diffusion from the internal regions of the food material, and also indicates the sensitivity of D_{eff} to microwave power. Results showed a linear relationship due to Arrhenius type dependence.

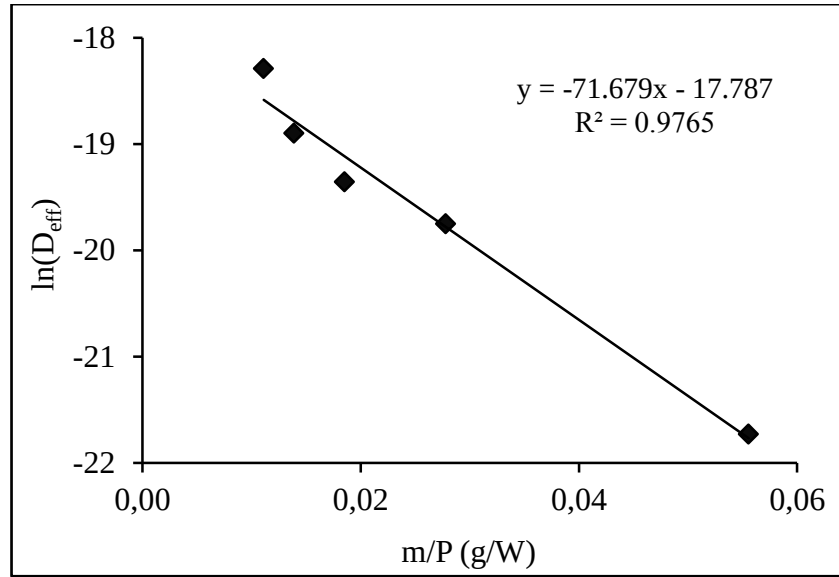


Figure 3.14. Arrhenius-type relationship between effective moisture diffusivity and microwave power.

The temperature dependence of the D_{eff} of the bee pollen during microwave drying is given in Eq. (3.3):

$$D_{\text{eff}} = 1.88 \times 10^{-8} \exp\left(-\frac{71.679 \times m}{P}\right) \quad (R^2 = 0.9765) \quad (3.3)$$

The estimated values of D_0 and E_a were $1.88 \times 10^{-8} \text{ m}^2/\text{s}$ and 71.679 W/g , respectively.

3.3.3. Effect of Microwave Drying Power Level on Physicochemical Properties

Table 3.10 shows the physicochemical composition of bee pollen samples subjected to different microwave drying powers between 180 and 900 W. Carbohydrates constituted the major component in fresh bee pollen. Total carbohydrate contents of fresh bee pollens with different botanical origin harvested in India ranged from 42.33 to 46.16 g/100 g [Thakur and Nanda, 2018]. The total carbohydrate content of fresh bee pollen ($61.96 \pm 0.59 \text{ g/100 g}$) in this work was within the range reported for the carbohydrates in different fresh bee pollens harvested in China (59.43-75.65 g/100 g) by Yang et al. (2013). Statistically significant differences ($p \leq 0.05$) were observed between the total carbohydrates of fresh and dried bee pollen samples. A high microwave power level allows faster

heating of the bee pollen samples and shortens the time needed to reach dryness, which may reduce metabolic losses of carbohydrates [Pelletier et al., 2010]. Total carbohydrates for microwave dried bee pollen samples in this study varied from 69.50 ± 0.16 to 71.21 ± 0.06 g/100 g. These results were close to total carbohydrate contents (60.82-70.76 g/100 g) of dried Portuguese bee pollen reported by Estevinho et al. (2012). As compared with the carbohydrate content of conventionally dried bee pollen, the bee pollen microwave dried at 180 W had almost the same amount of carbohydrates as the bee pollen hot air dried at 40°C [Isik et al., 2019a].

The second major component in fresh bee pollen after carbohydrates was proteins. The protein content of fresh bee pollen was found to be 30.36 ± 0.63 g/100 g which was higher than the protein contents of fresh bee pollens harvested in India (19.63-25.39 g/100 g) [Thakur and Nanda, 2018] and China (14.26-28.95 g/100 g) [Yang et al., 2013], respectively. Therefore, the fresh bee pollen in this study is said to be more nutritious than the bee pollens harvested in India and China [Thakur and Nanda, 2020a]. The crude protein content of microwave dried bee pollen samples varied from 16.37 ± 0.51 to 22.78 ± 0.26 g/100 g. The loss in crude protein may be linked to the denaturation of proteins as the microwave power level increases. The crude protein content of bee pollens dried at all microwave powers were significantly different ($p \leq 0.05$) from that of the fresh bee pollen. However, there was no statistically significant difference ($p > 0.05$) between the crude proteins of bee pollens dried at 180 and 360 W, and also 540 and 720 W. The most significant loss in the crude protein was observed in the bee pollens dried at 900 W. Isik et al. (2019a) determined that crude proteins in hot air drying of bee pollen decreased by 57% while the loss in crude proteins during microwave drying at the maximum power level of 900 W was 46%.

The fat content of the fresh bee pollen studied (5.50 ± 0.04 g/100 g) was within the range of fat contents of Chinese fresh bee pollens (0.66-10.79 g/100 g) reported by Yang et al. (2013), but the value was lower than the fat contents of Indian fresh bee pollens (7.13-12.38 g/100 g) found by Thakur and Nanda (2018). The fat content of microwave dried bee pollen samples changed between 5.57 ± 0.35 and 10.28 ± 0.46 g/100 g. Fat contents of bee pollens dried at 540 W, 720 W and 900 W microwave powers were significantly different ($p \leq 0.05$) from that of the fresh bee pollen. Our results are in good agreement with the results reported by Almeida-Muradian et al. (2005), who found a fat content of 5-9 g/100 g for oven dried bee

pollens collected in the south region of Brazil. Isik et al. (2019a) also reported that fat contents of hot air dried bee pollen changed between 5.60 and 7.22 g/100 g.

Microwave power levels did not significantly ($p > 0.05$) affect ash contents of microwave dried and fresh bee pollen samples. As compared with the ash content results of Indian fresh bee pollens (2.27-3.46 g/100 g) [Thakur and Nanda, 2018] and Chinese fresh bee pollens (1.70-5.01 g/100 g) [Yang et al., 2013], the ash content of fresh bee pollen in this study was 2.18 ± 0.02 g/100 g comparable with the ash contents reported for Indian and Chinese fresh bee pollens. Ash contents of microwave dried bee pollen samples did not change significantly ($p > 0.05$), and ranged from 2.14 to 2.18 g/100 g comparable with the ash contents of hot air dried bee pollen (Isik et al., 2019a). These results were within the range of ash contents (1.69-3.19 g/100 g) reported by Almeida-Muradian et al. (2005) and Barajas et al. (2012) for Brazilian and Colombian dried bee pollens, respectively.

Significant differences ($p \leq 0.05$) were observed between vitamin C contents of fresh and microwave dried bee pollen samples. Vitamin C contents of the microwave dried bee pollen samples decreased with increasing microwave power level. As the microwave power level was increased from 180 to 900 W, the loss of vitamin C for microwave dried bee pollen samples increased almost 5.1-fold. Among the microwave power levels used in this study, the lowest loss in vitamin C (14.5%) took place in the bee pollens microwave dried at 180 W. These results were in agreement with the results of the studies conducted by Dadali and Ozbek (2009), and Khraisheh et al. (2004), who found that the total vitamin C decreased progressively with the absorbed microwave power level. Isik et al. (2019a) used hot air between 40 and 60°C to dry bee pollen and found that, as the hot air drying temperature increased from 40 to 60°C, the loss in vitamin C content of the bee pollen samples increased from 24% to 69%. Infrared drying was also used in drying of bee pollen by Isik et al. (2019b) and, as the infrared power was increased from 50 to 88 W, the loss in vitamin C content of the bee pollen samples increased from 4% to 47%. Compared with the vitamin C loss in hot air and infrared drying, the highest loss in vitamin C (74%) was observed in the bee pollens dried at 900 W microwave power.

Table 3.10. Physicochemical composition of fresh and microwave dried bee pollen¹.

	Fresh	180 W	360 W	540 W	720 W	900 W
Crude proteins (g/100 g)	30.36 ± 0.63 ^a	22.78 ± 0.26 ^b	21.68 ± 0.36 ^b	19.71 ± 0.11 ^c	18.55 ± 0.52 ^c	16.37 ± 0.51 ^d
Fats (g/100 g)	5.50 ± 0.04 ^a	5.57 ± 0.35 ^a	6.13 ± 0.08 ^a	7.56 ± 0.17 ^b	8.63 ± 0.25 ^c	10.28 ± 0.46 ^d
Ash (g/100 g)	2.18 ± 0.02 ^a	2.15 ± 0.01 ^a	2.15 ± 0.01 ^a	2.18 ± 0.01 ^a	2.16 ± 0.02 ^a	2.14 ± 0.01 ^a
Total carbohydrates (g/100 g)	61.96 ± 0.59 ^a	69.50 ± 0.16 ^b	70.04 ± 0.33 ^{bc}	70.55 ± 0.27 ^{bc}	70.66 ± 0.72 ^{bc}	71.21 ± 0.06 ^c
Vitamin C (mg/100 g)	38.48 ± 0.69 ^a	32.90 ± 1.35 ^b	26.17 ± 1.35 ^c	18.58 ± 0.42 ^d	15.80 ± 0.73 ^d	9.93 ± 1.25 ^e

¹Values are expressed as mean ± standard deviation, and values with different letters (a-e) within the same row are statistically significant at $p \leq 0.05$.

3.3.4. Effect of Microwave Drying on Solubility Index

The solubility index of microwave dried bee pollen samples after 50 s of dissolution was between 4 and 6.5 (Figure 3.15). These values revealed that the microwave dried bee pollen had a high solubility index because the bee pollen samples had dissolution times around 50 s. Barajas et al. (2012) reported that after 50 s of dissolution, the solubility index values of two different hot air dried Colombian bee pollens were 3.5 and 5.5, respectively, which were close to the solubility index values of microwave dried bee pollen samples in this study. This means that the microwave dried bee pollen samples have almost same solubility in water with the hot air dried Colombian bee pollen samples.

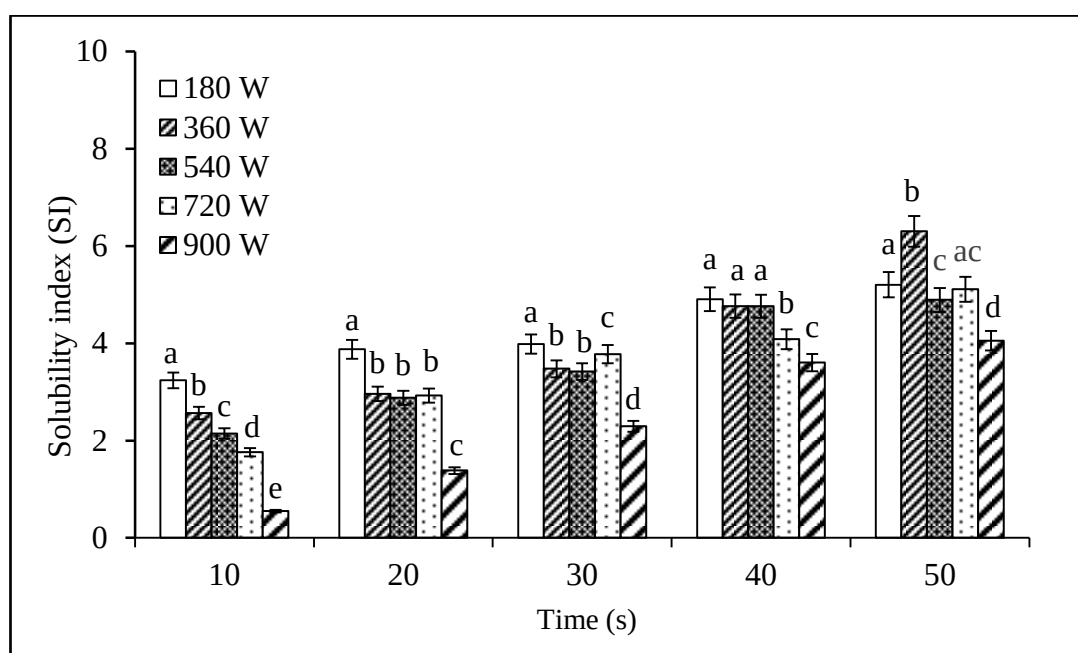


Figure 3.15: Solubility index of bee pollen microwave dried at various microwave power levels. Different letters indicate significant differences ($p \leq 0.05$) among treatments.

3.3.5. Effect of Microwave Drying on Color

Color values of the fresh and dried bee pollen samples are shown in Table 3.11 as a function of microwave power levels. All the microwave dried bee pollen samples showed lower L^* and b^* values than the fresh bee pollen, while a^* values of the

microwave dried bee pollen samples were higher than that of the fresh bee pollen. The increase in a^* value and decrease in L^* value could result from non-enzymatic browning (Maillard reaction) during the drying process [Doymaz et al., 2015], [Mireles-Arriaga et al., 2016], [Doymaz, 2018]. A significant difference ($p \leq 0.05$) was observed between the L^* , a^* and b^* values of the fresh and microwave dried bee pollens, except the a^* values at 180 and 360 W. The L^* value indicates the darkness or lightness, and the L^* value decreased from 42.83 to 30.38 when the microwave power was increased from 180 to 900 W, respectively. The b^* values of the microwave dried bee pollens decreased with increasing microwave power. The decrease in the b^* value is interpreted as a decrease in yellow color. Compared with the b^* value of the fresh bee pollen, a significant difference ($p \leq 0.05$) was observed between the b^* values of the microwave dried bee pollen samples.

The analysis of ΔE values of the microwave dried bee pollen samples indicated that ΔE was the lowest for the bee pollen microwave dried at 180 W, while the highest ΔE was attained for the bee pollen dried at 900 W, in which higher microwave power levels led to larger ΔE values. As lower ΔE values are favorable, the color of the bee pollen microwave dried at 180 W is the closest to the color of the fresh bee pollen. As compared with the hot air drying of bee pollen grains (Isik et al., 2019a), microwave drying changed the color parameters of bee pollen more than hot air drying.

Table 3.11: Color parameters of fresh and microwave dried bee pollen samples¹.

Microwave Power (W)	L^*	a^*	b^*	ΔE
Fresh bee pollen	56.50 ± 0.84^a	10.78 ± 0.38^a	64.23 ± 2.22^a	—
180	40.63 ± 4.58^b	13.22 ± 0.80^b	52.85 ± 4.62^{ab}	20.04 ± 5.00^a
360	36.26 ± 3.57^{bc}	11.13 ± 0.99^a	50.91 ± 7.17^b	24.94 ± 5.62^{ab}
540	35.59 ± 7.68^{bc}	10.95 ± 1.44^a	51.49 ± 5.45^b	25.85 ± 5.42^{ab}
720	32.75 ± 4.33^{bc}	9.47 ± 1.32^a	47.80 ± 5.89^b	29.08 ± 5.97^{ab}
900	30.58 ± 1.77^c	9.73 ± 0.86^a	46.66 ± 7.77^b	32.13 ± 4.75^b

¹Values are expressed in mean $\pm$ standard deviation, and values with the same letter within the same column are not statistically significant at $p > 0.05$.

3.3.6. Morphological Structure of Microwave Dried Bee Pollen

Scanning electron microscopy provides qualitative information regarding surface structures of foods and helps improve the understanding of drying mechanisms in relation to the changes in the food structure [Dhalsamant et al., 2018]. Fresh bee pollen grains were bilaterally symmetrical and mostly had an elliptical shape with a longitudinal crevice (Figure 3.16a). The surface of the fresh bee pollen grains is mostly smooth, as compared with those of the microwave dried bee pollens. Microwave power caused morphological changes in the size and surface of the bee pollen grains (Figures 3.16b-3.16f). Shrinkage was the predominant change in the microwave dried bee pollens. As the water evaporates, solid sugar bridges are formed and remain intact at the interface of the microwave dried bee pollen grains that hold the aggregates together more tightly. Compared with the hot air drying [Isik et al., 2019a] and infrared drying of bee pollen grains [Isik et al., 2019b], the effect of microwave drying on the morphological properties of bee pollens was more drastic.

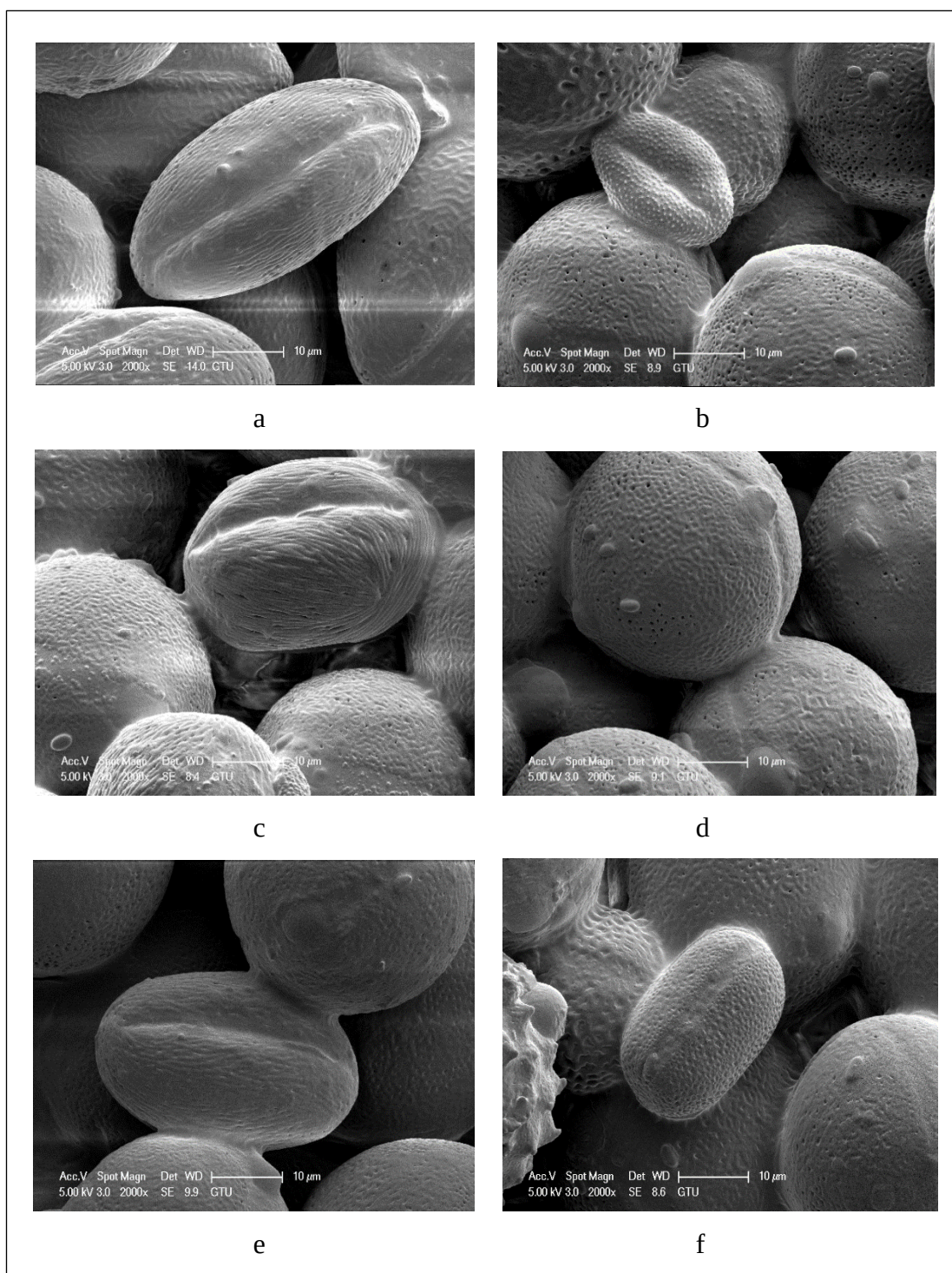


Figure 3.16: Scanning electron micrographs of (a) fresh and microwave dried bee pollens at different microwave power levels (b) 180 W, (c) 360 W, (d) 540 W, (e) 720 W and (f) 900 W. Scale bar is 10 µm. Magnification: 2000 ×.

3.3.7. Effect of Microwave Drying on Sensory Attributes

Visual appearances of dried foods play a very important role at the time of their sales. Therefore, analysis of some quality parameters such as appearance, color, texture, odor and taste can be used in the maintenance of product quality [Fernandes et al., 2011]. In this study, visual appearance, color, odor and overall acceptance of the bee pollen samples dried at different microwave power levels were evaluated by the panelists. Mean sensory scores are given in Table 3.12, which showed that significant differences ($p \leq 0.05$) exist among the sensory scores of microwave dried and fresh bee pollens. Compared with the sensory attributes of the fresh bee pollen, a significant difference ($p \leq 0.05$) was observed between the sensory attributes of the microwave dried bee pollen samples. The visual appearance, color, odor and overall acceptance scores decreased with increasing microwave power level. The bee pollen sample microwave dried at 180 W received the highest sensory scores from the panelists while the bee pollen microwave dried at 900 W had the lowest scores. The evaluation of the sensory attributes of the microwave dried bee pollen samples showed that the bee pollen microwave dried at 180 W retained its quality attributes better than the bee pollen samples microwave dried at 360, 540, 720 and 900 W.

Table 3.12: Effect of microwave power levels on the sensory scores of the microwave dried bee pollen samples.¹

Microwave Power (W)	Visual appearance	Color	Odor	Overall acceptance
Fresh bee pollen	8.74 ± 0.15^a	8.71 ± 0.18^a	8.77 ± 0.12^a	8.72 ± 0.13^a
180	6.60 ± 1.02^b	7.10 ± 0.81^b	6.35 ± 0.55^b	6.65 ± 0.47^b
360	6.00 ± 0.63^{ab}	6.90 ± 0.89^b	6.15 ± 0.39^b	6.48 ± 0.35^b
540	5.70 ± 0.46^b	6.45 ± 0.82^b	5.40 ± 0.49^c	5.85 ± 0.39^c
720	4.70 ± 0.46^c	5.25 ± 0.75^c	4.60 ± 0.49^d	4.85 ± 0.37^d
900	4.20 ± 0.40^c	2.20 ± 0.24^d	3.70 ± 0.46^e	3.03 ± 0.23^e

¹Values are expressed as mean $\pm$ standard deviation, and values with different letters (a-e) within the same column are statistically significant at $p \leq 0.05$.

3.4. Freeze Drying

The results of freeze drying experiments were examined from different aspects and presented under 3 headings.

3.4.1. Drying Characteristics

Bee pollen samples having an initial moisture content of 0.278 ± 0.003 g water/g dry solid were freeze dried to a final moisture content of 0.088 ± 0.002 g water/g dry solid for 15 hours. The variation of moisture content and the variation of moisture ratio (MR) with time for the freeze drying of bee pollen are shown in Figures 3.17 and 3.18, respectively. A linear relationship was observed between the moisture content and drying time. In other words, moisture content and MR decreased linearly with the drying time.

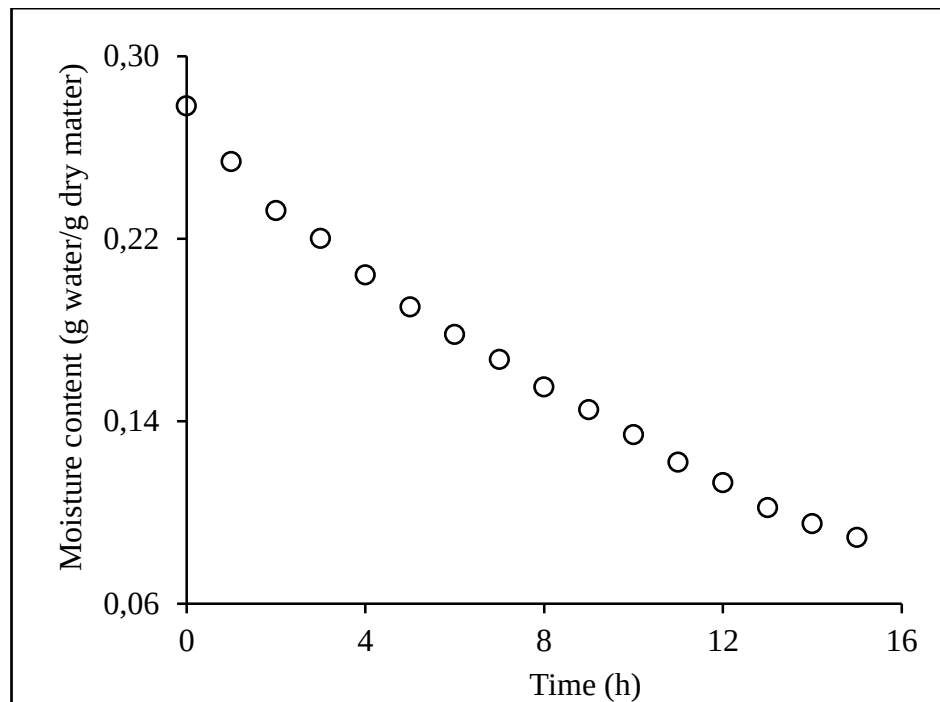


Figure 3.17: Variation of moisture content with time for the freeze dried of bee pollen.

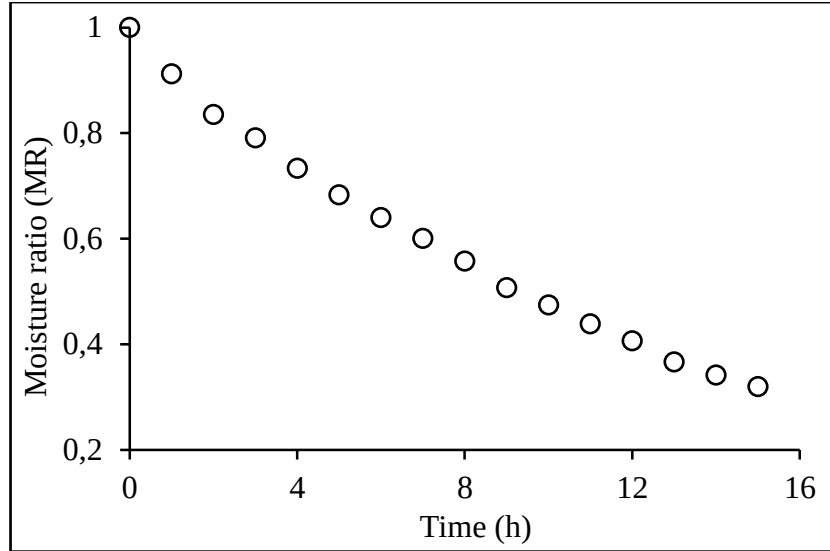


Figure 3.18: Moisture ratio versus time for the freeze dried bee pollen.

3.4.2. Effective Moisture Diffusivity for Freeze Drying

To calculate effective moisture diffusivity, the natural logarithm of the moisture ratio values was plotted against drying time based on the experimental data obtained for the freeze dried bee pollen as shown in Figure 3.19. D_{eff} value was calculated using Eq. (2.8) from the slope of the line and found as $1.90 \times 10^{-11} \text{ m}^2/\text{s}$ which was within the range of 10^{-12} to $10^{-6} \text{ m}^2/\text{s}$ given for food materials [Panagiotou et al., 2004].

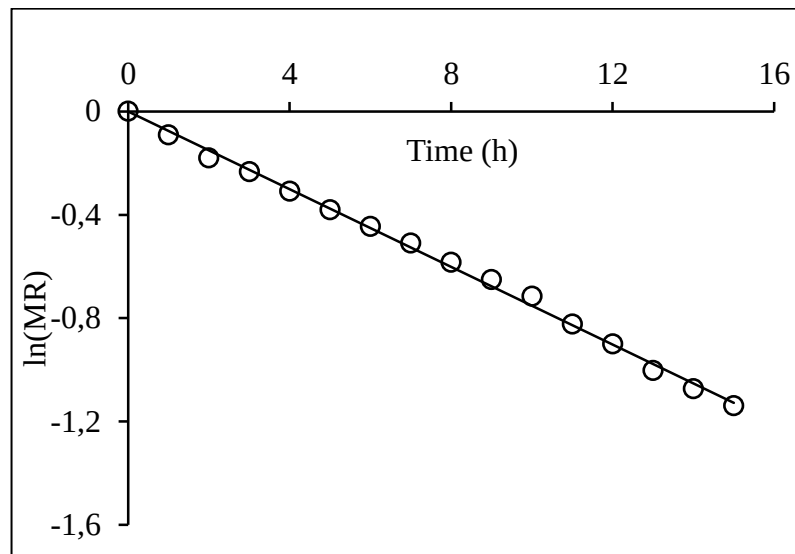


Figure 3.19: Variation of $\ln(\text{MR})$ with drying time.

3.4.3. Effect of Freeze Drying on Physicochemical Properties

Physicochemical properties of fresh and freeze dried bee pollen samples are given in Table 3.13. Fresh bee pollen was found to have crude proteins (30.36%), fats (5.50%), carbohydrates (61.96%), ash (2.18%) and vitamin C (38.48%). Although the crude protein of the fresh bee pollen changed from 30.36 ± 0.63 to 28.41 ± 0.30 g/100 g when it was freeze dried, freeze drying did not cause a significant ($p \leq 0.05$) decrease in the protein content of the fresh bee pollen. In other words, freezing and drying steps in freeze drying did not cause a serious impact on the denaturation of proteins due to low freezing and drying temperatures used. An increase in drying temperature in hot air drying, and increase in infrared and microwave power outputs in infrared and microwave dryings caused substantial losses in protein content of bee pollen as compared to freeze drying. Similar results were also observed by Shams et al. (2022) where freeze dried mushrooms had higher crude protein content than cabinet dried mushrooms at 50°C. Tolera and Abera (2017) reported that hot air oven drying of mushrooms at 60°C yielded dried mushrooms with the lowest protein content.

There was no significant difference ($p \leq 0.05$) in the fat contents of fresh and freeze dried bee pollens. Statistically insignificant difference in fat contents of fresh and freeze dried bee pollens can be interpreted that low temperatures during freeze drying prevents oxidation of polyunsaturated fatty acids which are prone to oxidation and allows better retention of crude fats in freeze dried bee pollens as compared to hot air, infrared and microwave drying methods.

Freeze drying did not significantly ($p > 0.05$) affect ash content of fresh bee pollen. As compared with the ash content results of Indian fresh bee pollens (2.27-3.46 g/100 g) [Thakur and Nanda, 2018] and Chinese fresh bee pollens (1.70-5.01 g/100 g) [Yang et al., 2013], the ash content of fresh bee pollen in this study was 2.18 ± 0.02 g/100 g comparable with the ash contents reported for Indian and Chinese fresh bee pollens. There was also no significant difference ($p > 0.05$) in ash contents of freeze dried, hot air, infrared and microwave dried bee pollen samples. Shams et al. (2022) also determined that there was no significant difference ($p > 0.05$) in ash contents of freeze dried and cabinet dried mushrooms.

Carbohydrates in bee pollen are important to obtain pleasant flavor, color and taste when the bee pollen is used as an ingredient in baked products [Yan et al., 2021]. Significant differences ($p \leq 0.05$) were observed between the total carbohydrates of fresh and freeze dried bee pollen samples. Total carbohydrates for the freeze dried bee pollen sample was found as 63.98 ± 0.42 g/100 g while the total carbohydrates in fresh bee pollen was 61.96 ± 0.59 g/100 g. Freeze drying is a critical operation as it can cause cell damage. Ice crystals within the cells of food being frozen increase as the temperature decreases, which causes rupturing the cell walls, thus facilitating the release of more food constituents including carbohydrates [Oyinloye and Yoon, 2020]. Higher total carbohydrates were obtained in hot air dried bee pollen samples. This implies that temperature has more pronounce effect on the total carbohydrate content of bee pollens dried by different drying methods.

Significant differences ($p \leq 0.05$) were observed between vitamin C contents of fresh and freeze dried bee pollen samples. As compared to hot air, infrared and microwave drying methods, freeze drying was a highly effective drying method in retention of vitamin C in bee pollen. Higher losses in vitamin C contents of hot air, infrared and microwave dried bee pollens are expected because vitamin C is heat sensitive and can degrade at high temperature [Shams et al., 2022]. Gebczynski et al. (2017) found that a higher loss (69%) in vitamin C content of pears occurred when they dried in a convention dryer at 60°C as compared to freeze dried pears in which freeze drying resulted in better retention of vitamin C than other drying methods.

Table 3.13: Physicochemical properties of fresh and freeze dried bee pollens¹.

	Fresh	Freeze dried
Crude proteins (g/100 g)	30.36 ± 0.63^a	28.41 ± 0.30^a
Fats (g/100 g)	5.50 ± 0.04^a	5.44 ± 0.18^a
Ash (g/100 g)	2.18 ± 0.02^a	2.17 ± 0.02^a
Total carbohydrates (g/100 g)	61.96 ± 0.59^a	63.98 ± 0.42^b
Vitamin C (mg/100 g)	38.48 ± 0.69^a	34.78 ± 1.22^b

¹Values are expressed in mean $\pm$ standard deviation, and values with the same letter within the same row are not statistically significant at $p > 0.05$.

3.4.4. Effect of Freeze Drying on Solubility Index

The solubility index of freeze dried bee pollen samples after 50 s of dissolution was between 9 and 10 (Figure 3.20). The solubility index values of hot air, infrared and microwave dried bee pollens were between 6.9 and 8.8, 8 and 8.5, and 4 and 6.5, respectively. The highest solubility index was obtained for freeze dried bee pollen. This means that the freeze dried bee pollen had the highest solubility in water as compared to the hot air, infrared and microwave dried bee pollens. This observation is in good agreement with the findings of Park et al. (2016) who determined that the solubility index of freeze dried kimchi powder was higher than the solubility index of hot air dried kimchi powder. Barajas et al. (2012) reported that after 50 s of dissolution, the solubility index values of two different Colombian bee pollens dried at 45°C were 3.5 and 5.5, which were lower than the solubility index values of dried bee pollens in this study. This means that the dried bee pollen samples in this study are more easily soluble in water than the dried Colombian bee pollen samples.

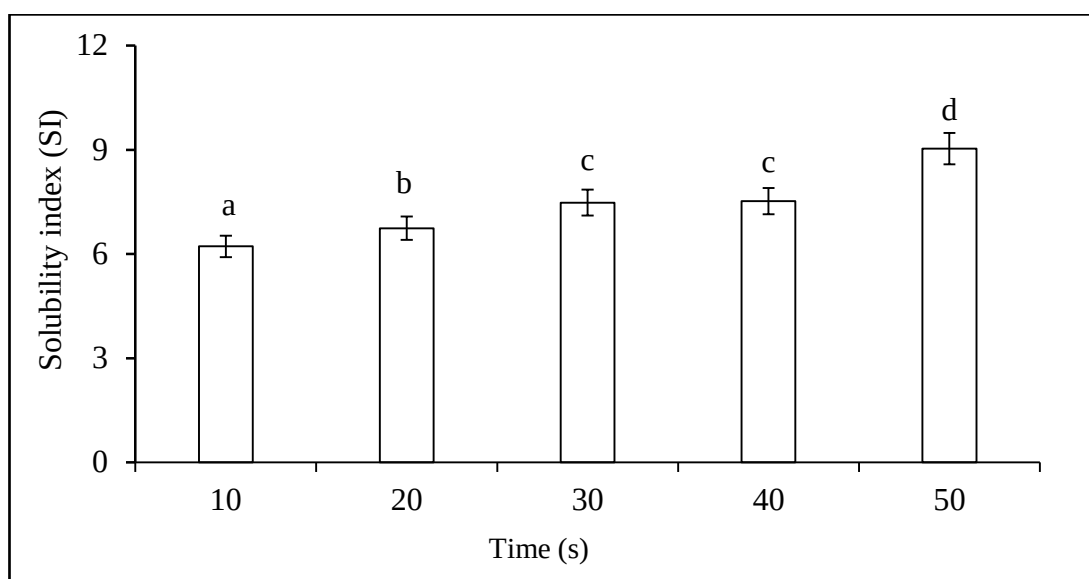


Figure 3.20: Solubility index of freeze dried bee pollen. Different letters indicate significant differences ($p \leq 0.05$) among treatments.

3.4.5. Effect of Freeze Drying on Color

Color is a commonly used quality attribute for dried foods by consumers. Color values of the fresh and freeze dried bee pollen samples are presented in Table 3.14.

There was no significant difference ($p > 0.05$) between the L^* , a^* and b^* values of the fresh and freeze dried bee pollens. A significant difference ($p \leq 0.05$) was observed between the L^* , a^* and b^* values of the freeze dried, and hot air, infrared dried and microwave dried bee pollens. Hot air, infrared dried and microwave dried bee pollens comparatively showed lower L^* and b^* values, but higher a^* values than the freeze dried bee pollen. The increase in a^* value and decrease in L^* value could result from non-enzymatic browning (Maillard reaction) during the drying process [Doymaz et al., 2015], [Mireles-Arriaga et al., 2016], [Doymaz, 2018]. The decrease in the b^* value is interpreted as a decrease in yellow color. Sublimation of frozen water (ice) during freeze drying reduces availability of oxygen and prevents occurrence of enzymatic browning, thus stabilizing color [Henríquez et al., 2013]. Low temperatures during freeze drying prevent or limit non-enzymatic browning reactions, thus maintaining color [Park et al., 2016]. This explains why freeze dried products are superior as compared to products dried by other drying methods including hot air, infrared and microwave dryings.

The analysis of ΔE values of the hot air, infrared dried, microwave dried and freeze dried bee pollen samples indicated that ΔE value was the lowest for the freeze dried bee pollen while hot air, infrared and microwave dried bee pollen samples had higher ΔE values than the freeze dried bee pollen. As lower ΔE values are favorable, the color of the freeze dried bee pollen is the closest to the color of the fresh bee pollen. All these findings reveal that freeze drying is the best drying method maintaining color of the bee pollen among other drying methods.

Table 3.14: Color parameters of fresh and freeze dried bee pollen samples¹.

	L^*	a^*	b^*	ΔE
Fresh	56.50 ± 0.84^a	10.78 ± 0.38^a	64.23 ± 2.22^a	-
Freeze dried	55.97 ± 0.76^a	10.42 ± 0.22^a	63.50 ± 2.09^a	1.99 ± 0.44

¹Values are expressed in mean $\pm$ standard deviation, and values with the same letter within the same column are not statistically significant at $p > 0.05$.

3.4.6. Morphological Structure of Freeze Dried Bee Pollen

Scanning electron microscopy provides qualitative information regarding surface structures of foods and helps improve the understanding of drying mechanisms in relation to the changes in the food structure [Dhalsamant et al., 2018]. Fresh bee pollen grains were bilaterally symmetrical and mostly had an elliptical shape with a longitudinal crevice (Figure 3.21a). The surface of the fresh bee pollen grains is mostly smooth, but the surface of the freeze dried bee pollen grains is rough, and tiny marks on the surface are noticeable (Figure 3.21b). SEM images of the freeze dried bee pollens are comparable with the SEM photos of bee pollens hot air dried at 40°C and infrared dried at 50 W. Shrinkage was minimal in freeze dried bee pollens, but shrinkage was the predominant change in the microwave dried bee pollens [Isik et al., 2021]. Freeze drying keeps the original shape of the wet material with minimum shrinkage because it removes frozen water by sublimation.

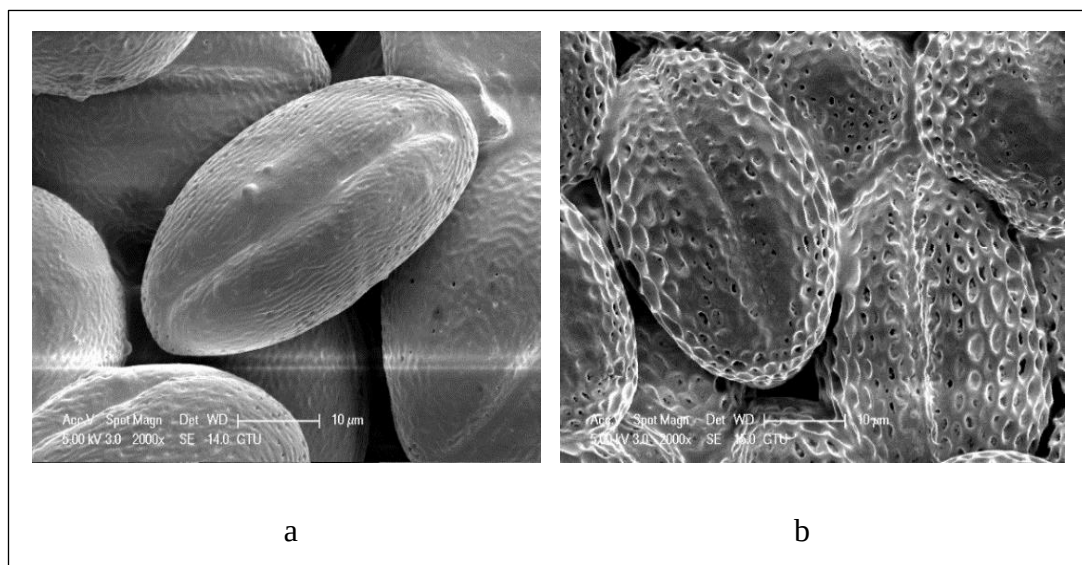


Figure 3.21: Scanning electron micrographs of (a) fresh and (b) freeze dried bee pollens. Scale bar is 10 μm. Magnification: 2000 ×.

3.4.7. Effect of Freeze Drying on Sensory Attributes

Evaluation of sensory properties of foods is of utmost importance as it reflects direct potential consumer preference. Visual appearances of dried foods play a very important role at the time of their sales. Therefore, analysis of some quality

parameters such as appearance, color, texture, odor and taste can be used in the maintenance of product quality [Fernandes et al., 2011]. In this study, visual appearance, color, odor and overall acceptance of the freeze dried bee pollen samples were evaluated by the panelists. Mean sensory scores (Table 3.15) showed that there was no significant difference ($p > 0.05$) between the visual appearance, color, odor and overall acceptance scores of fresh and freeze dried bee pollens. Compared to the sensory scores of hot air, infrared and microwave dried bee pollen samples, freeze dried bee pollen took the highest sensory scores while the bee pollen microwave dried at 900 W had the lowest sensory scores. This could be attributed to the occurrence of non-enzymatic browning reactions in freeze dried bee pollen in a much lesser extent. Hu et al. (2013) determined that freeze dried hairtail fish meat gel had higher overall acceptance score than hot air and microwave vacuum dried samples. Similarly, Park et al. (2016) reported that overall acceptability score of freeze dried kimchi powder was higher than that of hot air dried kimchi powder.

Table 3.15: Effect of freeze drying on the sensory scores of freeze dried bee pollen samples¹.

	Visual appearance	Color	Odor	Overall acceptance
Fresh	8.74 ± 0.15^a	8.71 ± 0.18^a	8.77 ± 0.12^a	8.72 ± 0.13^a
Freeze dried	8.70 ± 0.15^a	8.60 ± 0.49^a	8.50 ± 0.33^a	8.54 ± 0.21^a

¹Values are expressed in mean $\pm$ standard deviation, and values with the same letter within the same column are not statistically significant at $p > 0.05$.

4. CONCLUSIONS

Effect of different drying methods on quality, physicochemical properties, morphological structure and sensory characteristics of bee pollen were investigated. Higher temperatures, infrared and microwave power levels reduced the drying time, but they affected the physicochemical properties, morphological structure and sensory characteristics of the bee pollen. Crude proteins, fats and total carbohydrates of the dried bee pollens were affected by the drying temperature, and infrared and microwave power levels during drying. Freeze drying did not cause any significant change ($p > 0.05$) in crude protein, fat and total carbohydrate contents of the freeze dried bee pollen. Ash content was not affected by the drying method. As compared to hot air, infrared and microwave drying methods, freeze drying was a highly effective drying method in retention of vitamin C in bee pollen. The D_{eff} values for the bee pollens subjected to different drying methods varied from 1.90×10^{-11} to 0.04×10^{-8} m^2/s , which were within the range of 10^{-12} to 10^{-6} m^2/s given for food materials in literature. The freeze dried bee pollen had the highest solubility in water as compared to the hot air, infrared and microwave dried bee pollens. L^* , a^* and b^* values were affected by the drying temperature, and infrared and microwave power levels during drying. There was no significant difference ($p > 0.05$) between the L^* , a^* and b^* values of the fresh and freeze dried bee pollens. The ΔE value was the lowest for the freeze dried bee pollen while hot air, infrared and microwave dried bee pollen samples had higher ΔE values than the freeze dried bee pollen. The color of the freeze dried bee pollen was found to be the closest to the color of the fresh bee pollen. Morphological change and shrinkage were minimal in freeze dried bee pollens, but shrinkage was the predominant change in the microwave dried bee pollens whose morphological structure was highly affected. Compared to the sensory scores of hot air, infrared and microwave dried bee pollen samples, freeze dried bee pollen took the highest sensory scores while the bee pollen microwave dried at 900 W had the lowest sensory scores. Freeze dried bee pollen retained its quality attributes better than hot air, infrared and microwave dried bee pollens. Freeze drying is found to be more appropriate method for producing high quality dried bee pollen than the other drying methods, but it takes so long. Hot air drying at 40°C is recommended as a cost effective and faster alternative to freeze drying.

REFERENCES

- Adak N., Heybeli N., Ertekin C., (2017), "Infrared drying of strawberry", *Food Chemistry*, 219, 109–116.
- Aghbashlo M., Kianmehr M. H., Samimi-Akhijahani H., (2008), "Influence of drying conditions on the effective moisture diffusivity, energy of activation and energy consumption during the thin-layer drying of berberis fruit (Berberidaceae)", *Energy Conversion and Management*, 49 (10), 2865–2871.
- Al-Harashsheh M., Al-Muhtaseb A. H., Magee T. R. A., (2009), "Microwave drying kinetics of tomato pomace: effect of osmotic dehydration", *Chemical Engineering and Processing*, 48, 524–531.
- Almeida-Muradian L. B., Pamplona L. C., Coimbra S., Barth O. M., (2005), "Chemical composition and botanical evaluation of dried bee pollen pellets", *Journal of Food Composition and Analysis*, 18, 105–111.
- Association of Official Analytical Chemists–AOAC., (2005), "Official methods of analysis of AOAC international", 18th Edition, USA: Gaithersburg, MD.
- Baomeng Z., Xuesen W., Guodong W., (2014), "Effect of pre-treatments on drying characteristics of Chinese jujube (*Zizyphus jujuba* Miller)", *International Journal of Agricultural and Biological Engineering*, 7 (1), 94–101.
- Barajas J., Cortes-Rodriguez M., Rodriguez-Sandoval E., (2012), "Effect of temperature on the drying process of bee pollen from two zones of Colombia", *Journal of Food Process Engineering*, 35 (1), 134–148.
- Bayram N. E., (2019), "Quality evaluation and pollen profile of honey samples from different locations", *Progress in Nutrition*, 21, 928–934.
- Bogdanov S., (2004), "Quality and standards of pollen and beeswax", *Apiacta*, 38, 334–341.
- Bonvehí J. S., Jorda R. E., (1997), "Nutrient composition and microbiological quality of honeybee-collected pollen in Spain", *Journal of Agricultural and Food Chemistry*, 45, 725–732.
- Borel L. D. M. S., Marques L. G., Prado M. M., (2020), "Performance evaluation of an infrared heating-assisted fluidized bed dryer for processing bee-pollen grains", *Chemical Engineering and Processing*, 155, 108044.
- Campos M. G. R., Bogdanov S., de Almeida-Muradian L. B., Szczesna T., Mancebo Y., Frigerio, C., Ferreira F. J., (2008), "Pollen composition and standardisation of analytical methods", *Journal of Apicultural Research*, 47 (2), 154–161.
- Campos M. G. R., Frigerio C., Lopes J., Bogdanov S., (2010), "What is the future of Bee-Pollen?", *Journal of ApiProduct and ApiMedical Science*, 2 (4), 131–144.

Canale A., Benelli G., Castagna A., Sgherri C., Poli P., Serra A., Mele M., Ranieri A., Signorini F., Bientinesi M., Nicoletta C., (2016), “Microwave-assisted drying for the conservation of honeybee pollen”, *Materials*, 9, 363–374.

Cao Z., Zhou L., Bi J., Yi J., Chen Q., Wu X., Zheng J., Li S., (2016), “Effect of different drying technologies on drying characteristics and quality of red pepper (*Capsicum frutescens* L.): A comparative study”, *Journal of the Science of Food and Agriculture*, 96 (10), 3596–3603.

Carpes S. T., Cabral I. S. R., Rosalen P. I., de Alencar S. M., Masson M. L., (2009), “Caracterização do potencial antimicrobiano dos extratos de pólen apícola da região Sul do Brasil”, *Alimentos e Nutrição Araraquara*, 20 (2), 271–277.

Chandrasekaran S., Ramanathan S., Basak T., (2013), “Microwave food processing—a review”, *Food Research International*, 52, 243–261.

Crank J., (1975), “The Mathematics of Diffusion”, 2nd Edition, UK: Clarendon.

Dadali G., Demirhan E., Ozbek B., (2007), “Color change kinetics of spinach undergoing microwave drying”, *Drying Technology*, 25, 1713–1723.

Dadali G., Ozbek B., (2009), “Kinetic thermal degradation of vitamin C during microwave drying of okra and spinach”, *International Journal of Food Sciences and Nutrition*, 60, 21–31.

Darvishi H., (2017), “Quality, performance analysis, mass transfer parameters and modeling of drying kinetics of soybean”, *Brazilian Journal of Chemical Engineering*, 34, 143–158.

De-Melo A. A. M., Estevinho M. L. M. F., Sattler J. A. G., Souza B. R., Freitas A. S., Barth O. M., Almeida-Muradian L. B., (2016), “Effect of processing conditions on characteristics of dehydrated bee-pollen and correlation between quality parameters”, *LWT—Food Science and Technology*, 65, 808–815.

Demiray E., Seker A., Tulek Y., (2017), “Drying kinetics of onion (*Allium cepa* L.) slices with convective and microwave drying”, *Heat and Mass Transfer*, 53 (5), 1817–1827.

Denisow B., Denisow-Pietrzyk M., (2016), “Biological and therapeutic properties of bee pollen: a review”, *Journal of the Science of Food and Agriculture*, 96, 4303–4309.

Dhalsamant, K., Tripathy, P. P., Shrivastava, S. L., (2018), “Effect of sodium metabisulfite pretreatment on micrographs, surface roughness and X-ray diffraction analyses of solar dried potato cylinders”, *Innovative Food Science and Emerging Technologies*, 47, 399–411.

Dias L. G., Tolentino G., Pascoal A., Estevinho L. M., (2016), “Effect of processing conditions on the bioactive compounds and biological properties of bee pollen”, *Journal of Apicultural Research*, 55 (5), 357–365.

Doymaz I., (2004), “Effect of pre-treatments using potassium metabisulphide and alkaline ethyl oleate on the drying kinetics of apricots”, *Biosystems Engineering*, 89 (3), 281–287.

Doymaz I., (2012), “Mathematical modeling of drying of tomato slices using infrared radiation”, *Journal of Food Processing and Preservation*, 38, 389–396.

Doymaz I., (2015), “Infrared drying kinetics and quality characteristics of carrot slices”, *Journal of Food Processing and Preservation*, 39 (6), 2738–2745.

Doymaz I., (2018), “Infrared drying of kiwifruit slices”, *International Journal of Green Energy*, 15, 622–628.

Estevinho L. M., Rodrigues S., Pereira S. P., Feás X., (2012), “Portuguese bee pollen: palynological study, nutritional and microbiological evaluation”, *International Journal of Food Science and Technology*, 47 (2), 429–435.

Evin D., (2012), “Thin layer drying kinetics of *Gundelia tournefortii* L.”, *Food and Bioproducts Processing*, 90, 323–332.

Fernandes F. A. N., Rodrigues S., LAW C. L., Mujumdar A. S., (2011), “Drying of exotic tropical fruits: A comprehensive review”, *Food and Bioprocess Technology*, 4, 163–185.

Gebczynski P., Skoczeń-Słupska R., Kur K., (2017), “Effect of storage on the content of selected antioxidants and quality attributes in convection and freeze-dried pears (*Pyrus communis* L.)”, *Italian Journal of Food Science*, 29 (3), 454–462.

Henríquez M., Almonacid S., Lutz M., Simpson R., Valdenegro M., (2013), “Comparison of three drying processes to obtain an apple peel food ingredient”, *CyTA-Journal of Food*, 11 (2), 127–135.

Hu Y., Que T., Fang Z., Liu W., Chen S., Liu D., Ye X., (2013), “Effect of different drying methods on the protein and product quality of hair tail fish meat gel”, *Drying Technology*, 31, 1707–1714.

Isik A., Ozdemir M., Doymaz I., (2019a), “Effect of hot air drying on quality characteristics and physicochemical properties of bee pollen”, *Food Science and Technology*, 39, 224–231.

Isik A., Ozdemir M., Doymaz I., (2019b), “Infrared drying of bee pollen: Effects and impacts on food components”, *Czech Journal of Food Sciences*, 37, 69–74.

Isik A., Ozdemir M., Doymaz I., (2021), “Investigation of microwave drying on quality attributes, sensory properties and surface structure of bee pollen grains by scanning electron microscopy” *Brazilian Journal of Chemical Engineering*, 38, 177–188.

Jiang H., Zhang M., Mujumdar A. S., Lim R. X., (2014), “Comparison of drying characteristic and uniformity of banana cubes dried by pulse-spouted microwave

vacuum drying, freeze drying and microwave freeze drying”, *Journal of the Science of Food and Agriculture*, 94 (9), 1827–1834.

Khalifa S. A. M., Elashal M. H., Yosri N., Du M., Musharraf S. G., Nahar L., Sarker S. D., Guo Z., Cao W., Zou X., El-Wahed A. A. A., Xiao J., Omar H. A., Hegazy M-E. F., El-Seedi H. R., (2021), “Bee Pollen: Current Status and Therapeutic Potential”, *Nutrients*, 13, 1876-1891.

Khraisheh M. A. M., McMinn W. A. M., Magee T. R. A., (2004), “Quality and structural changes in starchy foods during microwave and convective drying”, *Food Research International*, 37, 497–503.

Kostić A. Ž., Milinčić D. D., Barać M. B., Shariati M. A., Tešić Ž. L., Pešić M. B., (2020), “The application of pollen as a functional food and feed ingredient—the present and perspectives”, *Biomolecules*, 10, 84-120.

Kouhila M., Moussaoui H., Bahammou Y., Tagnamas Z., Lamsyehe H., Lamharrar A., Idlimam A., (2020), “Exploring drying kinetics and energy exergy performance of *Mytilus Chilensis* and *Dosidicus gigas* undergoing microwave treatment”, *Heat and Mass Transfer*, 56, 2985–2999.

Li Q. Q., Wang K., Marcucci M. C., Sawaya A. C. H. F., Hu L., Xue X. F., Wu L. M., Hu F. L., (2018), “Nutrient-rich bee pollen: a treasure trove of active natural metabolites”, *Journal of Functional Foods*, 49, 472–484.

Madamba P. S., Driscoll R. H., Buckle, K. A., (1996), “The thin-layer drying characteristics of garlic slices”, *Journal of Food Engineering*, 29 (1), 75–97.

Meilgaard M. C., Civille C. V., Carr B. T., (2016), “Sensory Evaluation Techniques”, 5th Edition, USA: CRC Press.

Mireles-Arriaga A. I., Ruiz-López I. I., Hernández-García P. A., Espinosa-Ayala E., López-Martínez L. X., Márquez-Molina O., (2016), “The impact of convective drying on the color, phenolic content and antioxidant capacity of noni (*Morinda citrifolia* L.)”, *Food Science and Technology*, 36, 583–590.

Ojha K. S., Kerry J. P., Tiwari B. K., (2017), “Investigating the influence of ultrasound pre-treatment on drying kinetics and moisture migration measurement in *Lactobacillus sakei* cultured and uncultured beef jerky”, *LWT–Food Science and Technology*, 81, 42–49.

Olanipekun B. F., Tunde-Akintunde T. Y., Oyelade O. J., Adebisi M. G., Adenaya T. A., (2015), “Mathematical modeling of thin-layer pineapple drying”, *Journal of Food Processing and Preservation*, 39, 1431–1441.

Oyinloye T. M., Yoon W. B., (2020), “Effect of freeze-drying on quality and grinding process of food produce: A review”, *Processes*, 8 (3), 354-377.

Panagiotou N. M., Krokida M. K., Maroulis Z. B., Saravacos G. D., (2004), “Moisture diffusivity: Literature data compilation for foodstuffs”, *International Journal of Food Properties*, 7, 273–299.

Park H., Lee Y., Eun J., (2016), “Physicochemical characteristics of kimchi powder manufactured by hot air drying and freeze drying”, *Biocatalysis and Agricultural Biotechnology*, 5, 193–198.

Pelletier S., Tremblay G. F., Bertrand A., Bélanger G., Castonguay Y., Michaud R., (2010), “Drying procedures affect non-structural carbohydrates and other nutritive value attributes in forage samples”, *Animal Feed Science and Technology*, 157, 139–150.

Phukasmas P., Songsermpong S., (2019), “Instant rice process development: effect of rice cooking methods on the quality of Jasmine instant rice dried by industrial microwave oven”, *Journal of Microbiology, Biotechnology and Food Sciences*, 9, 330–334.

Ratti C., (2001), “Hot air and freeze-drying of high-value foods: a review”, *Journal of Food Engineering*, 49 (4), 311–319.

Rayaguru K., Routray W., Mohanty S. N., (2011), “Mathematical modeling and quality parameters of air-dried betel leaf (*Piper Betle* L.)”, *Journal of Food Processing and Preservation*, 35 (4), 394–401.

Reis R. C., Castro V. C., Devilla I. A., Oliveria C. A., Barbosa L. S., Rodovalho R., (2013), “Effect of drying temperature on the nutritional and antioxidant qualities of cumari peppers from Pará (*Capsicum chinense* Jacqui)”, *Brazilian Journal of Chemical Engineering*, 30, 337–343.

Sadin R., Chegini, G. R., Sadin H., (2014), “The effect of temperature and slice thickness on drying kinetics tomato in the infrared dryer”, *Heat and Mass Transfer*, 50, 501–507.

Salamanca Grosso G., Pérez Figueredo C. R., Vargas, E. F. (2008), “Origen botánico propiedades fisicoquímicas microbiológicas del polen colectado en algunas zonas apícolas de la Campiña de Boyacá [Resumen]. II Congreso Iberoamericano sobre Seguridad Alimentaria, V Congreso Español de Ingeniería de Alimentos”, In V Congreso Español de Ingeniería de Alimentos. Barcelona, España.

Schulte F., Lingott J., Panne U., Kneipp J., (2008), “Chemical characterization and classification of pollen”, *Analytical Chemistry*, 80, 9551–9556.

Shams R., Singha J., Dashb K. K., Dar A. H., (2022), “Comparative study of freeze drying and cabinet drying of button mushroom”, *Applied Food Research*, 2 (1), 100084.

Shewale S. R., Rajoriya D., Hebbar H. U., (2019), “Low humidity air drying of apple slices: Effect of EMR pretreatment on mass transfer parameters, energy efficiency and quality”, *Innovative Food Science and Emerging Technologies*, 55, 1–10.

Soni A., Smith J., Thompson A., Brightwell G., (2020), “Microwave induced thermal sterilization—a review on history, technical progress, advantages and challenges as compared to the conventional methods”, *Trends in Food Science and Technology*, 97, 433–442.

Thakur M., Nanda V., (2018), “Assessment of physico-chemical properties, fatty acid, amino acid and mineral profile of bee pollen from India with a multivariate perspective”, *Journal of Food and Nutrition Research*, 57, 328–340.

Thakur M., Nanda V., (2020a), “Composition and functionality of bee pollen: a review”, *Trends in Food Science and Technology*, 98, 82–106.

Thakur M., Nanda V., (2020b), “Exploring the physical, functional, thermal, and textural properties of bee pollen from different botanical origins of India”, *Journal of Food Process Engineering*, 43, e12935.

Tolera K., D., Abera S., (2017), “Nutritional quality of Oyster Mushroom (*Pleurotus Ostreatus*) as affected by osmotic pretreatments and drying methods”, *Food Science and Nutrition*, 5, 989–996.

Villanueva M. T. O., Marquina A. D., Serrano, R. B., Abellan G. B., (2002), “The importance of bee-collected pollen in the diet: a study of its composition”, *International Journal of Food Sciences and Nutrition*, 53 (3), 217–224.

Wang Z., Sun J., Chen F., Liao, X., Hu X., (2007), “Mathematical modelling on thin layer microwave drying of apple pomace with and without hot air pre-drying”, *Journal of Food Engineering*, 80, 536–544.

Węglińska M., Szostak R., Kita A., Nemš A., Mazurek S., (2020), “Determination of nutritional parameters of bee pollen by Raman and infrared spectroscopy”, *Talanta*, 212, 120790.

Wojdyło A., Figiel A., Legua P., Lech K., Carbonell-Barrachina Á. A., Hernández F., (2016), “Chemical composition, antioxidant capacity, and sensory quality of dried jujube fruits as affected by cultivar and drying method”, *Food Chemistry*, 207, 170–179.

Yan S., Wan Y., Wang F., Xue X., Wu L., (2021), “Fortification of bread with bee pollen, and its effects on quality attributes and antioxidant activity”, *International Food Research Journal*, 28, 517–526.

Yang K., Wu D., Ye X., Liu D., Chen J., Sun P. L. (2013), “Characterization of chemical composition of bee pollen in China”, *Journal of Agricultural and Food Chemistry*, 61, 708–718.

BIOGRAPHY

Ayla IŞIK started B.S. study in Celal Bayar University, Faculty of Engineering Department of Food Engineering, and she received her undergraduate degree in 2008. She also holds two B.S. degrees from Anadolu University, Faculty of External Education, Departments of Public Relations and Business Administration. She worked at Darıca Farabi Public Hospital as a food engineer between 2009 and 2014. She has been working as a quality control officer in Beyazpınar Confectionery Factory since 2021. She is currently pursuing Ph. D. study at Gebze Technical University, Institute of Natural Sciences.