GAZİANTEP UNIVERSITY GRADUATE SCHOOL OF NATURAL & APPLIED SCIENCES

DRIED AND CONCENTRATED PRODUCTS FROM MULBERRY (*Morus alba* and *Morus nigra*) AND THE CHANGES DURING THEIR PROCESSING

M.Sc. THESIS
IN
FOOD ENGINEERING

BY
DERYA KOÇAK
JANUARY 2009

Dried and Concentrated Products from Mulberry (*Morus alba* and *Morus nigra*) and the Changes during Their Processing

M.Sc. Thesis
In
Food Engineering
University of Gaziantep

Supervisor
Prof. Dr. Sami EREN

by
Derya KOÇAK
January 2009

T.C.
GAZİANTEP UNIVERSITY
GRADUATE SCHOOL OF NATURAL & APPLIED SCIENCES
(Department of Food Engineering)

Name of the thesis: Dried and Concentrated Products from Mulberry (*Morus alba* and *Morus nigra*) and the Changes during Their Processing
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Exam date: 12 January 2009

Approval of the Graduate School of Natural and Applied Sciences

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TO MY PARENTS

AYSEL & MİKAİL KOÇAK

ABSTRACT

DRIED AND CONCENTRATED PRODUCTS FROM MULBERRY (*Morus alba* and *Morus nigra*) AND THE CHANGES DURING THEIR PROCESSING

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M.Sc. in Food Eng.
Supervisor: Prof. Dr. Sami EREN
January 2009, 85 pages

In this study, some drying behaviors (drying curves, shrinkage and color changes) of white mulberry (*Morus alba*) and black mulberry (*Morus nigra*) have been studied for hot air and microwave drying. Hot-air drying was followed at three different temperatures as 60, 70 and 80^{o}C and it was observed that the increasing temperature caused significant reduction in drying time. When microwave energy was used instate of hot-air drying, reduction in drying time was also observed. It was found that both microwave and hot-air drying methods caused strong change on color as a function of time. The color deterioration rate was faster for microwave drying; however when the samples reached to equilibrium in drying, there was no significant difference in their final color values. Both type of mulberries showed a significant shrinkage.

The mulberry juice concentrates were produced by using various heating methods such as atmospheric, microwave and rotary vacuum. The concentration by microwave technique reduced time of evaporation considerably. A shorter time compared to atmospheric conditions and the best color quality compared to the two other techniques have been found for rotary vacuum concentration.

Key Words: mulberry, drying, shrinkage, color, concentration

ÖZET

DUTTAN (*Morus alba & Morus nigra*) KURUTULMUŞ VE KONSANTRE ÜRÜNLER ÜRETİLMESİ VE İŞLENMELERİ ESNASINDA MEYDANA GELEN DEĞİŞİMLER

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Tez Yöneticisi: Prof. Dr. Sami EREN
Ocak 2009, 85 sayfa

Bu çalışmada, beyaz (*Morus alba*) ve siyah (*Morus nigra*) dutun bazı kuruma özelliklerinin (kuruma eğrisi, büzüşme, renk değişikliği gibi) havalı kurutma ve mikrodalga enerjisiyle kurutma metotları esnasında nasıl seyrettiği incelendi. Sıcak hava ile kurutma 60, 70 ve 80^{o}C olmak üzere üç farklı sıcaklıkta takip edildi ve sıcaklık artışının kuruma süresini önemli derecede kısalttığı görüldü. Havalı kurutma yerine mikrodalga enerjisi kullanıldığında da kuruma süresinde kısalma gözlendi. Her iki kurutma metodunun da dutların renklerinde önemli değişimlere neden olduğu saptandı. Mikrodalga enerjisinin renk değişim hızını arttırdığı fakat önemli derecede azaltıcı veya arttırıcı etkisi olmadığı tespit edildi. Her iki dut çeşitininde kuruma esnasında önemli derecede hacim daralmasına uğradığı fakat sıcaklık artışının büzüşmenin azalması veya artması yönünde önemli bir etkiye sahip olmadığı görüldü.

Mikrodalga, açık hava ve vakumlu kurutma olmak üzere, farklı ısıtma metotları kullanılarak dut suyu konsantreleri üretildi. Mikrodalga ile ısıtma tekniğinin evaporasyon süresini önemli derecede kısalttığı görüldü. Vakum altında ısıtmanın, evaporasyon süresini açık havada ısıtmaya nazaran kısalttığı ve renk açısından da diğer iki tekniğe göre daha kaliteli ürün verdiği tespit edildi.

Anahtar Kelimeler: dut, kurutma, büzüşme, renk, konsantre

ACKNOWLEDGEMENT

I would like to express my sincere gratitude to my supervisor, Prof. Dr. Sami EREN for his guidance, interest and patience and also encouragement he provided throughout the course of this study.

I am also very grateful to Prof. Dr. Fahrettin GÖĞÜŞ for his guidance and invaluable discussions, but also for his kindness and readiness in assistance throughout this study.

I would like to thank to Assist. Prof. Dr. Bülent BELİBAĞLI for his help and support.

Special thanks to my family and my sweetheart, Rıza YANIK, for their kind help and patience at every stage of this study.

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ABREVIATIONS

AOAC	Association of Official Analytical Chemists
ANOVA	Analysis of variance
a,b	Constants for Equation 3.7
a^*	Redness-greenness
b^*	Yellowness-blueness
B,	The ^oBrix of samples at any time *t* respectively
B_o	The initial ^oBrix
B_1	Constant for Equation 3.8
^oBrix	Total soluble solid
C	Any quality parameters
C_o	Initial value of quality parameter
D_o	Diffusivity value for a infinite moisture content
ΔE^*	Total color differences
D_{eff}	Effective moisture diffusivity (m^2s^{-1})
Ea	Activation energy (kJ mol^{-1})
FB	Fluidized bed drying
GAE	Gallic acid equivalent
k	Rate constant
L^*	Lightness-darkness
MW	Microwave
r, r^2	Correlation coefficient
R	Universal gas constant ($8.3145x10^{-3}$ kJ $mol^{-1}K^{-1}$)
RH	Relative humidity
S_b	Bulk shrinkage ratio
SPSS	Statistical package for social sciences
T_{abs}	Absolute drying air temperature (K)
TPC	Total phenolic content
V	Volume of samples
V	Air velocity (m s^{-1})
$(V/V_o)_c$,	Critical volume ratio
$(V/V_o)_f$,	Final volume ratio
X	Moisture content (g water/g dry solid)

X_o	Initial moisture content
X_e	Equilibrium moisture content
$(X/X_o)_c$,	Critical moisture content ratio

CHAPTER I
INTRODUCTION

1.1. Mulberry

The mulberry belongs to the genus *Morus* of the family *Moraceae.* There are 24 species of *morus* and one subspecies, with at least 100 known varieties (Ercisli and Orhan, 2007). Black mulberry (*Morus nigra*) is one of the most important species of the *morus* genus. The other two are white mulberry (*Morus alba*) and purple mulberry (*Morus rubra*) (Elmacı and Altuğ, 2002). The fruits can be consumed fresh or dried as well as in the form of a cold drink. Black mulberry fruits are used in various confectionery products and also for medicinal purposes (Baytop, 1984).

Morus nigra

Morus alba

Morus rubra

1.1.1. Mulberry growth

Mulberry is found from temperate to subtropical regions of the Northern hemisphere to the tropics of the Southern hemisphere and they can grow in a wide range of climatic, topographical and soil conditions. These are widely spread throughout all regions from the tropics to the sub-arctic and from sea level to altitudes as high as 4000 m (Machii, Koyama and Yamanouchi, 2000; Tutin et al., 1996).

In most mulberry growing countries, in particular in India and China, mulberry is used for its foliage, to feed the silkworm (Bombyx mori L.). Mulberry breeding has focused on enhancing foliage production in these countries (Vijayan et al., 1997). However, in most European countries, including Turkey and Greece, mulberries are

grown for fruit production rather than foliage (Ercisli, 2004; Gerasopoulos and Stavroulakis, 1997).

Because mulberry has not selectivity for growing conditions, nearly all region of Turkey suitable for cultivating high quality mulberry fruits, mainly *morus alba (M. alba)*, *morus nigra* (*M. nigra*) and *morus rubra (M. rubra)*. 95% of the mulberry trees grown in Turkey are *M. alba,* 3% are *M. rubra* and 2% are *M. nigra* (Ercisli, 2004). Also Turkey is a natural spreading area of mulberry. However, this genetic potential was not good used; a lot of genotype which have an ability to give high quality fruits was obliterated to only use their board.

Table 1.1 shows the highest mulberry production regions of Turkey. The production of mulberry in Turkey in 2003 was 50,000 tones from 2,130,000 mulberry trees (Anonymous, 2003). In this production Malatya has the largest portion, Ankara and Erzincan follows Malatya.

Table 1.1. The highest mulberry production regions of Turkey

Cities	Production(ton)	Number of total trees	Number of trees (bearing fruits)
Malatya	5,278	139,500	133,200
Ankara	4,168	105,149	75,614
Erzincan	4,139	171,224	151,256
Elazığ	3,843	153,580	127,650
Erzurum	2,514	68,407	60,845
Ordu	1,841	59,010	54,040
K.Maraş	1,642	65,130	57,530
Kütahya	1,614	52,800	48,620
Artvin	1,406	69,130	51,330
Kastamonu	1,342	56,264	41,744
Türkiye	**50,000**	**2,495,000**	**2,130,000**

1.1.2. Mulberry products

A lot of traditional and conventional mulberry products are available. Fruits, leaves, roots of mulberry trees may have medical uses, but these have to be more studied. Most interesting cultivars are these who give black fruits. They are full aromatic, and have an exceptional coloring power, especially for jam and ice cream. Granulated

mulberry is used as a flavouring material for a cultured milk drink manufactured in Armenia (Aganova, 1989). In addition, it is widely used in mulberry pekmez (Turkish concentrated mulberry juice product) production (Anonymous, 1986), canned mulberries, mulberry jams and juices (Snapyan et al., 1981), mulberry pulp, paste, marmalade and jelly (Khatiashvili et al., 1979). It is also used in alcoholic beverages such as wine production (Alian and Musenge, 1977)

1.1.3. Chemical composition of mulberry

Ercisli and Orhan (2007) studied the chemical composition of white (*M. alba*), red (*M. rubra*) and black (*M. nigra*) mulberry fruits grown in the East Anatolia Region of Turkey and reported the approximate composition as shown in Table 1.2.

Table 1.2. The chemical composition of *Morus alba, Morus nigra* and *Morus rubra*

	Morus alba	*Morus nigra*	*Morus rubra*
Moisture content (%)	71.5	72.6	74.6
Total soluble solids (%)	20.4	16.7	15.9
Total fat (%)	1.10	0.95	0.85
Major fatty acid (%)	Linoleic(54.2)	Palmitic(19.8)	Oleic (8.41)
Acidity (%)	0.25	1.40	1.37
pH	5.60	3.52	4.04
Ascorbic acid (mg/100ml)	22.4	21.8	19.4
TPC(mgGAE/100g fresh mass)	181	1422	1035
N (mg/100g)	0.75	0.92	0.82
P (mg/100g)	247	232	226
K (mg/100g)	1668	922	834
Ca (mg/100g)	152	132	132
Mg (mg/100g)	106	106	115
Na (mg/100g)	60	59	61
Fe (mg/100g)	4.2	4.2	4.5
Cu (mg/100g)	0.5	0.4	0.4
Mn (mg/100g)	3.8	4.2	4.0
Zn (mg/100g)	2.8	3.3	3.2

1.1.4. Phenolic content of mulberry

Epidemiological and experimental studies reveal a negative correlation between the consumption of diets rich in fruits, and vegetables and the risks for chronic angiogenic diseases, such as cardiovascular diseases, arthritis, chronic inflammation and cancers (Chen et al., 2005; Middleton et al., 2000; Prior, 2003; Saleem et al., 2002; Zhang et al., 2005). These physiological functions of fruits and vegetables may be partly attributed by their abundance of phenolics. Deep-colored vegetables and fruits are good sources of phenolics, including flavonoids and anthocyanins, and carotenoids (Cieslik et al., 2006; Qian et al., 2004; Sass-Kiss et al., 2005; Trappey et al., 2005).

Traditionally, deepcolored fruits, vegetables or foods are recognized as more healthy to human body, especially in the oriental countries. There has been a growing interest in pigment components of fruits and vegetables, which may promote human health or lower the risk for disease. Recent studies have focused on health functions of phenolics, including flavonoids and anthocyanins, and carotenoids from fruits and vegetables. Mulberry is one of these fruits. Jin-Yuarn Lin and Ching-Yin Tang (2007) studied about the total phenolic and flavonoid contents of strawberry, loquat, oriental plum, and mulberry. The results of this study shows that, mulberry has the highest phenolic content and its total phenolic content is about 1515.9 ± 5.7 mg gallic acid equevalent (GAE) /100 g fresh matter.

1.2. Preservation of Fruits and Vegetables by Dehydration

Dehydration of foods is one of the most common processes used to improve food stability, since it decreases considerably the water activity of the material, reduces microbiological activity and minimizes physical and chemical changes during its storage (Mayor and Sereno, 2003).

Fruits and vegetables have a high level of moisture content at harvest. They are very perishable; since they soften very quickly it causes difficulties during transportation and marketing. Because of the short season and the sensitivity to storage, several process technologies have been employed on an industrial scale to preserve fruits and vegetables; the major ones are canning, freezing, and dehydration. Among these,

dehydration is especially suited for developing countries with poorly established low-temperature and thermal processing facilities. Drying and concentrations are characteristic operation in dehydration, they offer a highly effective and practical means of preservation to reduce post harvest losses and offset the shortages in supply.

The dehydration of fruits and vegetables is most important in the production of the convenience foods. A great volume of fruits is sold as dry fruit with moisture content of 15 to 25 %. Also the production of concentrated fruit juices is of interest at industrial level since they can be used as ingredients in many products such as ice creams, fruit syrups, jellies and fruit juices beverages (Cassano et al., 2004).

The advantages of dehydrated fruits and vegetables are fairly obvious (1) their flavor stability at room temperature over long periods of time; (2) their protection from enzymatic and oxidative spoilage; (3) their light weight for shipping; (4) the fact that they do not need refrigeration and (5) their availability at all times of year (Spicer, 1974).

1.2.1. Drying

The drying of foods in order to preserve them during seasons of abundance for consumption during seasons of shortage is an ancient art. Its origins are unrecorded, but many of its practices have been handed down even in to the present day, and in some cases form the basis of modern food manufacturing processes (Van Arsdel et al, 1973). It is a process copied from nature. Drying, which is the characteristic operation in dehydration is the more or less complete removal of water from a moist material. The term is usually reversed for those processes which accomplish removal of water by evaporation rather than by pressure or by other physical means. More fruits are preserved by drying than by any other methods of food preservation (N-Desrosier and J-Desrosier, 1977).

Drying methods and equipments for fruits and vegetables

Dehydration involves the application of heat to vaporize moisture and some means of removing water vapor after its separation from the fruit and vegetable tissue. Hence it is a combined and simultaneous heat and mass transfer operation for which energy must be supplied. Several types of dryers and drying methods, each better suited for a particular situation, are commercially used to remove moisture from a wide variety of fruits and vegetables (Somogyi and Luh, 1986). Some of them are considered below.

(a) Solar drying

Whereas sun drying of fruit crops is still practiced for certain fruits such as prunes, grapes, and dates. Continuous processes, such as tunnel, belt-trough, and fluidized bed, are mainly used for vegetables (Jayaraman and Das Gupta, 2006).The main disadvantage of this process is that it is subject to wide variations in climatic and environmental conditions, and is thus poorly controllable. These problems include rain and cloudiness; contamination from dust and by insects, birds, and animals; lack of control over drying conditions; and possibility of chemical, enzymic, and microbiological spoilage due to long drying times. The final product has widely varying moisture content and organoleptic properties. Because of these reasons, sun-drying is being replaced by drying in a hot-air convection chamber, in which drying conditions can be selected (Holdsworth, 1971).

(b) Fluidized bed drying

The fluidized bed (FB) type of dryer was originally used for the finish drying of potato granules. In FB drying, hot air is forced through a bed of food particles at a sufficiently high velocity to overcome the gravitational forces on the product and maintain the particles in a suspended (fluidized) state (Van Arsdel and Copley, 1973).

Fluidizing is a very effective way of maximizing the surface area of drying within a small total space. Air velocities required for this will vary with the product and more specifically with the particle size and density. A major limitation is the limited range of particle size (diameter usually 20 mm–10 mm) that can be effectively fluidized (Jayaraman and Das Gupta, 2006).

(c) Explosion puffing

The technique of explosion puffing was initially developed to fulfill the objective of dehydrating relatively large pieces of fruits and vegetables that would reconstitute rapidly; the system would be operable at a cost comparable to conventional hot air drying. The method, adequately described and extensively reviewed in several articles (Holdsworth, 1971; Sullivan and Craig, 1984) consisted of initially partially dehydrating the fruit and vegetable pieces, then imparting a porous structure by explosion puffing, and subsequently drying to a low moisture content.

(d) Freeze drying

Freeze-drying, which involves a two-stage process of first freezing of water of the food materials followed by application of heat to the product so that ice can be directly sublimed to vapor, is already a commercially established process. Sublimation from ice to water vapor can only be accomplished below the triple point of water, that is, at 0.00626 atm at a temperature of approximately 164^{o}C. Since the moisture removal does not pass through a liquid phase, the structure of the product remains in a more acceptable state. In addition, drying takes place without exposing the product to excessively high temperatures.

The advantages of freeze-drying are: shrinkage is minimized; movement of soluble solids within the food material is minimized; the porous structure of the product facilitates rapid rehydration; and retention of volatile flavor compounds is high. It has therefore proved to be the superior method of dehydration for many fruits. The major limitation to its commercial application is its very high capital and processing costs and the need for special packaging to avoid oxidation and moisture pick up (Jayaraman and Das Gupta, 2006).

(e) Microwave drying

Although the primary objective of food drying is preservation, depending on the drying mechanism, some undesirable changes may be observed in the product due to browning reaction, lipid oxidation, nutritional loss and microbial growth. High temperatures or long drying times in conventional air drying may cause serious damage to the color, flavor, nutrients and rehydration capacity of the dried product. Microwave drying may be an alternative to reduce product degradation. It is suitable

for products having high moisture content like carrot, mushroom and cabbage because of the high dielectric properties of water that can quickly absorb the microwave energy (Parakash et al., 2003). The physical mechanisms involved in microwave drying are different from the mechanisms of conventional drying. The internal heat generated during microwave heating provides a vapour pressure within the product and pumps the moisture to the surface (Turner and Jolly, 1991). Case hardening does not occur in microwave drying because of this moisture pumping effect. Thus, an increased drying rate without increased surface temperature and improved product quality are obtained. However, the progress of microwave drying at the industrial level has been relatively slow due to its high initial capital investment. Non-uniform heating is another problem that hinders the commercial application of microwave drying (Mullin, 1995).

The usual means of applying microwaves to a drying process is at the end of the falling rate period, which is referred to as finish drying (Schiffmann, 2001) Generally, microwave drying of foods or food ingredients with a high moisture content (over 20 percent moisture) is not economical. Although water has a high dielectric constant and absorbs microwaves easily, it also has a very high specific heat capacity. Therefore, if the bulk of water is high, a considerable amount of microwave energy will be needed to raise significantly the temperature for dehydration (Owusu-Ansah, 1991). In general, microwave energy has been combined with hot air to shorten the drying times especially in the falling rate periods. In recent years, there have been a great number of studies about the applications of combined 'microwave- hot air' to different foods.

(f) Hot-air drying

Currently most of the dehydrated fruits and vegetables are produced by the technique of hot air drying, which is the simplest and most economical among the various methods. Air is generally used as the drying medium because it is plentiful, convenient, and overheating of food can be controlled. Different types of dryers have been designed, made, and commercially used based on this technique.

In this method, heated air is brought into contact with the wet material to be dried to facilitate heat and mass transfer; convection is mainly involved. Two important

aspects of mass transfer are the transfer of water to the surface of the material that is dried and the removal of water vapor from the surface.

The dryers generally used for the drying of piece form fruits and vegetables are cabinet, kiln, tunnel, belt-trough, bin, pneumatic, and conveyor dryers.

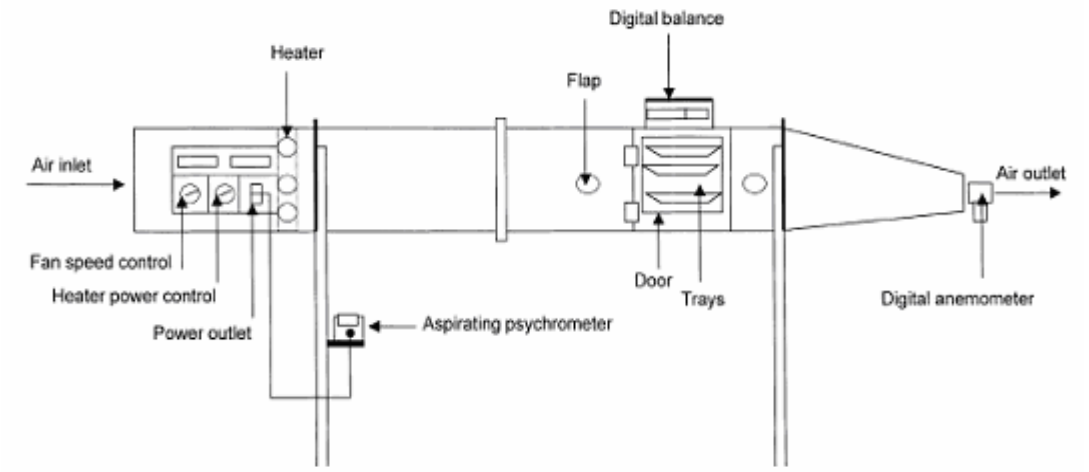

Figure 1.1 Schematic diagram of a laboratory tray type hot air drying equipment

Figure 1.1 is a diagrammatic sketch of a cabinet type dryer (Maskan, 2000), designed specifically for study of the air drying of cut pieces of fruits and vegetables, uniformly spread on shallow mesh-bottom trays. Fresh air drawn in past the adjustable recirculation damper is pulled through multiple fin-coil steam heaters by the centrifugal fan. The fan discharges through a set of turning vanes and perforated screens intended to straighten out and equalize the air flow through the tray section. The entire tray section is supported on a balance arm so that its gross weight can be followed continuously during the drying. Usually, individual trays are easily removable for quick weighing on separate scales.

1.2.2. Concentration

In concentration processes the water is removed from a fluid by molecular and eddy transport. The minimum water content is about 30 %. The process is generally steady state. This as distinct from drying where the water content is reduced to less than 10% by unsteady state molecular diffusion from more or less rigid slabs, droplets or particles (Thijssen, 1974).

Juices are produced from various fruits that are not necessarily harvested the whole year round. Before shipping to its final destination the extracted juice is concentrated

to ensure longer storage life (because of its low water activity) and easier transportation. Concentration processes have to be inert with respect to the chemically unstable constituents and to be selective, all components except water being retained in the concentrate.

Concentration of fruit juices, a major unit operation in fruit processing industry, is of critical importance as it determines the quality of the final product such as flavour, color, aroma, appearance and mouth feel (Cassano et al., 2004; Ramteke et al., 1993).

Concentration methods for fruit Juice

Liquid foods such as fruit juices are of high nutritive value as they are naturally enriched with minerals, vitamins, and other beneficial components required for human health. When fruit juices extracted from their sources, they have low solid content, color strength and high water load. Water, being the major constituent of liquid foods, contributes to the growth of the microorganisms. Removal of water helps to reduce microbial load, thereby favoring an increase in the shelf-life of the liquid foods. Hence it is desirable to concentrate these liquid foods to improve shelf life, stability and to reduce storage/transportation costs (Philip, 1984; Petrotos and Lazarides, 2001). Different concentration methods such as evaporative concentration, freeze concentration and membrane concentration are applicable.

(a) Membrane concentration:

Membrane processing is a technique that permits concentration and separation of macro- and micromolecules based on molecular size and shape. Membrane processing is fast emerging among various unit operations available for separation processes, especially in the field of chemical engineering, biotechnology and food processing. Better process economy, higher yield, improved product quality, utilizing of by-products and a solution to some environmental problems, can all be achieved by using membrane processing.

(b) Freeze concentration:

Freeze concentration is another method employed for the concentration of liquid foods such as fruit juices. Freeze concentration involves partial freezing of the product and removing of ice crystals, thus leaving behind all the non-aqueous constituents in the concentrated phase. In freeze concentration two distinctive steps are involved, i.e. ice crystallization and ice separation from the concentrate. The major advantages that the freeze concentration process offers over evaporation is that it can concentrate the fruit juices without appreciable loss in taste, aroma, color and nutritive value.

(c) Evaporative concentration:

Evaporation is one of the oldest methods employed for concentrating liquid foods. Evaporation is defined as the removal of water by vaporization from the solution to produce a concentrated solution.

In this study evaporative concentration with different heating methods such as microwave heating, direct heating at open atmosphere and heating by using vacuum have been used. This section is considering these methods.

i) Open Kettle or Pan

The simplest form of evaporator consists of an open pan or kettle in which the liquid is boiled. The heat is supplied by condensation of steam in a jacket or in coils immersed in the liquid. In some cases the kettle is direct-fired. These type evaporation systems are inexpensive and simple to operate, however, due to high boiling point and open atmosphere it can cause heat damage.

ii) Vacuum evaporation

Vacuum evaporation is the process of causing the pressure in a container to fall until water's boiling point has been lowered. Most dehydration methods utilize heat to vaporize and remove water. The heat and oxygen sensitivity of many foods necessitates vacuum dehydration for high quality. Under vacuum, water can be removed at reduced temperature, and oxidative changes are minimized.

iii) Evaporation by Microwave Heating

A lot of studies about the evaporative concentration of fruit juice are available in literature. However, studies on concentrating the fruit juice by microwave energy are lacking. As an alternative heating method, the microwave heating for concentration of pomegranate juice was studied by Maskan (2006) and it was concluded that, microwave energy could be used in production of pomegranate juice concentrate successfully. Gerard and Roberts (2004) studied the microwave heating of apple mash to improve juice yield and quality. They suggested that, microwave heating has the advantage of heating the juice rapidly and uniformly, thus inactivating enzymes more quickly and minimizing browning.

1.3. Drying of Solid Materials by Hot Air

Drying of foods is a complicated process involving simultaneous coupled heat and mass transfer phenomena which occur inside the material being dried. Mass transfer is extremely important in food processing. The movement of water in particular, is most importance in the majority of unit operations. Drying, concentrating, freeze drying and most heating processes involve some movement of water. Mass transfer can either be encouraged (e.g., drying of grains, concentrating coffee) or discouraged (e.g., proper packaging to prevent moisture loss). In either case, proper control of mass transfer is essential to both the quality of the product and the economics of the process. Chemical reaction kinetics, microbial activity and physical structure in foods all depend on moisture content, or the corresponding water activity. Therefore much is to be gained by predicting or measuring moisture profiles in food during processing. Average moisture content is measured by standard oven drying and other methods, but little is known about moisture content as a function of spatial position within food material.

Several models are found in the literature, representing mass and energy transfer which take place during food drying (King, 1968; Sokhansanj and Gustafson, 1980). Usually, approximate solutions are obtained with these models by fixing geometric characteristics (slab, sphere or cylinder) and considering constant transport properties e.g., effective diffusion coefficient, conductivity.

These coupled phenomena make the analysis of the drying mechanism so complicated that extensive effort have been devoted to the development of theoretical models of heat and mass transfer to describe the drying process in materials of biological origin (Bakker-Arkema, 1986).

Drying Curves

If we assume that the temperature and relative humidity of air above the drying surface remain constant throughout the drying cycle and that all the necessary heat is supplied to the material by convection, under these constant environmental conditions the process of drying may be divided into constant rate period and one or more falling rate periods as shown in Figure 1.2.

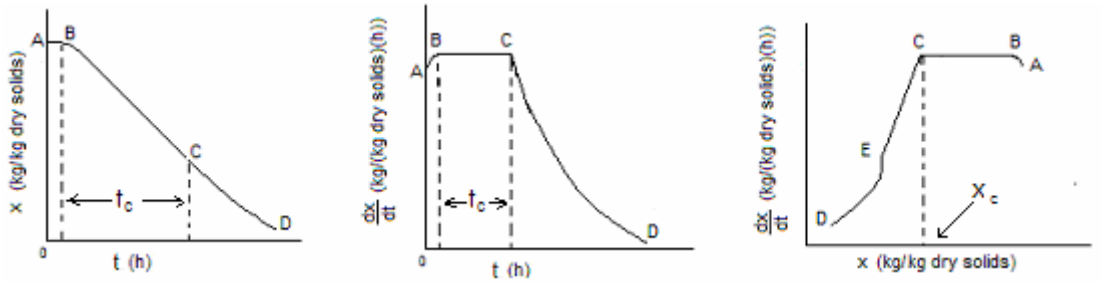

Figure 1.2 Drying curves for a wet solid in heated air at constant temperature and humidity, (Brennan et al., 1976)

A study of these curves shows that the drying cycle can be considered to consist of a number of stages.

Stage A-B This stage represents a 'settling down' period during which the solid surface conditions come into equilibrium with the drying air. It is often a negligible portion of the overall drying cycle but in some cases it may be significant.

Stage B-C This stage is known as the constant rate period of drying. During this period the surface of solid remains saturated with liquid water by virtue of the fact that movement of water within the solid to the surface takes place at a rate as great as the rate of evaporation from the surface. Drying takes place by movement of water vapour from the saturated surface through a stagnant air film into the main stream of the drying air. The rate of drying is dependent on the rate of heat transfer to the

drying surface. The rate of mass transfer balances the rate of heat transfer, and so the temperature of the drying surface remains constant.

Stage C-D As drying proceeds, a point is reached at which the rate of movement of moisture within the material to the surface is reduced to the extent that the surface begins to dry out. At this point, C, the rate of drying begins to fall and the falling rate period commences. The moisture content of the material at point C is known as the critical moisture content. From point C onwards the surface temperature begins to rise and continues to do so as drying proceeds, approaching the dry-bulb temperature of the air as the material approaches dryness. Often the falling rate period consists of two parts known as the first and second falling rate periods, *C-E* and *E-D* respectively. In the first falling rate period the surface is drying out and the drying rate falls. At point E the plane of evaporation moves in to the solid and the drying rate falls further. In the falling rate periods the rate of drying is mainly influenced by the rate of movement of moisture within the solid and the effects of external factors, in particular air velocity, are reduced, especially in the latter stage. Usually the falling rate periods represent the major proportion of the overall drying time.

The nature of the mechanism of moisture movement within the solid has received much attention in the literature. There appear to be four probable major modes of transfer:

- liquid movement caused by capillary forces
- liquid diffusion resulting from concentration gradients
- vapor diffusion due to partial pressure gradients
- diffusion in liquid layers adsorbed at solid interfaces.

In most studies carried out on drying, liquid diffusion is generally accepted to be the main mechanism during the transport of moisture to the surface to be evaporated. This important phenomenon was described analytically by Fick in 1855.

When liquid diffusion controls the internal movement of moisture and diffusion occurs only in one dimension Fick's equation can be written as

$$\frac{\partial C}{\partial t} = D_{eff} \frac{\partial^2 C}{\partial x^2} \tag{1.1}$$

where C is the concentration of the diffusing substances (g m^{-3}) at distnce x (m) along the diffusion path, and D_{eff} is the diffusion coefficient (m^2 s^{-1}).

Equation (1.1) is usually referred to as Fick's second law of diffusion, which describes the relation between the concentration, the space coordinate and the time.

If we assume that the D_{eff} is constant over the entire falling rate period, or at least over significant portion of this period and if diffusion occurs in a slab of infinite extent with the appropriate boundary conditions;

$$t = 0 \qquad C = x_o \quad 0 < x < L$$

$$x = 0 \qquad C = x_e \quad t > 0$$

$$x = L \qquad C = x_e \quad t > 0$$

The solution of Fick's second law under such conditions is

$$\frac{(x - x_e)}{(x_o - x_e)} = \frac{8}{\pi^2} \exp\left(-\pi^2 D_{eff} t / L^2\right) \tag{1.2}$$

Where t is time, x is the average moisture content (kg water per kg dry solid), x_e is the equilibrium moisture content (kg water per kg dry solid), x_o is the initial moisture content (kg water per kg dry solid), L is the thickness of the slab (m).

It should be pointed out that there is a more serious and significant deviation from the assumed situations lies in the changes of geometry and structure of drying food materials. In dehydration process other than freeze drying structural changes in the food materials occur freely, since there is a possibility for liquid flow, solute distribution and shrinkage. As a result the mass transport properties change drastically during the dehydration process. Fish (1958) reports that in scalded

potatoes the distribution coefficient for water is 10^{-8} - 10^{-7} cm^2/s when the moisture is still at a level of 15 – 20 % (dry basis), but that it drops to less than 10^{-10} when moisture is less than 1%. Similar data were reported for other materials, but in some foods diffusion coefficients do not fall off as sharply with decreasing water content as in the case of potatoes noted by Fish (1958). However, mass transport in air drying depends on drying conditions and can not be described adequately by constant diffusion coefficients.

1.4. Changes in Foodstuffs during Drying

The present demand of high-quality products in the food market requires dehydrated foods that maintain at a very high level the nutritional and organoleptical properties of the initial fresh product. A thorough understanding of the factors responsible for the decrease in the quality of the product during the dehydration process is thus of major relevance.

High temperatures and long drying times, required to remove the water from the sugar containing fruit material in conventional air drying, may cause serious damage to the flavour, color, nutrients, reduction in bulk density and rehydration capacity of the dried product (Lin et al., 1998; Drouzas et al., 1999). Some of the significant features of food dehydration processes, which affect both drying rate and final product characteristics, are considered below.

1.4.1 Movement of solubles

Water is not the only substances which changes its location within a body as drying occurs. The water in living tissue exists as a solution of scores or hundreds of constituents, some of which are small molecules like simple sugars, while others are very large and highly hydrated structures. If there is a flow of liquid water to the surface during drying, the water carries with it various soluble materials. The movement of some soluble compounds may be hindered by cell walls acting as semi-permeable membranes. Shrinkage of the material, setting up pressure in the interior of the pieces, may also contribute to the migration of solids. The net results may be building up of such soluble materials at the surface as the water evaporates (Göğüş, 1994).

1.4.2. Case hardening

It has been observed that during the drying of some fruits, meat and fish, a hard impermeable skin often forms at the surface. This results in a reduction in drying rate, and phenomenon is usually known as case hardening. 'Case hardening' is an inexact descriptive term and the phenomenon is influenced by a number of factors, including migration of soluble solids to the surface and high surface temperature toward the end of drying resulting in complex physical and chemical changes in the surface layer (Göğüş, 1994).

1.4.3. Effect upon bulk density

The large differences in moisture content that may exist within a single piece of material create shrinkage effects that depend upon the rate of drying. If a piece of a highly shrinking material is dried so slowly that its center never very much wetter than the surface, internal stresses are minimized and the material shrinks down fully onto a solid core. On the other hand, if it is dried rapidly the faces become much drier than the center and are placed under sufficient tension to give them a permanent set in nearly the original dimensions of piece; when the interior finally dries and shrinks, the internal stresses pull tissue apart. The dry piece then contains numerous crack and holes. One consequence of this difference is that the bulk density is likely to be strongly affected by the conditions of drying. (Göğüş, 1994).

1.4.4. Browning

The color of an object is another important quality factors and plays a significant role in appearance, processing, and acceptability of food materials. The color change of food materials during thermal processing takes place because of the reactions occurring inside the food material. These reactions can be pigment degradation, especially carotenoid and chlorophyll, and browning reactions such as Maillard, condensation of hexoses and amino components and oxidation of ascorbic acid, etc. (Barreiro *et al*., 1997; Lozano and Ibarz, 1997).

To optimize the process, it is important to determine the kinetic parameters (reaction order, reaction rate constant, and activation energy) for color change. The color kinetics of food products is a complex phenomenon and dependable models to

predict experimental color change, which can be used in engineering calculations, are limited. Therefore, experimental studies and application of various simplified models to represent the behavior are required.

The final values of color parameters can be used as quality indicators to evaluate deterioration due to thermal processing (Shin and Bhownik, 1995). The color measurements of food materials can be used in an indirect way to determine the color change, since they are simpler and faster than chemical analysis. Hunter color parameters (L; lightness-darkness, a; redness-greenness, b; yellowness-blueness) have previously proved valuable in describing visual color deterioration and providing useful information for quality control in fruits and vegetables (Tijskens et al., 2001; Garza et al., 1999; Gunawan and Barringer, 2000). There are other parameters derived from the Hunter L, a, and b scale: the total color change (ΔE), chroma, which indicates color saturation and is proportional to its intensity; hue angle is frequently used to specify color in food products.

1.4.5. Shrinkage

Shrinkage during drying plays an important role in determining the rate of drying and the quality of the dried product. Loss of water and heating cause stresses in the cellular structure of the food leading to change in shape and decrease in dimension (Mayor and Sereno, 2003). Due to structural differences of food materials they exhibit different shrinking behavior. If the extension of shrinkage during the drying process is controlled, quality of the dehydrated product may be improved. For this purpose, a good knowledge of shrinkage mechanism and the influence of process variables on shrinkage are needed (Mayor and Sereno, 2003).

(a) Mechanism of shrinkage

Solid and semi-solid food systems are highly heterogeneous materials that may be considered as consisting of a three-dimensional solid network or matrix holding usually large quantities of a liquid phase, in most cases an aqueous solution. Biopolymers are the common structural elements of the solid matrix. In more complex cases a composite structure is formed by the incorporation of additional structural elements (Aguilera, 1992). The particular structure of the material and the mechanical characteristics of its elements at equilibrium, define sample volume and

determine its size and shape. When water is removed from the material, a pressure unbalance is produced between the inner of the material and the external pressure, generating contracting stresses that lead to material shrinkage or collapse, changes in shape and occasionally cracking of the product. This is also the reason why drying under vacuum, as in freeze-drying, leads in general to much less shrinkage.

(b) Factors affecting the magnitude of shrinkage

i) Volume of removed water

Shrinkage of food materials increases with the volume of water removed, since the more the water removed the more contraction stresses are originated in the material. In some cases the mechanical equilibrium is reached when shrinkage of the material equals volume of removed water. In other cases, however, the volume of removed water during the final stages of drying is larger than the reduction in sample volume. This behavior can be explained by the decrease in the mobility of the solid matrix of the material at low moisture contents.

ii) Drying rate

If rapid drying rate conditions are used and intense moisture gradients through the material are observed, low moisture content of the external surface may induce a rubber–glass transition and the formation of a porous outer rigid crust or shell that fixes the volume and complicates subsequent shrinkage of the still rubbery inner part of the food. If low drying rate conditions are used, diffusion of water from the inner to the outer zone of the material happens at the same rate than evaporation from the surface, no sharp moisture gradients are formed in the material that shrinks uniformly until the last stages of drying.

iii) Mobility of the solid matrix

The mobility of the solid matrix is closely related to its physical state; high mobility corresponds to a viscoelastic behavior typical of a rubbery state while low mobility corresponds to an elastic behavior typical of a glassy state. At high moistures, when the material is in the rubbery state, shrinkage almost entirely compensates for moisture loss, and volume of the material decreases linearly with moisture content. At low moisture contents Tg increases, allowing the material to pass from rubbery to glassy state, and the rate and extension of shrinkage decreases significantly.

iv) Other processing conditions

Several authors have tried to study the influence of different process conditions in volume change of the materials during dehydration. In most cases such analysis has been done studying the effect of each single process condition like temperature (McMinn and Magee, 1997), velocity of air (Ratti, 1994 and Khraisheh, et al., 1997) or relative humidity of air (Ratti, 1994; Lang and Sokhansanj, 1993). Unfortunately the results of these works are often unclear as to the influence of those process conditions on shrinkage.

1.5. The Aim of Present Study

The recent investigation about positive effects of fruits and vegetables on human health increased the portion of fruits and vegetables in human diet. In order to contribute these demand different techniques was investigated to provide the consumption of some fruits and vegetables during the seasons of shortage**.**

Mulberry is one of the fruit which has a wide growth area in Turkey. The studies about the chemical composition of mulberry shows that it has high phenolic content and it contains a lot of minerals which are important for human body. However, in Turkey, the sufficient interest has not been shown to this valuable fruit, a lot of genotype which have an ability to give high quality fruits was obliterated to only use their board. Due to its high moisture content, limited harvesting time and sensitivity to storage its consumption is limited.

Therefore the aim of this study is to determine the characteristics of mulberry during drying and concentration, to produce a value added product with long shelf life by drying or concentrating mulberry. Particularly our purpose is to provide the best drying, concentration and storage conditions for this valuable crop. As a consequence by this way it was aimed to increase the consumption of mulberry.

CHAPTER II
MATERIAL & METHODS

2.1. Materials

Mulberries, *M. alba* (moisture content: 4 kg water/kg dry solid) which were freshly harvested from a mulberry tree were used for this study. Uniform sized fruits (length: 1.37-1.68 cm, diameter: 1.03-1.12 cm) with an average weight of 1.22 g were selected and used.

Mulberries, *M. nigra* (moisture content: 4.7 kg water/kg dry solid) at commercially mature stage were obtained from local supplier in Gaziantep. Fruits were selected according to the uniformity of shape and color. Uniform sized fruits (length: 1.80-1.92 cm, diameter: 1.27- 1.5 cm) with an average weight of 2.72 g were used.

2.2. Methods

2.2.1. Drying

(a) Hot-air Drying

The hot-air drying experiments were performed in a pilot plant tray dryer (UOP 8 tray dryer, Armfield, UK), (Figure 2.1).

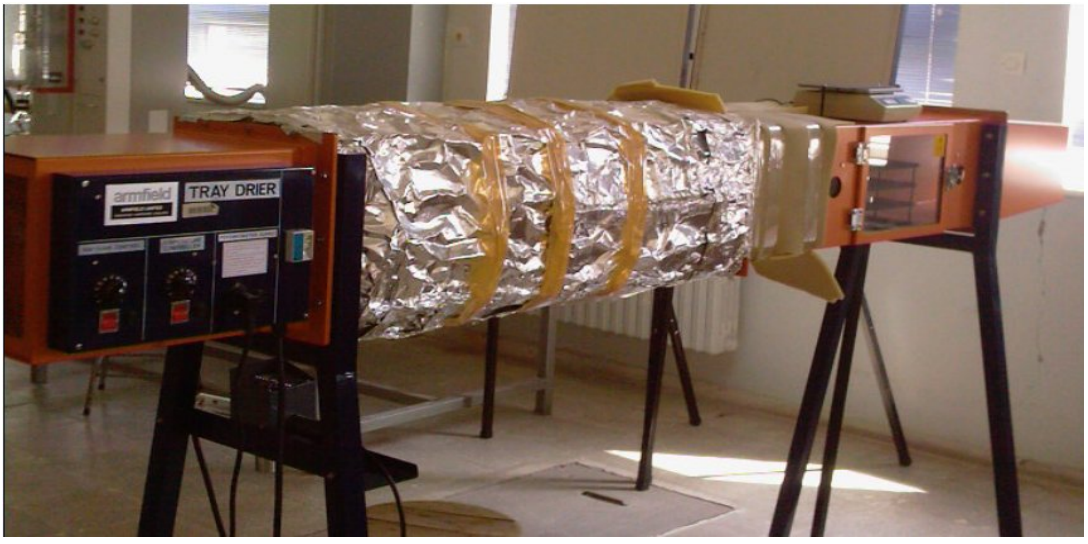

Figure 2.1 Photograph of tray drier

The dryer was operated at an air velocity of 1.2 ms^{-1}, temperatures 60, 70 and 80 °C and relative humidity (RH) 11.7, 8.6 and 5.9 % respectively. Samples were placed as a single layer of 1.09 cm thickness and about 46 g for *M. alba* and 1.4 cm thickness and about 64 g for *M. nigra* completely covering the base of the drying pan (glass petri dish, 8 cm diameter x 1.45 cm deep). Therefore, drying took place only from the top surface. Air was blown into the dryer by means of a centrifugal fan with adjustable flow rate parallel to the drying surface of the sample. Moisture loss was recorded by taking out and weighing the dish at 1 hr interval by a digital balance. Temperature and RH of heated air was measured just before contact with samples using a digital thermo-hygrometer (Accuracy at 25^{o}C ±3%RH). Mulberries were dried until equilibrium (no weight change) was reached.

(b) Microwave (MW) drying

A programmable domestic microwave oven (Arçelik ARMD 580, TURKEY) with maximum output of 700 W at 2450 MHz was used (Figure 2.2). The oven has adjustable power (wattage) and time controllers and was fitted with a turntable.

Figure 2.2 Photograph of microwave oven

The study was carried out at 350 W power levels. Samples were placed as a single layer of 1.03 cm thickness and about 47 g for *M. alba* and 1.34 cm and about 61 g for *M. nigra*, completely covering the base of the drying pan (glass petri dish, 8 cm

diameter x 1.45 cm deep). It was placed on the centre of a turntable fitted inside (bottom) the microwave cavity during treatment for even absorption of microwave energy. The presence of the turntable was necessary to achieve the optimum oven performance and to reduce the levels of reflected microwaves onto the magnetron (Khraisheh et al., 1997b). The drying was performed according to a present power and time schedule. Moisture loss was measured by taking out and weighing the dish on the digital balance periodically. When the material reached a constant weight, equilibrium moisture content was assumed to be reached. Attention was paid to ensure that the sample was not charred.

(c) Microwave finish drying

Drying was also carried out by a combination of hot air-MW technique. *M. nigra* was used in this study. The samples were dried by hot air initially for 280 minutes, then, finish dried by MW.

Mulberry sample was dried at 80°C and 1.2 m s^{-1} air velocity to 2.33 kg water/kg dry solids moisture content, the point where drying slows down. Then, sample was taken out and dried in the microwave oven. Some preliminary tests conducted on partially air dried sample resulted in burning of sample at high microwave power levels. Hence, a microwave power of 350 W and 70 W was selected for finish drying purpose.

2.2.2. Preparation of fresh Mulberry juice

The fresh samples were purchased and immediately, without storage, squeezed to juices. The edible portions of fruits were squeezed to fruit juices by a manual stainless screw squeezer. It was stored at 4 ^{o}C overnight, for settling of suspended particles, then filtered and concentrated.

2.2.3 Concentration

Three different heating/evaporation processes were used for production of mulberry juice concentrate. The juice of *Morus nigra* was concentrated to a final 60.5 ^{o}Brix from an initial ^{o}Brix of 15.5 and the juice of *Morus alba* was concentrated to a final 68 ^{o}Brix from an initial ^{o}Brix of 16 by the following processes;

(a) Concentration by microwave heating

The concentration by microwave heating was performed by using the same oven which was used in microwave drying of mulberry. Heating above or below 350 W powers level resulted in some problems such as foaming, charring of juice or lengthy of concentration time. Therefore, the concentration of *M. alba* was carried out at 350 W power levels. Similarly, heating above the 70 W resulted in some problems for *M. nigra* so the concentration of juice of *M. nigra* was carried out at 70 W power levels. A 500 mL of the juice sample was put in a beaker and replaced on the center of turntable in the microwave cavity. Samples were taken for measurement of ^{o}Brix and color periodically and replaced again.

(b) Concentration by rotary vacuum evaporator

A 500 mL of the juice of *M. alba* and *M. nigra* samples were concentrated in a laboratory rotary vacuum evaporator (RE 100 Model, Bibby Sterilin Ltd., England, has a power consumption of 65 W and requires a supply rated at 220-240 V, 50/60 Hz) (Figure 2.3), rotating at 40 rpm and 40 ^{o}C. Samples were taken from the bulk of juice periodically for ^{o}Brix and color measurements and replaced again after used.

Figure 2.3 Photograph of rotary vacuum evaporator

(c) Concentration by evaporating at atmospheric pressure

The mulberry juice was concentrated by using an electromagnetic heater (VELP Scientifica, Italy, has a power consumption of 800 W and requires a supply at 230 50 Hz) (Figure 2.4). A 500 mL of juice sample (about 6.5 cm deep) was put in a beaker (11 cm diameter x 13.5 cm deep) and replaced on the heater open to atmosphere. The sample was continuously heated and stirred during this process. Samples were taken for measurement of ^{o}Brix and color periodically and replaced again after used. Boiling point of sample was measured as 99 ^{o}C, (the boiling point of pure water is 96.7 ^{o}C in Gaziantep)

Figure 2.4 Photograph of open pan evaporation

2.2.4. Determination of moisture content

It was determined according to the method proposed by AOAC Official Methods of Analysis (Association of Official Analytic Chemists Washington D.C. (15th ed.)) that 5 grams of sample in triplicate was ground and dried at 105 ^{o}C until constant weight was reached. The loss in weight was reported as moisture.

2.2.5. Measurement of Shrinkage

For the measurement of shrinkage, similar drying experiments which are given in Section 2.2.1 were conducted separately using the same hot air and microwave drying conditions.

Changes in dimensions (D = diameter of sample; L = length of sample) were measured by a caliper. Samples were assumed in cylindrical shape and volumes (V) of fruits were calculated using the Equation 2.1.

$$V = \pi D^2 L / 4 \tag{2.1}$$

The shrinkage / volume change of the samples were expressed as bulk shrinkage ratio (S_b) of samples at any time to initial volume.

$$S_b = V / V_o \tag{2.2}$$

2.2.6. Measurement of Color

The color of mulberries and mulberry juices was measured before the experiments and at pre-specified time intervals during drying and concentration by a Hunter-Lab ColorFlex, A60-1010-615 model colorimeter (HunterLab, Reston, VA). The instrument ($45^o/0^o$ geometry, D65 optical sensor, 10^o observer) was standardized each time with a black and a white (L^* =93.76, a^* =-1.05, b^*= 0.74) tile. The $L^*a^*b^*$ color space (also referred to as CIELAB) was used to expressed the color changes. The L^* shows whiteness or brightness/darkness, a^* (redness/ greenness) and b^* (yellowness/blueness) . The total color difference ΔE^* was also calculated (Equation 2.3) from the CIE L^*, a^*, b^* values and used to describe the colour change during drying.

$$\Delta E^* = \sqrt{\left(L^*_o - L^*\right)^2 + \left(a^*_o - a^*\right)^2 + \left(b^*_o - b^*\right)^2} \tag{2.3}$$

Where subscript, *o*, refers to the color reading of fresh mulberries. Fresh mulberries were used as the reference and a larger ΔE denotes greater color change from the reference material.

Figure 2.5 Photograph of HunterLab colorimeter

2.2.7. Determination of soluble solids content

During concentration processes, the soluble solids content of the juice samples was measured by PTR 46X refractometer (Index instruments, Ltd., Ramsey, Huntingdon, Cambs, England) (Figure 2.6) at 20 ^{o}C and expressed in ^{o}Brix.

Figure 2.6 Photograph of refractometer

2.3. Statistical analysis

One-way analyses of variance (ANOVA) were conducted to determine;

- the effect of drying time on Hunter color parameters (L^*, a^*, b^*) of *M. alba* and *M. nigra* for hot air drying (T=60^{o}C)
- the effect of drying time on total color differences, (ΔE), of *M. alba*, in MW-drying
- the effect of two drying methods (Hot-air drying- MW drying) on ΔE of *M. alba*.
- the effect of drying time on shrinkage of *M. alba* for air drying (T=60^{o}C)
- the effect of drying time on shrinkage of *M. alba* for MW drying (350 W)
- the effect of different temperature 60, 70, 80 ^{o}C on extent of shrinkage of *M. alba* and *M. nigra*,
- the effect of two drying methods (Hot-air drying- MW drying) on shrinkage of *M. alba*.

The SPSS Statics 15.0, version 2.0 (2006), (SPSS Inc., Chicago) was used. Each measurement was triplicated. In order to determine which means are significantly different from each other, Duncan multiple range test method was used. Trends were considered significant when means of compared parameters differed at $P < 0.05$ significance level.

CHAPTER III
RESULTS & DISCUSSION

3.1. White Mulberry (*M. alba*)

3.1.1. Hot Air Drying Characteristics of *M. alba*

3.1.1.1. Drying curves

Most cereals, vegetables and fruits can be preserved after drying. The major objective in drying agricultural products is the reduction of the moisture content to a certain level, which allows safe storage over an extended period. Thorough understanding of the drying kinetics is the most important information needed for dryer simulation and design. The drying kinetics of food is a complex phenomenon and requires simple representations to predict the drying behavior, and for optimizing the drying parameters. The behavior of the fruits and vegetables during the drying has been the subject of several studies (e.g., Azzouz et al., 2002; Krokida et al., 2003; Cao et al., 2004; Jain and Pathare, 2004; Sacilik and Elicin, 2006; Srikiatden and Roberts, 2008).

Figure 3.1 shows the drying curves for *M. alba* dried by hot-air drying at 60, 70 and 80 ^{o}C. The moisture - time relationship exhibited typical drying behavior. It was non-linear and the decrease in moisture being higher initially as compared to the later part of drying. Constant rate drying period was not observed and the drying had occurred in the falling rate period. The same trend was reported by Maskan and Gögüs (1998) for mulberry, Togrul and Pehlivan (2002) for apricot, Kingsly et al. (2006) for ber fruit. Figure 3.1 also shows the strong effect of temperature on dehydration time. The increase in temperature reduces the time needed to reach equilibrium moisture content. The similar behavior was observed by Ochoa et al., (2001) for drying of rose hip fruit; Ochoa et al., (2007) for drying of sweet cherry and Hassini et al., (2007) for potato. There is 45% reduction in time to reach equilibrium by increasing the temperature from 60 to 80 ^{o}C.

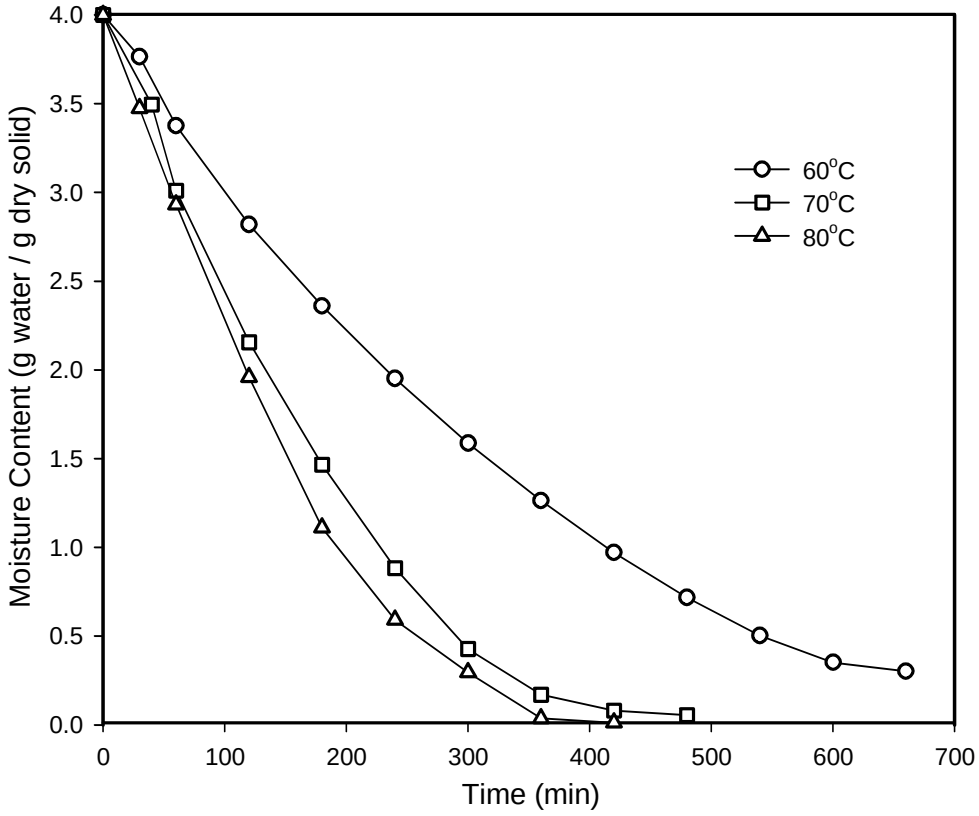

Figure 3.1 Effect of temperature on drying behavior of mulberry *(M. alba)*

3.1.1.2. Effective moisture diffusivity (D_{eff})

The effective moisture diffusivity is an important transport property in modeling of food drying processes, being a function of temperature and material moisture content. However, due to the complex food composition and physical structure, accurate estimates of this property are difficult to obtain, thus leading to the need of experimental measurements, as reported by Vagenas and Karathanos (1993).

Moisture diffusivity is identified by adjusting the experimental moisture contents to calculated ones, using an appropriate Fickian model with specific boundary conditions representing water transport in the studied food system. As Fickian models are not strictly representative of the various prevailing mechanisms of water transport in food products, the identified diffusion coefficient is considered as an apparent or effective diffusivity (D_{eff}). Analytical resolutions of Fick's second law have been largely used for D_{eff} estimation in food products. They involve simplifying assumptions, like simple material geometries without deformation, classical boundary conditions (i.e. negligible external mass transfer resistance at the interface) and constant or linear variations of D_{eff} (Crank, 1975). Nevertheless, some studies

(e.g. Zogzas and Maroulis, 1996; Teixeira and Tobinaga, 1998) pointed out that these assumptions may influence significantly the predicted D_{eff}, even using the same experimental data. It is well-known that the physical and physicochemical structures of food products also play a decisive role on water transport mechanisms and on D_{eff} (Aguilera, 2005).

In this study the following equation which expressed the diffusion of liquid in a slab shaped solid in terms of dry basis moisture content was applied to model mass transfer during the falling rate period of drying;

$$\frac{(x - x_e)}{(x_o - x_e)} = \frac{8}{\pi^2} \exp\left(-\pi^2 D_{eff}\, t / L^2\right) \tag{3.1}$$

where x is the average moisture content (kg water/kg dry solid), x_e is the equilibrium moisture content (kg water/kg dry solid), x_o is the initial moisture content (kg water/kg dry solid), L is the thickness of the slab (m) for drying from one side, D_{eff} is the effective moisture diffusivity (m^2s^{-1}) and t is the drying time (second). The effective diffusivity can be determined from the slope of the straight line obtained by plotting ln $[(x-x_e)/(x_o-x_e)]$ as a function of t of experimental data.

The non-linear shape of the drying curves (Figure 3.2) indicates variable moisture diffusivity. Each curve consists of two approximately linear falling rate periods. Similarly two falling rate periods were reported by Giovanelli et al. (2002) for tomato products. Three falling rate periods were reported by Maskan and Göğüş (1998) for mulberry, Batista et al. (2007) has found both constant and two falling rate periods for drying of chitosan.

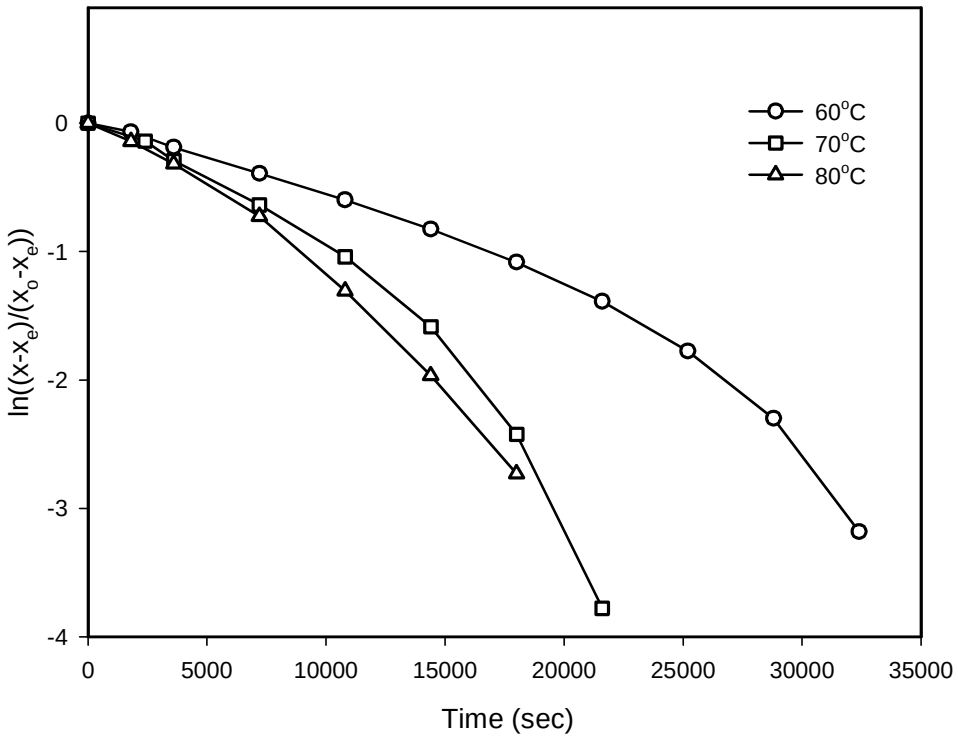

Figure 3.2 Logarithmic drying curves at various temperatures for *M. alba*

The linear regression analysis was employed to calculate the diffusion coefficients from the slopes of the straight lines. The L value (thickness of the slab) is the most effective parameter in the calculation of D_{eff} from the Equation 3.1. Therefore, the volumetric changes of mulberry samples were measured in this study and corresponding L value was used during the calculation of D_{eff} for different falling rate periods in order to show the effect of volumetric change on D_{eff}.

The diffusion coefficients were calculated for the first falling rate periods at each temperature by substituting the initial thickness of samples in Equation 3.1 and these are named as D_{eff1}. The diffusion coefficients for second falling rate periods were also calculated by using initial thickness of sample, without considering the changes on thickness, and these are named as D_{eff2} (without shrinkage). Then, to evaluate the effect of shrinkage on the diffusion coefficient, D_{eff2} were recalculated by using the thickness pertaining at the beginning of the second falling rate periods and these are called as D_{eff2} (with shrinkage). The results for both cases are stated in Table 3.1. Higher diffusion coefficients were found when the initial thicknesses were used for second falling rate periods. Whereas, when the reduced thickness were used,

diffusion coefficients of second falling rate periods reduced and closed to diffusion coefficient of first falling rate periods.

Table 3.1 Effective diffusivity values (m^2s^{-1}) for drying (*M. alba)*

Temperature (^oC)	D_{eff1}	D_{eff2} (without shrinkage)	D_{eff2} (with shrinkage)
60	$8.66x10^{-10}$	$2.48\ x10^{-9}$	$7.11\ x10^{-10}$
70	$1.06x10^{-9}$	$2.72\ x10^{-9}$	$1.01\ x10^{-9}$
80	$1.27x10^{-9}$	$2.33\ x10^{-9}$	$1.45\ x10^{-9}$

The increase in D_{eff} can be explained by the structural deformation of the sample during drying. Mulberry has a lot of buds-like structure on its surface and this structure deforms and cracks are formed during drying. These cracks increase the drying rate in the later stages of drying. This phenomenon does not usually occur; commonly, diffusivity decreases as drying proceed but, Vaccarezza et al., (1974), Mazza (1984), Hawlader et al., (1991), Göğüş (1994), Maskan and Göğüş (1998), and Giovanelli et al., (2002) also found similar trend for D_{eff}. They suggested the followings about this situation. Mazza (1984) stated that, in the low moisture range, the drying is so slow that the cooling effect of evaporation is insignificant and drying material assumes the dry bulb temperature of the air. In this phase of drying, the rate of moisture movement to the surface of the material increases with temperature. Therefore, even at very low moisture content, the drying rate is appreciably greater. Another reason of the greater diffusivity at low moisture content may be due to the cell wall destruction, because wet bulb temperature of the samples approaches the dry bulb temperature and therefore decreases the resistance to the moisture diffusion within the sample. This means that heating leads to changes in the physical properties of tissue, among them destroying the semipermeability of the cell membranes (Vaccarezza et al., 1974). Göğüş (1994) has also found the similar results performed on some model systems, and suggested that if the drying mechanism is of liquid diffusion and the driving force is the concentration gradient, the diffusion coefficient may be constant throughout the drying process, but the shorter diffusion distance caused by shrinkage increases the rate of drying as drying proceeds.

Hawlader et al. (1991) also pointed out that, the increasing D_{eff} was ascribed to the shrinkage of samples during drying and, hence, to a reduction of thickness, resulting in faster water removal.

Table 3.1 shows the effect of temperature on D_{eff}. Effect of temperature on effective moisture diffusivity is generally expressed by using an Arrhenius-type relationship given as;

$$D_{eff} = D_o \exp\left(-\frac{E_a}{T_{abs} R}\right) \tag{3.2}$$

where *Ea* is the activation energy of the moisture diffusion in kJ mol^{-1}; D_o is the diffusivity value (m^2s^{-1}) for a infinite moisture content; *R* represents the universal gas constant 8.3145x10^{-3} kJ$mol^{-1}K^{-1}$and T_{abs} is the absolute drying air temperature in K.

The activation energy was calculated by plotting the ln (D_{eff}) versus the reciprocal of the absolute temperature ($1/T_{abs}$), and presented in Figure 3.3. The activation energy for diffusion was calculated as 19.02 kJmol^{-1}. The E_a value lies within the range of 15–40 kJ mol^{-1} for various foods (Rizvi, 1986). It can be compared with 21.2 kJmol^{-1} obtained for mulberry by Maskan and Gögüs (1998), 19.950-22.624 kJmol^{-1} obtained for red delicious apple by Kaya et al., (2007).

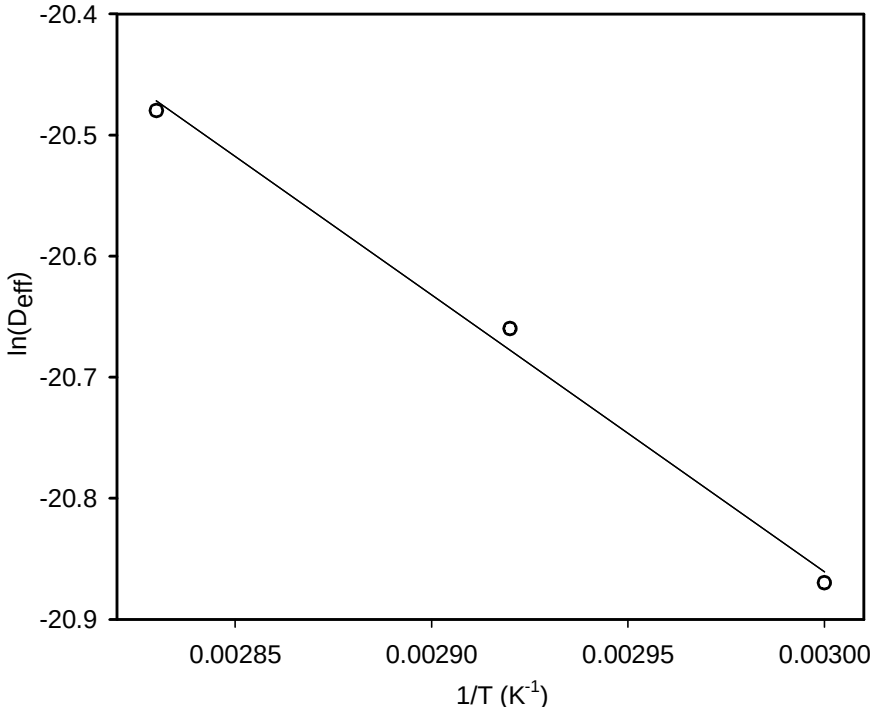

Figure 3.3. Relationship between diffusivity and reciprocal of absolute temperature

3.1.1.3. Shrinkage

Volumetric shrinkage occurs due to the evaporation of moisture from the fruits. Such shrinkage affects the physical attributes and the transport properties of the solids. The volume change during drying is not an easily predictable function. Visual examination of the samples throughout the drying process reveals that the shrinkage is not perfectly homogeneous. In the initial stage of drying, the samples keep the original geometry, i.e., cell structure appears to be intact. As drying proceeds, however, the shrinkage is accompanied by particle deformation. Changes in volume or the volumetric ratio are the most studied aspect in representing the shrinkage coefficient. This phenomenon was studied and mathematical models were developed for shrinkage of some fruits and vegetables by Göğüş (1994), McMinn and Magee (1997), Raghavan and Venkatachalapathy (1999), Maskan (2001), Mayor and Sereno (2004), Kingsly et al. (2007).

Ochoa et al. (2002, 2007) studied the shrinkage of sweet cherry fruit and rose hip fruit during convective dehydration and they found linear relationship between volumetric shrinkage and moisture content. Ratti (1994) studied the shrinkage of potatoes, apples and carrots during convective dehydration and suggested a simple model in which volumetric ratio is presented by one or two line segments. The volumetric shrinkage of ber fruit during sun drying was studied by Kingsly et al. (2007) and exponential relation was indicated between volume change and moisture content. Khraisheh et al. (2004) studied the structural changes in potatoes during microwave and convective drying. They indicated that in volumetric shrinkage a linear relationship with moisture content were observed with convective drying and two shrinkage periods were observed with microwave drying. Raghavan and Venkatachalapathy (1999) found direct relationship between shrinkage and moisture content in their studies about strawberry. McMinn and Magee (1997) also studied on volumetric shrinkage of potatoes with drying and found linear correlation with moisture content. Suziki et al. (1976) studied on the shrinkage in dehydration of root vegetables such as carrots, potatoes, sweet potatoes and radishes.

In this study, volumetric change was calculated by direct measurement of sample's dimension by using Equation 2.1. Figure 3.4 indicates relation of moisture content and volumetric shrinkage at 60, 70 and 80^{o}C. In all temperature treatments shrinkage

initially and gradually was leveling off towards the end of drying. The similar behavior was observed by Ratti (1994) on drying of potatoes, apples and carrots, Maskan (2001) on drying of kiwifruits. Ratti (1994) indicated that for some food stuff such as carrot and pears, the v/v_o versus x/x_o function is linear in the whole range of water content, for other products such as potatoes, garlic and apples the graph can be represented by two straight line segments. The same author also suggested that in the latter case there is change in the dehydration mechanism after the critical moisture content $(x/x_o)_c$, owing to modifications in the proportion of external and internal resistance to water transfer. If mechanism of transfer of water changes, it possibly changes the velocity of shrinkage phenomena, so it is important to check the experimental value of critical moisture content. The $(x/x_o)_c$ and $(v/v_o)_c$ were obtained from the interception point of these straight line segments for each temperature. The fitting proposed were:

$$\frac{v}{v_o} = a_1 + b_1 \frac{x}{x_o}, \quad \left(\frac{x}{x_o}\right)_c \leq \frac{x}{x_o} \leq 1 \tag{3.3}$$

$$\frac{v}{v_o} = a_2 + b_2 \frac{x}{x_o}, \quad 0 \leq \frac{x}{x_o} \leq \left(\frac{x}{x_o}\right)_c \tag{3.4}$$

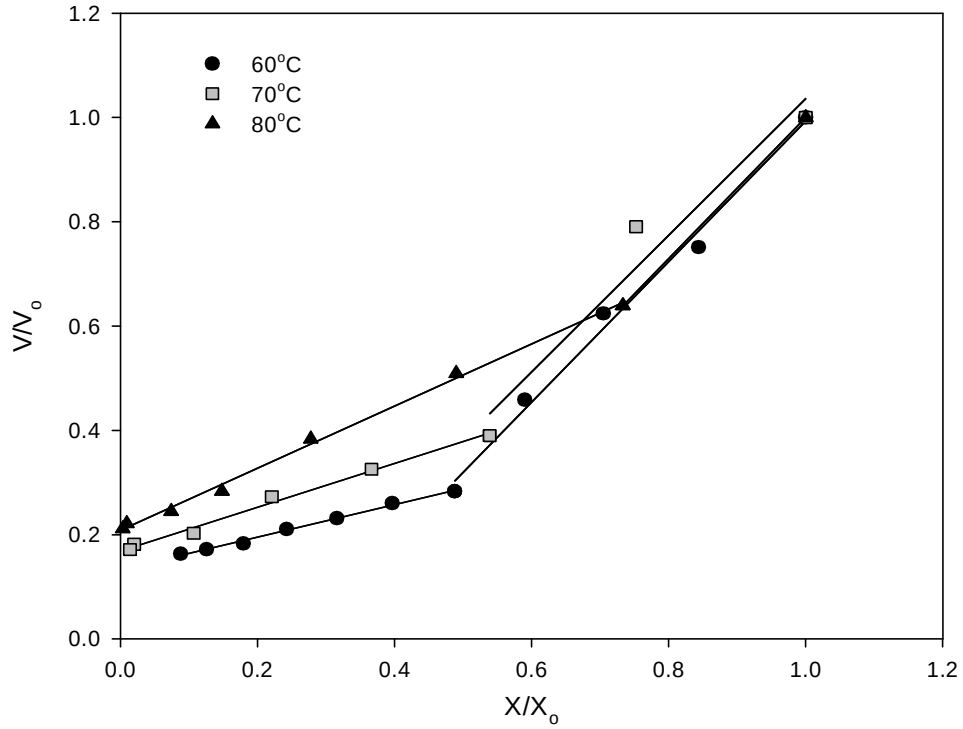

Figure 3.4. Relation of moisture content and volumetric shrinkage (*M. alba*)

The characteristic parameters for Equation 3.3 and Equation 3.4 indicated in Table 3.2 for each temperature. The magnitude of parameter *b* indicates that the degree of shrinkage is greater in the initial stages than that of second stage. Khraisheh et al. (2004) found similar results and suggested that, in the lower moisture content range the reduction of shrinkage rate is due to the fact that the shrinkage in this range due to the loss of water, air-filled pores are being formed, i.e., puffing occurs to counter the shrinkage effect.

Table 3.2 Characteristic parameters for Equation 3.3 and Equation 3.4 (*M. alba*).

Temperature (^{o}C)	a_1	b_1	a_2	b_2
60	-0.35	1.35	0.13	0.31
70	-0.27	1.31	0.17	0.42
80	-0.35	1.35	0.20	0.59

In Table 3.3 critical moisture content $(x/x_o)_c$ and corresponding volumetric shrinkage ratio $(v/v_o)_c$, and final volumetric shrinkage ratio $(v/v_o)_f$ are presented for 60, 70 and 80°C. It can be seen from the table, that the increasing the temperature shifts the critical moisture content and the corresponding volumetric shrinkage ratio to higher values. Table 3.3 also shows that the extension of shrinkage at lower temperature is bigger than the higher temperature. Gögüs (1994) explained that the larger differences in moisture content that may exist within a single piece of material create shrinkage effects that are dependent upon the rate of drying. If a piece of highly shrinkable material is dried so slowly that its center is never very much wetter than the surface, the internal stresses are minimized and the material shrinks down fully onto a solid core. On the other hand, if it is dried rapidly the walls become drier than the centre and are placed under sufficient tension to give them a permanent set with similar dimensions to the original piece. In addition to that when percent volume reduction was calculated, the 84%, 83% and 79% reduction were found for 60, 70 and 80 °C, respectively. This also shows that drying at 60 ^{o}C promoted more shrinkage, which was explained by the fact that a long drying time gives more time for the product to shrink (Ratti, 1994). But the results of statistical analysis of data showed that, there are no significant differences between 60 and 70 ^{o}C and 70 and 80^{o}C but the increase from 60 to 80 ^{o}C can cause significant difference ($P>0.05$) (Appendices A.).

Table 3.3 Effect of temperature on critical moisture content $(x/x_o)_c$ and corresponding volumetric shrinkage ratio $(v/v_o)_c$ and final volumetric shrinkage ratio$(v/v_o)_f$

Temperature (^oC)	$(x/x_o)_c$	$(v/v_o)_c$	$(v/v_o)_f$
60	1.88	0.28	0.16
70	1.98	0.38	0.17
80	2.96	0.65	0.21

3.1.1.4. Changes in CIELAB color parameters (L^*, a^* and b^*) and Color kinetics

The Commission Internationale de l'Eclairage (CIE), CIELAB color parameters (L^*; lightness-darkness, a^*; redness-greenness, b^*; yellowness-blueness) have previously proved valuable in describing visual color deterioration and providing useful information for quality control in fruits and vegetables (Garza et al., 1999; Gunawan and Barringer 2000; Tijskens et al., 2001). The study of the color change behavior of foods during drying has recently been subject of interest for various investigators: for example, pear puree (Ibarz et al. (1999), kiwifruit (Maskan 2001), pestil (Maskan 2002), mulberry fruit extract (Joo Suh 2003), pineapple (Rattanathanalerk 2005), and pomegranate juice concentrate (Maskan 2006), potato (Bondaruk et al., 2007).

Therefore the changes of CIELAB color parameters and kinetic modeling for these parameters were also studied for mulberry. The results of color parameters obtained for three different temperatures are presented in Figures 3.5, 3.6 and 3.7 for L^*, $a^*$and b^*, respectively. The ANOVA results showed that there is significant change in all color parameters with drying ($P<0.05$) (Appendices B., C., and D.). The L^*-value decreased with drying time and remains constant after a certain point. Since L* is a measure of the color in the light-dark axis, this falling value indicates that the samples lose their brightness and were turning darker. It can be explained by nonenzymic browning reactions, such as maillard browning and caramelization. Ibarz et al., (1999) have found similar result in their studies for pear puree.

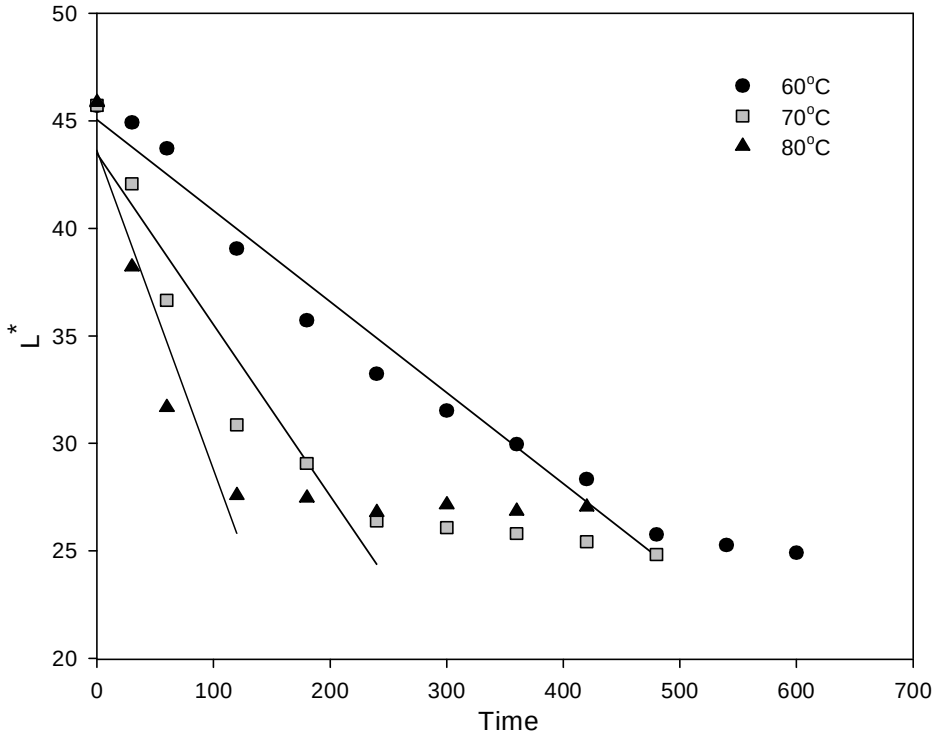

Figure 3.5 Changes of L^* value as a function of time during drying

Both a^* and b^* values exhibited change up to a certain point, then remain constant as observed in L^* value. When a^* value exhibited increasing trend, b^* value followed decreasing trend. Therefore sample lost their yellowness and become redder when dried. A similar behavior was found by Maskan (2001), Dadali et al. (2007) and Barreiro et al (1997). This may be explained by decomposition of carotenoids pigments and formation of brown pigments.

The total color differences, ΔE^* which were calculated from the measured L^*, a^* and b^* values was indicated in Figure 3.8. It shows the overall effects of drying on color, so it can be said that the drying cause significant change on color of samples.

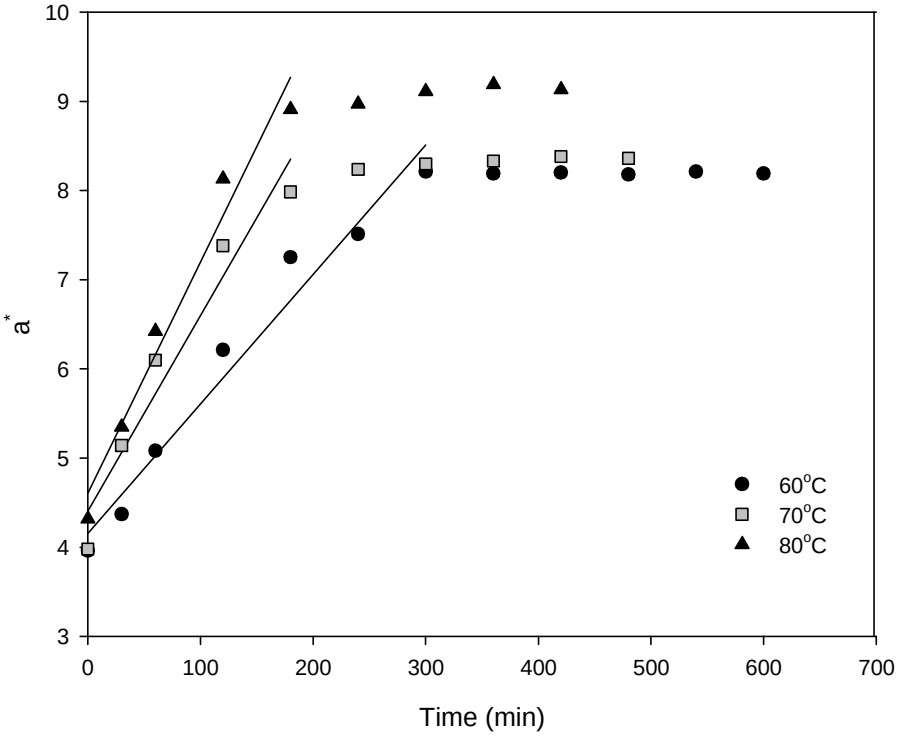

Figure 3.6 Changes of a^* value as a function of time during drying

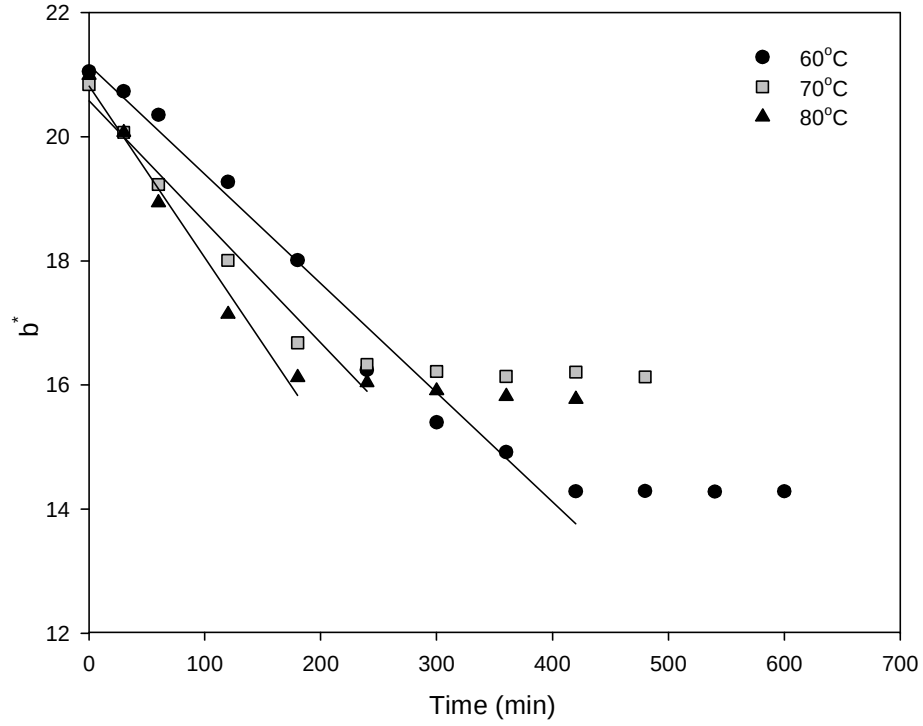

Figure 3.7 Changes of b^* value as a function of time during drying

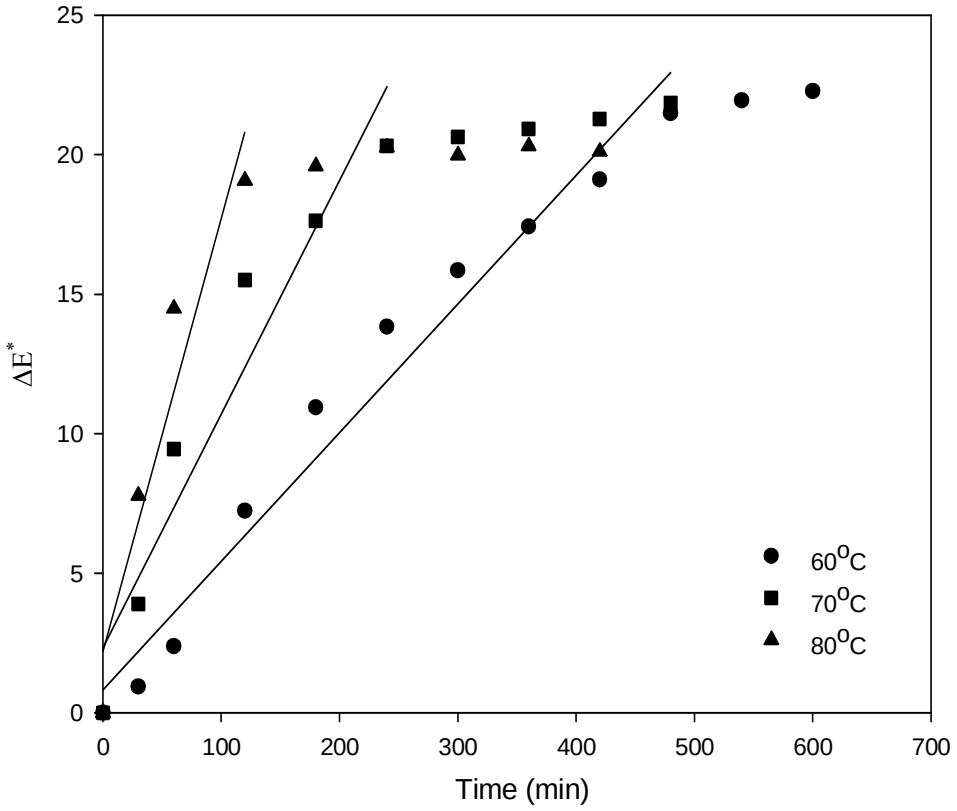

Figure 3.8 Change in total color differences of mulberry during drying time

For the design process, also, the kinetic modeling for color change is necessary to derive basic kinetic information for a system in order to describe the reaction rate as a function of experimental variables and, hence, to predict changes in a particular food during processing and storage (Van Boekel, 1996). There are numerous references on the kinetics of colour of food materials in the literature. The majority of these works report zero-order (Equation 3.5) or first-order (Equation 3.6) degradation reaction kinetics;

$$C = C_o \pm kt \tag{3.5}$$

$$C = C_o \exp(\pm kt) \tag{3.6}$$

where, C represents any quality parameters, C_o initial value of quality parameters, k is kinetic rate constant, (+) and (-) indicate formation and degradation of any quality parameter, respectively.

The kinetic rate constant for color parameters (L^*, a^*, b^* and ΔE^*) were calculated by using the linear section of their related curves. In this study the linear sections of curves were concerned, because these sections are the rate determining sections. Table 3.4 shows the rate constants of color parameters for each temperature. It can also be seen from this table that the increasing the temperature causes to increase in kinetic rate constant.

Table 3.4 Linear regression analysis results of color parameters from zero order reaction kinetics.

Temperature	Parameters	k	r^2
60 ^oC	L^*	0.0423	0.97
	a^*	0.0145	0.97
	b^*	0.0176	0.98
	ΔE^*	0.0460	0.97
70 ^oC	L^*	0.0796	0.92
	a^*	0.0219	0.95
	b^*	0.0195	0.97
	ΔE^*	0.0840	0.92
80 ^oC	L^*	0.1484	0.90
	a^*	0.0276	0.97
	b^*	0.0260	0.98
	ΔE^*	0.1550	0.92

3.1.2. Drying Characteristics of *M. alba* for Microwave Drying

3.1.2.1. Drying curve

As it has been mentioned in Section 1.2.2, hot-air drying has been to date the most common drying method employed for food materials. However, this method has many disadvantages, including poor quality of dried products, low energy efficiency and a long drying time. The desire to eliminate this problem, to prevent significant quality deterioration, as well as to achieve fast and effective thermal processing has resulted in the increasing use of microwaves for food drying. It enables to shorten dehydration time and to control undesirable biological transformations. The different application of microwave drying technique was studied by Ohlsson (1998) for apples and mushrooms; Maskan (2000) for bananas; Bondaruk et al. (2007) for potato cubes; Contreras et al. (2008) for apple and strawberry.

Figure 3.9 shows the drying curve for *M. alba* dried by microwave drying at 350W. The moisture-time relationship exhibited typical drying behavior as seen in Section 3.1.1.1. Total drying times to reach equilibrium moisture content were 600, 420 and 360 min for 60, 70 and 80 ^{o}C, respectively for convective drying. These results indicated that the drying time to reach the equilibrium of the sample was shortened from these ranges to 30 min when the mulberry was dried by microwave. This difference can be explained by the energy supplied during microwave heating, it reaches all parts of dried material at the same time and heat is generated inside the sample, creating a large vapour pressure differential between the centre and the surface of products so the mass transfer within the sample is rapid during microwave heating (Lin et al., 1998).

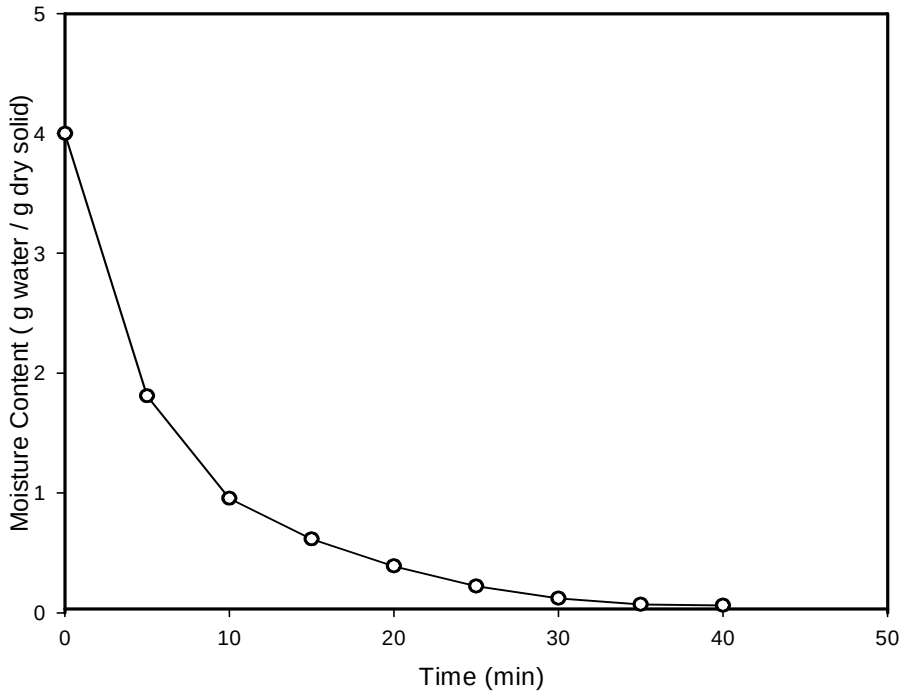

Figure 3.9. Drying curve formicrowave drying of mulberry (*M. alba*) at 350W

The reduction in drying time with microwave energy was also observed in the study of different authors on drying of fruits and vegetables (e.g. Maskan, 2000; Bondaruk et al., 2007). These authors explained that the shorter drying time under microwave heating conditions could be due to the additional energy input, rapid heat penetration by microwave and forced expulsion of gases.

3.1.2.2. Shrinkage

In this section volumetric change of *M. alba* with microwave drying was studied and it was calculated as described in Section 3.1.1.3 by using Equation 2.1. Raghavan and Venkatachalapathy (1999), Maskan (2001) and Khraisheh et al. (2004) also studied the volumetric change of some fruits and vegetables during microwave drying. Some of them found a linear relationship between moisture content and volumetric shrinkage; some others indicated two line segments.

Figure 3.10 indicates relation of moisture content and volumetric shrinkage at 350W for *M. alba*. The experimental data shows a linear behavior between shrinkage and moisture content, which suggests that the shrinkage is predominantly due to the volume of removed water. A linear relation between shrinkage and water content was fitted to the experimental data:

$$\frac{v}{v_o} = a\frac{x}{x_o} + b \tag{3.7}$$

Where x_o is the initial moisture content (kg water / kg dry solid). The constants a and b, were calculated as 0.844 and 0.1908 (r^2=0.98), respectively. The linear shrinkage behavior of food materials were also reported by Krokida and Maroulis (1997) and Raghavan and Venkatachalapathy (1999) for microwave drying. In contrast to that, Maskan (2001) and Khraisheh et al. (2004) reported two linear segments for relationship between moisture content and shrinkage in their microwave drying studies.

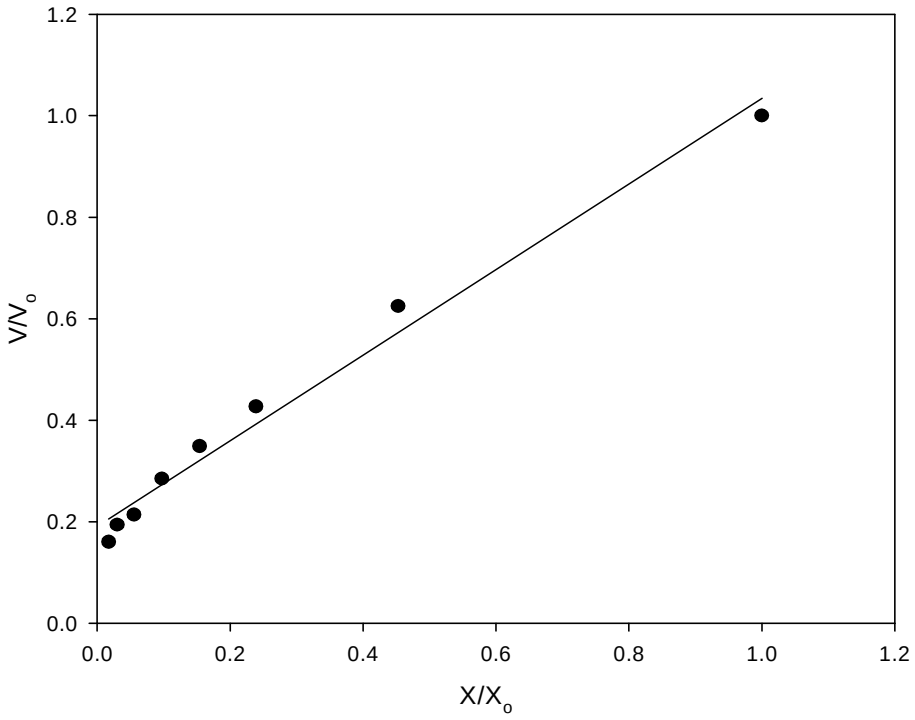

Figure 3.10. Relation of moisture content and volumetric shrinkage at 350W

The initial volume of *M. alba* which was used in microwave treatment was 1.34 cm^3 and after the drying it was calculated as 0.22 cm^3. These data show that there is an 84% volume reduction. When both methods of hot air drying and microwave were compared there is almost no difference between hot air (60^oC) and microwave drying (350W) methods with respect to extent of shrinkage. Differences between drying methods were also not significant ($P>0.05$) statistically with respect to the percent volume reduction of samples (Appendices E.). Krokida and Maroulis (1997) found that shrinkage does not vary significantly for drying methods except freeze drying. However Khraisheh et al. (2004) indicated that samples dried in a microwave field exhibit less shrinkage than those undergoing classical air drying.

The calculated *a*-values (slope of shrikage curves) were between the 0.13-0.35 for hot air drying but it was 0.84 for MW drying. Therefore, it could be concluded that the microwave (MW) energy causes rapid shrinkage. Maskan (2001) has also found rapid and higher shrinkage of samples when dried by microwave and suggested that it is because of extensive heat generation, accelerating removal of water from the tissues in the sample by microwave.

3.1.2.3. Changes in CIELAB color parameters (L^*, a^* and b^*) and color kinetics

The changes of CIELAB color parameters were also studied for microwave drying. The ANOVA results showed that microwave drying cause significant color change with drying ($P<0.05$) (Appendices F.). The changes in L*, a*, b* and ΔE^* are indicated in Figures 3.11, 3.12, 3.13 and 3.14, respectively. All parameters showed similar trend with classical air drying. When L* and b* follow the decreasing trend up to certain point, a* and ΔE* exhibited increasing trend and remain constant after 15 min of drying.

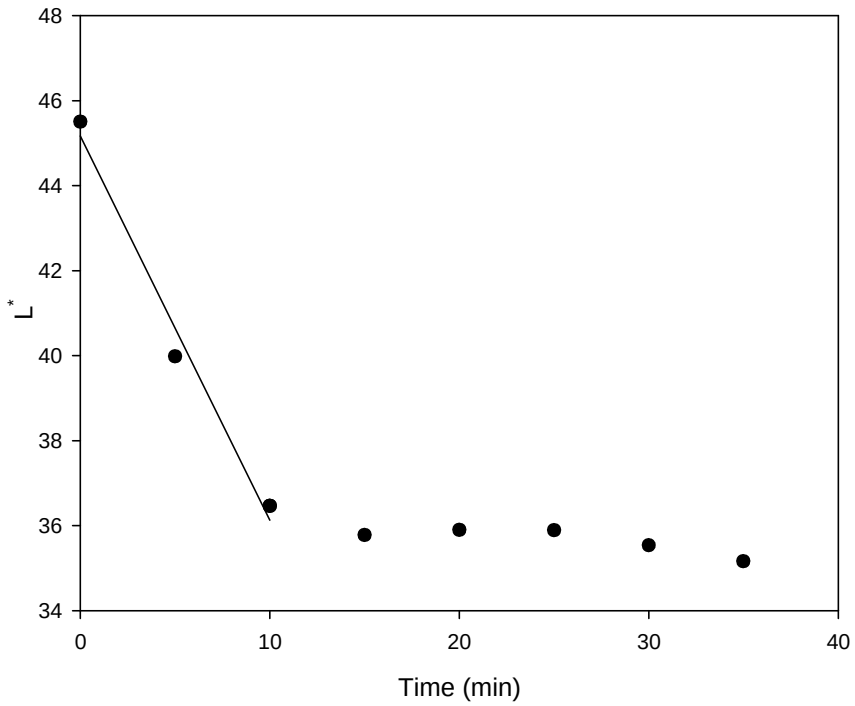

Figure 3.11 Changes of L^* value of mulberry (*M. alba*) during drying time at 350W

When the mulberry (*M. alba*) samples dried by microwave, they lost their yellowness and became brawn. Similar observations were reported by Ibarz et al. (1999), and Maskan (2001). This may be due to decomposition of carotenoid pigments and formation of brown pigments.

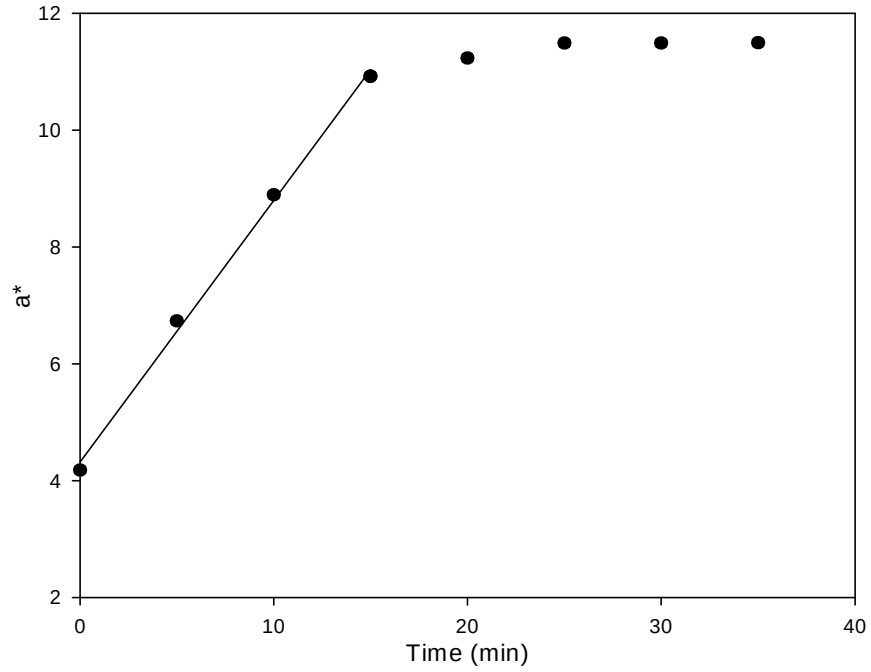

Figure 3.12 Changes of a^* value of mulberry (*M. alba*) during drying time at 350W

When the color change behavior of sample was compared for hot (60^{o}C) and microwave drying (350W) methods, the ANOVA results (Appendices G.) revealed that there is a significant differences between these methods with respect to ΔE^* ($P<0.05$).

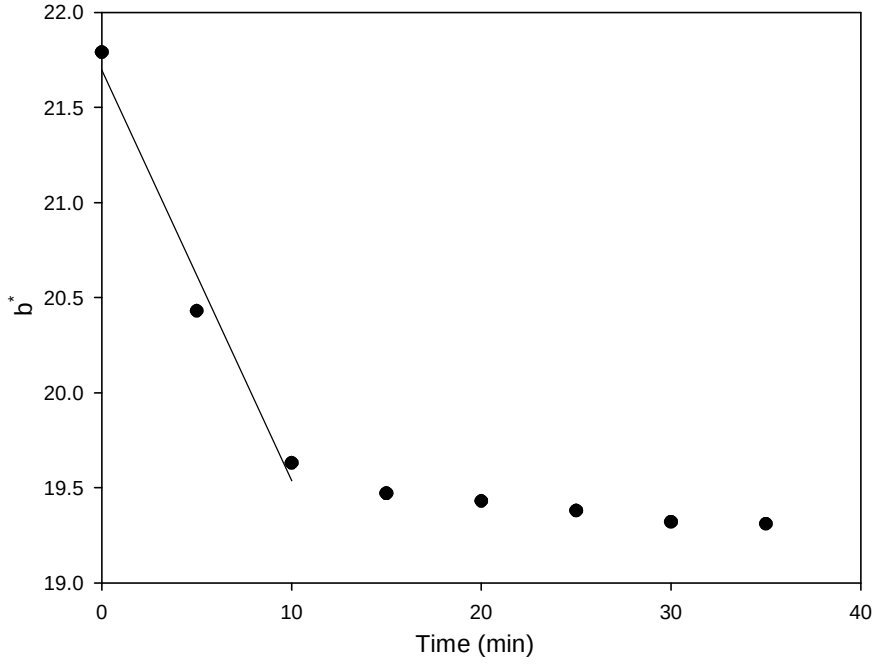

Figure 3.13 Changes of b^* value of mulberry (*M. alba*) during drying time at 350W

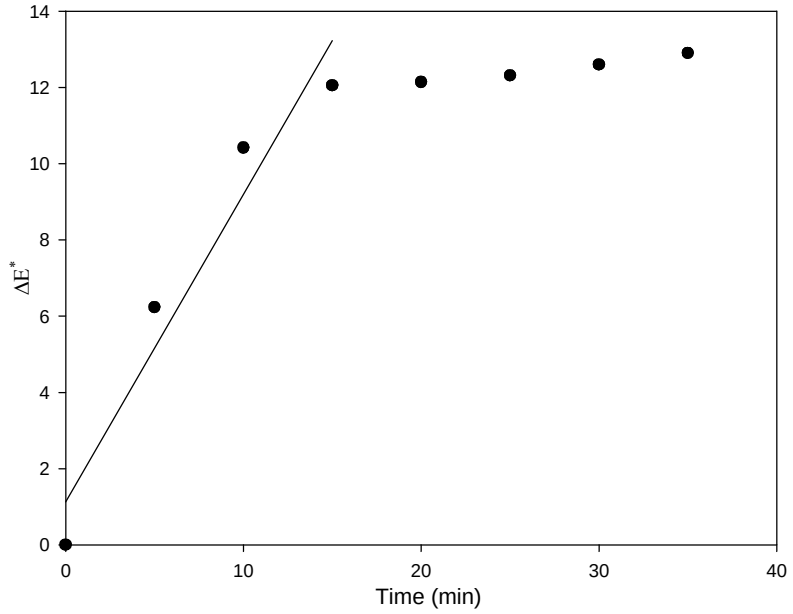

Figure 3.14. Change in ΔE^* value of mulberry (*M. alba*) during drying time at 350W

The linear relation, $C = C_o \pm kt$ (zero-order degradation reaction kinetics), between color and time was fitted to the linear sections of experimental data. The rate constants of color parameters were calculated and indicated in Table 3.5. When these results was compared with those of air drying (Table 3.4), *k* values were higher for MW drying. So, it can be concluded that the deterioration is faster for microwave drying.

Table 3.5 Linear regression analysis results of color parameters from zero order reaction kinetics.

Power	Parameters	k	r^2
350W	L^*	0.904	0.98
	a^*	0.448	0.99
	b^*	0.216	0.95
	ΔE^*	0.806	0.94

3.1.3. Concentration of mulberry (*M. alba*) juice extract

In concentration process the vapor from a boiling solution is removed and a more concentrated solution is obtained by evaporation. Fruit juices are usually concentrated by multi-stage vacuum evaporation. This process results in a loss of

fresh juice flavors, color degradation and a ‘‘cooked’’ taste due to the thermal effects. To overcome these problems many efforts have been done and different concentration methods such as freeze concentration, sublimation concentration and membranes (ultrafiltration and reverse osmosis) have been developed and membrane concentration suggested as most promising alternative method. However, main disadvantages of this method are its high operating cost and inability to reach the concentration of standard products produced by evaporation because of high pressure limitation (Jiao et al., 2004). Therefore, as a different way, microwave energy was also tested in the production of mulberry juice in this study. It has the advantage of heating the juice rapidly and uniformly, thus inactivating enzymes more quickly and minimizing browning (Gerard and Roberts, 2004).

3.1.3.1. Change in total soluble solids content (^{o}Brix)

Figure 3.15 shows soluble solids content (^{o}Brix) of mulberry juice against time of concentration process for three evaporation techniques. The time required to obtain the final concentrations of 68.6, 65.7 and 72.8 ^{o}Brix was 68, 100 and 150 min for microwave, rotary vacuum and atmospheric heating processes, respectively.

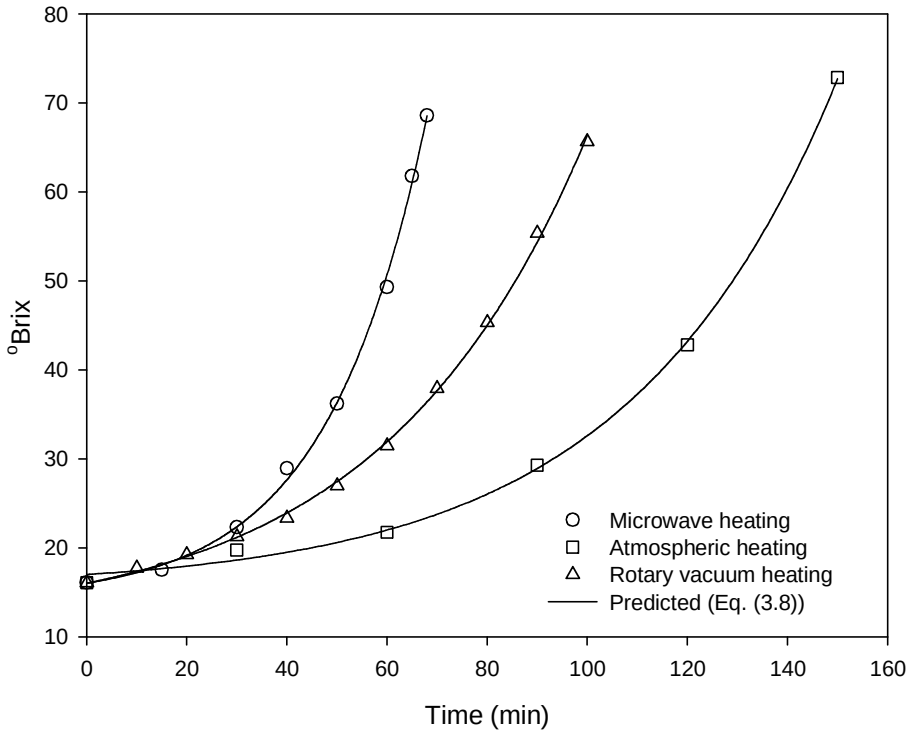

Figure 3.15. Changes in mulberry (*M. alba*) juice concentration (^{o}Brix) produced by various concentration processes

The change in concentration (^{o}Brix) of juice samples against time was fitted to a three-parameter exponential equation (Equation 3.8).

$$B = B_o + B_1 * \exp(k * t) \quad (3.8)$$

Where, B and B_o are the soluble solids content of samples at any time t and initial concentration (^{o}Brix), respectively; B_1 is a constant and k is the evaporation rate constant (min^{-1}).

Fitted parameters of Equation 3.8 and corresponding correlation coefficients (r^2) values for the change in total soluble solid content (^{o}Brix) during concentration process were reported in Table 3.6. The r^2 values were greater than 0.99 in all cases. The evaporation rate constant, k, for microwave heating process was 5.8 and 8.4 times greater than rotary vacuum and atmospheric heating concentration processes, respectively. This is because of the rapid heating and, hence, water evaporating effect of microwave process.

Table 3.6 Kinetics parameters of Equation 3.8 for changes in concentration of mulberry juice during concentration process

Concentration Processes	B_o	B_1	k	r^2
Microwave	14.210	1.835	0.0498	0.998
Rotary vacuum	11.280	4.757	0.0245	0.999
Atmospheric	15.470	1.539	0.0241	0.998

3.1.3.2. Changes in CIELAB color parameters (L*, a* and b*)

In this section the color change of mulberry fruit extract during the concentration with the different methods were studied. It was observed that all concentration processes changed the color parameters (L*, a*, b*values) of mulberry juice (Figure 3.16, 3.17 and 3.18) and the products turned reddish brown.

Lightness (L*) value decreased with treatment time (Figure 3.16). Decrease in L* value was 55.27%, 71.78% and 67.27% for microwave, atmospheric and rotary vacuum heating concentration processes, respectively. As it is seen, the intense of

lose of L^* value is higher in samples treated with atmospheric heating process. Furthermore, MW-heating causes rapid but less deterioration on color.

The Figures 3.17 and 3.18 show the change of a^* and b^* value, respectively. Both of them followed increasing trend, which means that the redness and yellowness of concentrate increase with concentration time.

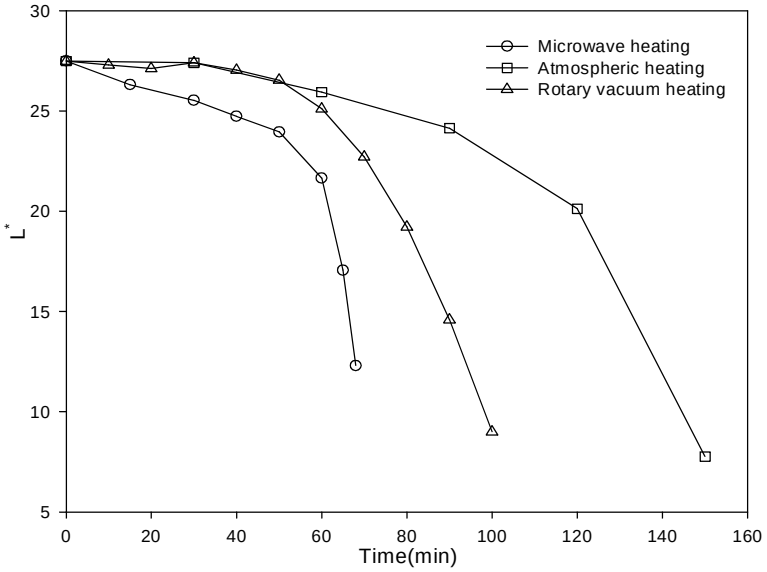

Figure 3.16. Variation of L^* value of mulberry juice (M. *alba*) produced by various concentration processes

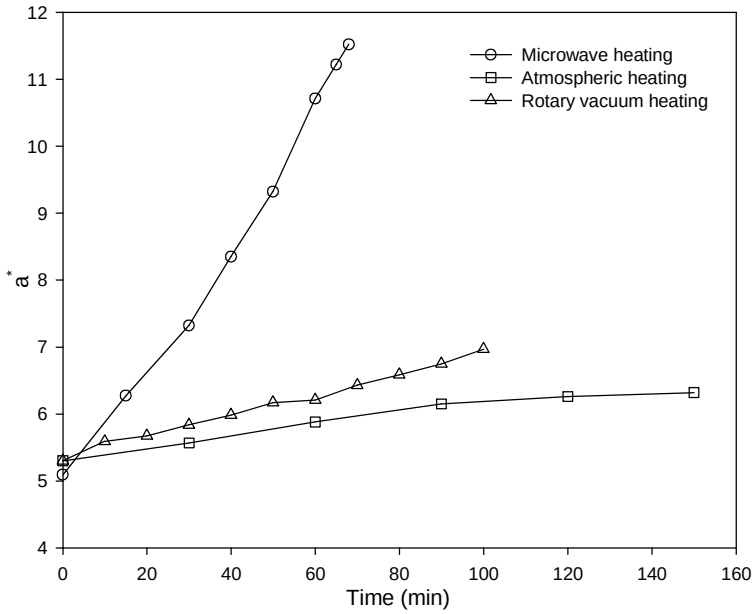

Figure 3.17 Variation of a^* value of mulberry juice (M. *alba*) produced by various concentration processes

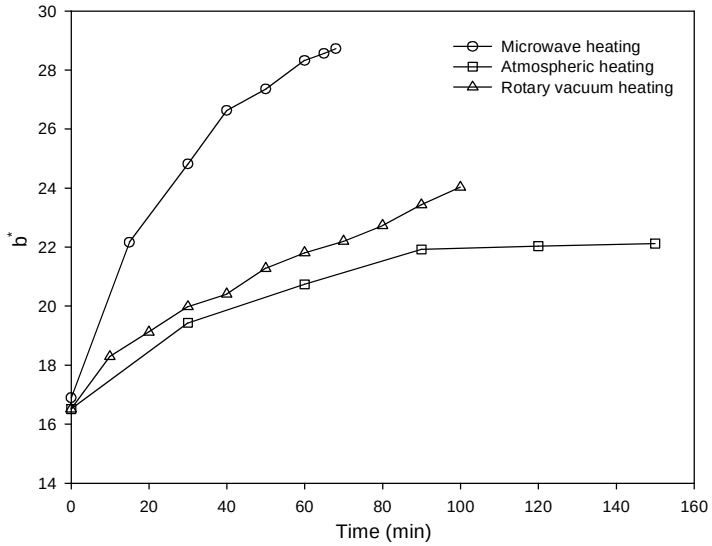

Figure 3.18. Variation of b^* value of mulberry juice (M. *alba*) produced by various concentration processes relations of moisture content and time

3.2. Black Mulberry (*M. nigra*)

In this section, the drying characteristics of *M. nigra* during the hot air, microwave and microwave finish drying were discussed. Moisture loss, effective moisture diffusivity, shrinkage and color change of *M. nigra* were studied during these processes. Also, mulberry (*M. nigra*) juice extract was concentrated with different heat treatments (microwave, atmospheric and rotary vacuum heating) and the changes of total soluble solid content (^{o}Brix) and color during these processes were investigated.

3.2.1. Hot Air Drying Characteristics of *Morus nigra*

3.2.1.1. Drying curves

Figure 3.19 shows the time-moisture relationship of *M. nigra* at different temperatures. *M. nigra* exhibited a similar drying behavior to that of *M. alba*. The time needed to reach equilibrium moisture content decreased with increase in temperature. This reduction was 46.7% with increase in temperature from 60 to 80^{o}C. The equilibrium moisture content (x_e) is 0.35 for *M. alba* at 60^{o}C (RH=11.7%, V=1.2 ms^{-1}) and time to reach this point is 600 min, but x_e is 1.78 and time to reach to this point is 900 min for *M. nigra* at the same drying conditions. As it is given, when the moisture content of *M. alba* could be reduce up to 0.35 (kg water/kg dry solid), *M. nigra* reduced to 1.78. This can be related with the structural and compositional differences between them. For example, the initial moisture content of

M. nigra. was 82.4% and initial diamensions were 1.85 cm (length) and 1.37 cm (diameter), these values were 80.0% (m.c.), 1.51 cm (length) and 1.12 cm (diameter) for *M. alba*.

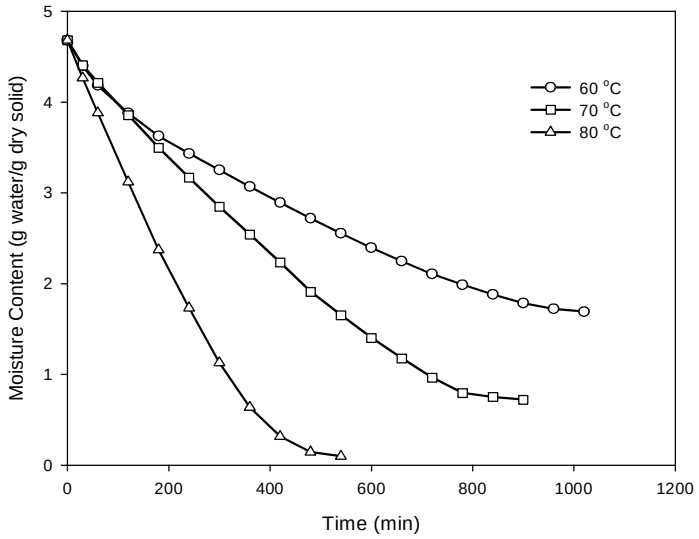

Figure 3.19 Effect of temperature on drying behavior of mulberry *(M. nigra).*

3.2.1.2. Effective moisture diffusivity (D_{eff})

The procedures which were used in the determination of effective moisture diffusivity of *M. alba* (Section 3.1.1.2) was also followed for determination of D_{eff} for *M. nigra*. Figure 3.20 shows the plot of $\ln[(x-x_e)/(x_o-x_e)]$ versus t for the experimental data obtained at various temperature. The non-linear shape of the drying curves (Figure 3.20) indicated two approximately linear falling rate periods.

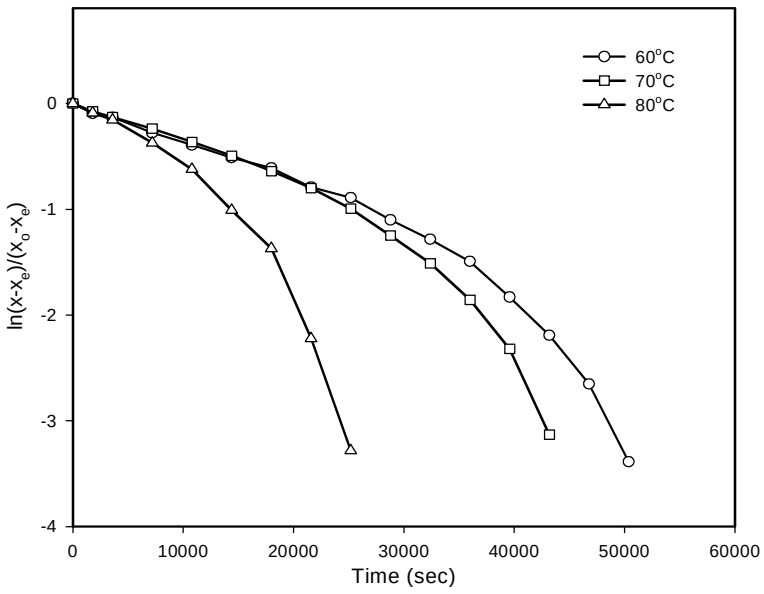

Figure 3.20. Logarithmic drying curves at various temperatures for *M.nigra*

The effect of shrinkage on D_{eff} was also considered for *M. nigra* and D_{eff} was calculated for both cases, with and without shrinkage. Table 3.7 shows the results for both. Similar to the results of *M. alba*, higher diffusion coefficients were found when shrinkage was not considered for second falling rate periods. Whereas, when shrinkage was taken into account, diffusion coefficients of second falling rate periods reduced and closed to diffusion coefficient of first falling rate periods. But it is still higher than that of the first falling rate period.

Table 3.7 Effective Diffusivity Values (m^2s^{-1}) for drying *M. nigra*.

Temperature(^oC)	D_{eff1}	D_{eff2} (withoutshrinkage)	D_{eff2} (with shrinkage)
60	$7.48x10^{-10}$	$2.43\ x10^{-9}$	$1.05\ x10^{-9}$
70	$8.85x10^{-10}$	$2.97\ x10^{-9}$	$1.39\ x10^{-9}$
80	$1.36x10^{-9}$	$4.79\ x10^{-9}$	$1.59\ x10^{-9}$

As it is seen in Table 3.7, D_{eff} increases with increase in temperature. As it was explained before, the effect of temperature on D_{eff} can be expressed by using an Arrhenius-type relationship. The activation energy was calculated by plotting the $\ln(D_{eff})$ versus the reciprocal of the absolute temperature ($1/T_{abs}$), and presented in Figure 3.21. The activation energy for diffusion was calculated as 29.43 kj/mol. The higher activation energy indicates the more sensitivity to temperature, so it can be said that *M. nigra* is more sensitive to increasing temperature than *M. alba* in the studied range.

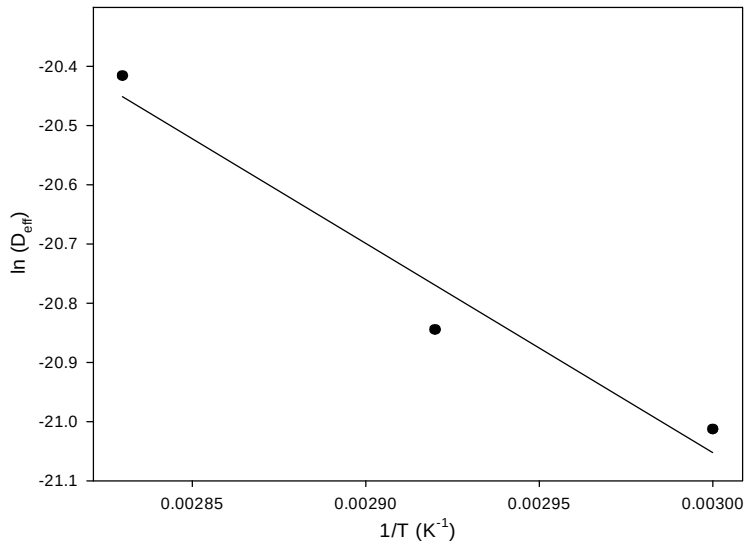

Figure 3.21. Relationship between diffusivity and reciprocal of absolute temperature

3.2.1.3. Shrinkage

The shrinkage behavior of *M. nigra* was evaluated as explained in Section 3.1.1.3. The relation of moisture content and volumetric shrinkage at 60, 70 and 80°C is given in Figure 3.22. *M. nigra* exhibited similar shrinkage behavior to that of *M. alba*, two segments was also seen for *M. nigra.*

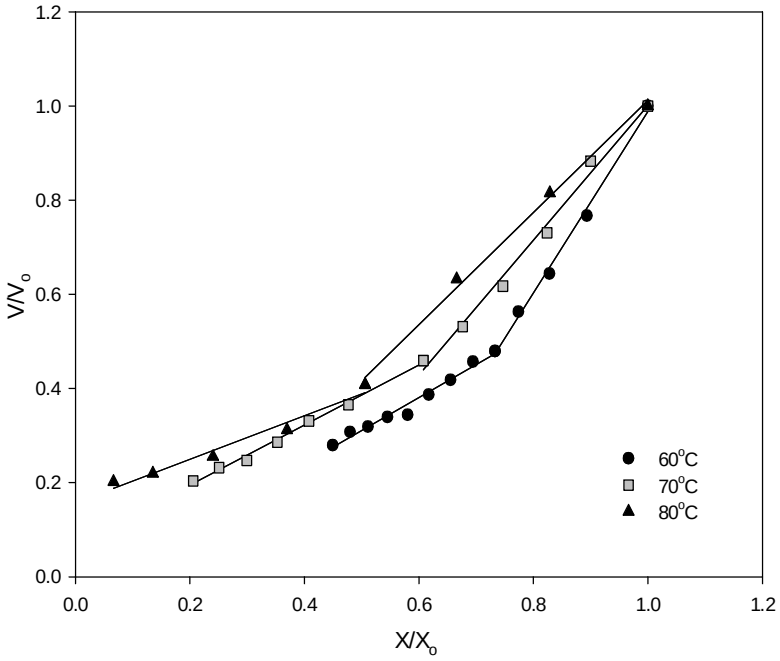

Figure 3.22. Relation of moisture content and volumetric shrinkage (*M. nigra*)

The shrinkage data show best fitting with Equation 3.3 and Equation 3.4. The characteristic parameters for these equations were indicated in Table 3.8. The reducing of *b-values* from b_1 to b_2 show that the shrinkage is leveling off towards the end of drying.

Table 3.8 Characteristics parameters for Equation 3.3 and Equation 3.4 (*M. nigra*)

Temperature (°C)	a_1	b_1	a_2	b_2
60	-0.94	1.93	0.04	0.70
70	-0.43	1.43	0.07	0.64
80	-0.18	1.19	0.16	0.46

The critical moisture content $(x/x_o)_c$, the point at where breaking occur, corresponding critical volume ratio and $(v/v_o)_c$ and final volume ratio $(v/v_o)_f$ (Figure 3.22) were given in Table 3.9. As it is seen the extent of shrinkage increased with

increasing temperature for *M. nigra* in opposite to the results of *M. alba*. The difference may be related with the compositional differences of mulberries. For example, total soluble solids content of *M. alba* is higher than the *M. nigra*, therefore during drying these soluble matters migrate together with water to the surface of the samples, and case hardening could occur, and due to volume fixation shrinkage stops even at lower temperature.

Table 3.9 Effect of temperature on critical moisture content $(x/x_o)_c$ and corresponding volumetric shrinkage ratio $(v/v_o)_c$ and final volumetric shrinkage ratio $(v/v_o)_f$

Temperature (^oC)	$(x/x_o)_c$	$(v/v_o)_c$	$(v/v_o)_f$
60	0.73	0.47	0.28
70	0.63	0.46	0.20
80	0.46	0.37	0.19

Shrinkage characteristics of *M. nigra* shows opposite behavior to that of *M. alba*, the extent of shrinkage increased with increasing temperature. But the results of statistical analysis show that, there is no significant differences ($p>0.05$) between the mean values of percent shrinkage of both *M. alba* and *M. nigra* for 60-70 ^oC and 70-80^oC but there is significant difference between 60 and 80^oC (Appendices H.). Besides, similar results have been reported earlier. Ratti (1994) for potatoes, apples and carrots; Ochoa et al. (2002) for rose hip fruit, Talla et al. (2004) for banana and Ochoa et al. (2007) for sweet cherry fruit, suggested that the temperature does not significantly affect the extent of shrinkage.

3.2.1.4. Changes in CIELAB color parameters (L^*, a^* and b^*) and color kinetics

The changes of Hunter color parameters, L^*, $a^*$and b^*, and ΔE^* for *M. nigra* were stated in Figure 3.23, at 60, 70 and 80^oC. When the L^* value exhibited increasing trend with drying time, a^* and b^* values showed decreasing. There is an increas in L^* and decrease in a^*. This means that the sample turns from dark to a bright color, with lose of coloring components.

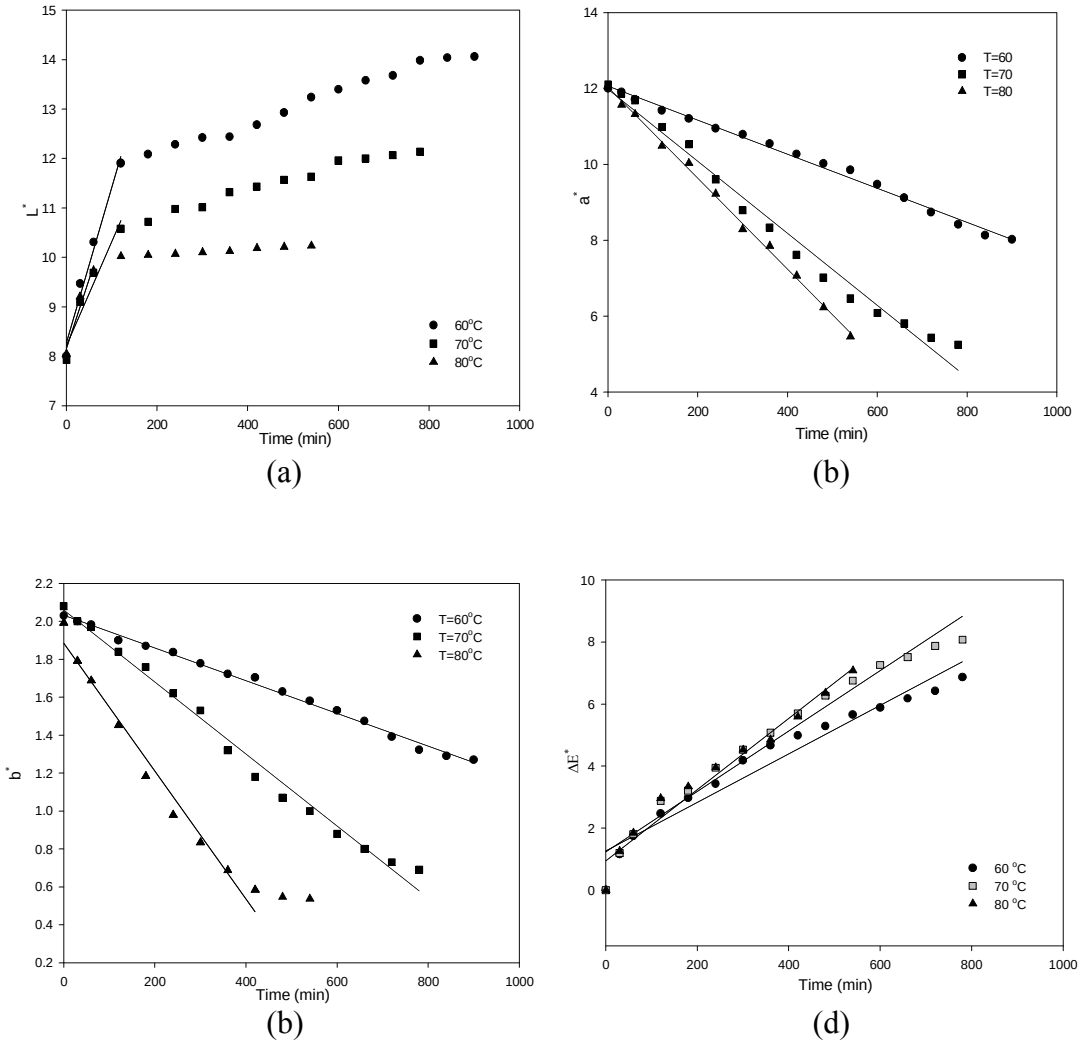

Figure 3.23 Changes of CIELAB color parameters, (a) L^*, (b) a^*, (c) $b^*$and total color differences (d) $\Delta E^*$with drying time at different temperatures

The reaction kinetics was studied for color parameters of *M. nigra*. The rate constants were calculated for each parameter as described in section 3.1.1.4 and results were given in Table 3.9 with their correlation coefficients. The higher correlation coefficients show that the parameters follow zero order reaction kinetics. The increase in temperature increased the rate constants. It might be the result of improving thermal degradation with incresing temperature.

Table 3.10 Linear regression analysis results of color parameters from zero order reaction kinetics.

Temperature	Parameters	k	r^2
60 °C	L^*	0.03146	0.97
	a^*	0.00448	0.99
	b^*	0.00086	0.99
	ΔE^*	0.00782	0.94
70 °C	L^*	0.02100	0.94
	a^*	0.00950	0.98
	b^*	0.00189	0.99
	ΔE^*	0.00973	0.96
80 °C	L^*	0.02793	0.95
	a^*	0.01198	0.99
	b^*	0.00337	0.98
	ΔE^*	0.01149	0.97

3.2.2. Microwave Drying Characteristics of *M. nigra*

Figure 3.24 shows the moisture-time relationship of *M. nigra* during microwave drying at 70 W. Total drying time to reach equilibrium moisture content was 115 min. The results indicate that the drying time to reach the equilibrium of the sample shortened when the samples of *M. nigra* dried by microwave drying.

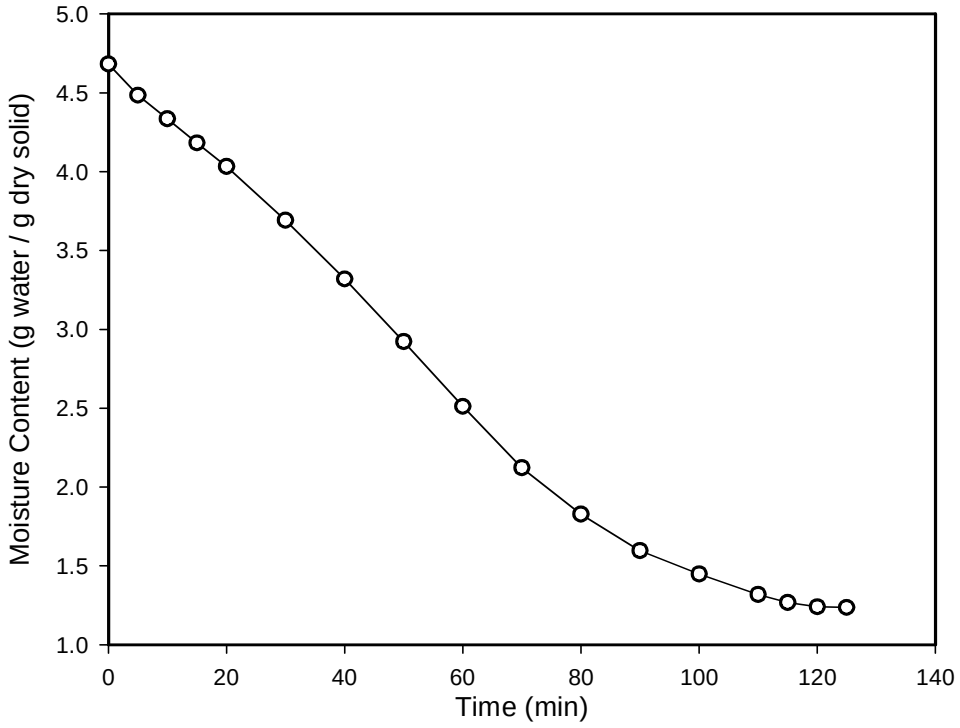

Figure 3.24. Drying curve for mulberry (*M. nigra*) at 70 W

3.2.3. Microwave Finish Drying Characteristics of *M. nigra*

Zhang et al. (2006) stated that, microwave (MW)-related (MW-assisted or MW-enhanced) combination drying is a rapid dehydration technique that can be applied to specific foods, particularly to fruits and vegetables. In recent years, great number of studies have been done about the applications of microwave energy either alone or combined with hot air or as microwave finishing after convective drying. For example, Feng and Tang (1998) for apple, Maskan (2000) for banana and Yang et al. (2005) for tapioca, were studied microwave finish drying. Funebo and Ohlsson (1998) for apple and mushroom, Contreras et al. (2008) for apple and strawberry and Bilbao et al. (2004) for apple were studied the combined 'hot air-microwave' drying.

The advantages of MW-related combination drying include the following: shorter drying time, improved product quality, and flexibility in producing a wide variety of dried products. But current applications are limited to small categories of fruits and vegetables due to high start-up costs and relatively complicated technology as compared to conventional convection drying. The reduction of drying time and color degradation in microwave application of this study allowed recommending the combination of conventional drying with microwave treatment of mulberry. Therefore the use of microwave energy for the finish drying of *M. nigra* has been evaluated in present study.

Drying Curve

The moisture-time relationship of *M. nigra* during microwave finish drying at 70W and 350W was indicated in Figure 3.10. The results show that the microwave finish application (70W) decreased the drying time by about 65% when compared to drying with hot-air (60^{o}C) alone. This is a good example of how only a small amount of microwave power can have a large benefit on a process with respect to drying time. Yang et al. (2005) and Maskan (2000) were also reported similar results. Figure 3.25 also shows that drying rate increased with power output of microwave oven, the percent reduction in time was 67.55 when 350 W used instead of 70W.

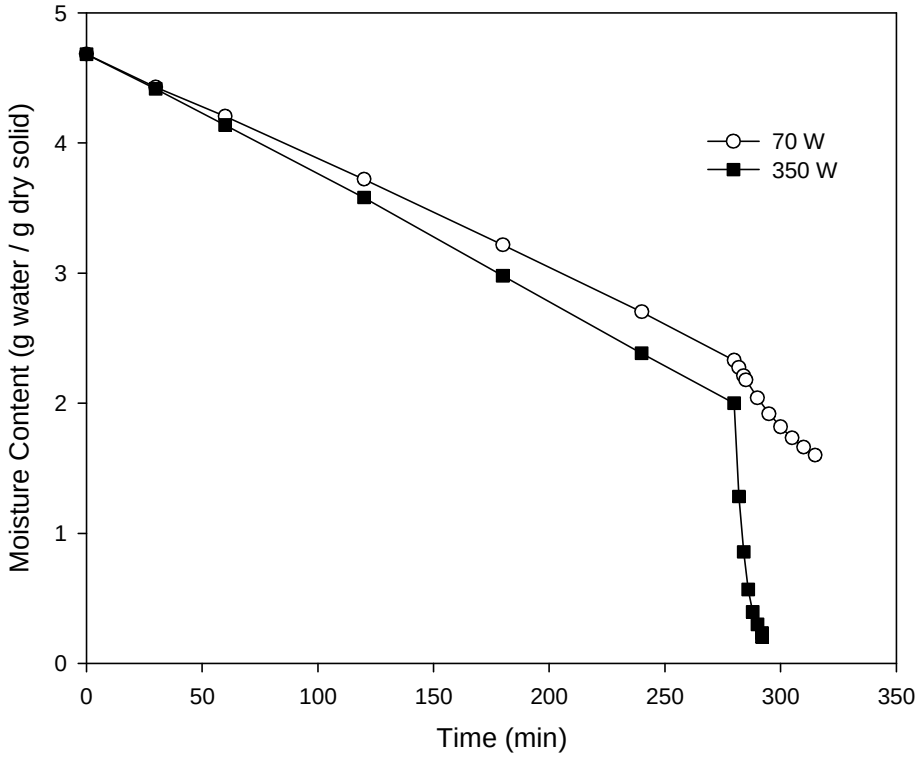

Figure 3.25. Drying curve for mulberry (*M. nigra*) dried by microwave finishing

Many researchers have successfully dried vegetables with high heat-sensitivity and fruits with high sugar contents. In all cases the drying time is reduced significantly, and in most cases the quality of the dried food products is improved.

3.2.4. Concentration of Mulberry (*Morus nigra*) Juice Extract and its Effects

3.2.4.1. Change in total soluble solids content (^{o}*Brix*)

Figure 3.26 shows soluble solids content (^{o}Brix) of mulberry juice against time of concentration process for three evaporation techniques. The times required to obtain the final concentrations of 60.00, 59.68 and 57.45 ^{o}Brix were 65, 105 and 150 min, for microwave, rotary vacuum and atmospheric heating processes, respectively. It shows that dehydration methods cause differences in evaporation rate. This result correlated with the obtained *k* value which indicated in Table 3.10.

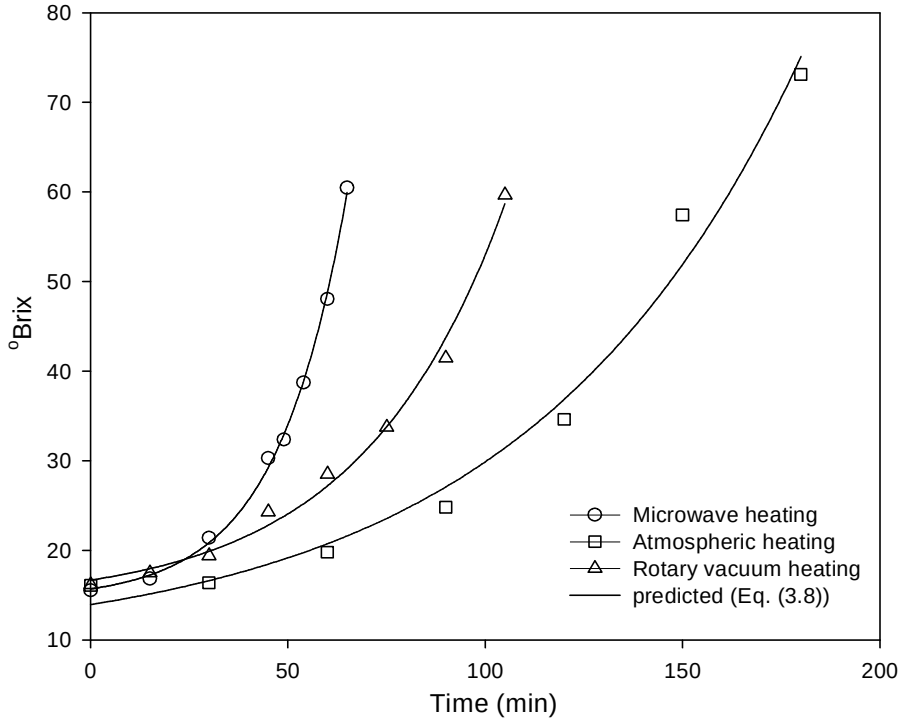

Figure 3.26. Changes in mulberry (*M. nigra*) juice concentration (°Brix) produced by various concentration processes

The change in concentration (°Brix) of juice samples against time was fitted to a three-parameter exponential equation (Equation 3.8). Fitted parameters of Equation 3.8 and their correlation coefficients (r^2) were reported in Table 3.10. The greater r^2 values indicate good fiting. The rate constants show that the evaporation rate of microwave is greater than rotary vacuum and evaporation rate of rotary vacuum is greater than that of atmospheric heating methods (Table 3.11).

Table 3.11 Kinetics parameters of Equation 3.8 for changes in °Brix of mulberry juice during concentration processes

Concentration process	B_o	B_1	k	r^2
Microwave	14.52	1.166	0.0563	0.998
Rotary vacuum	14.11	2.551	0.0272	0.993
Atmospheric	8.99	4.942	0.0144	0.980

3.2.4.2. Changes in CIELAB color parameters (L^*, a^* and b^*)

The change of Hunter color parameters was also studied for concentration of mulberry (*M. nigra*) juice. The changes of these parameters were indicated in Figure 3.27.

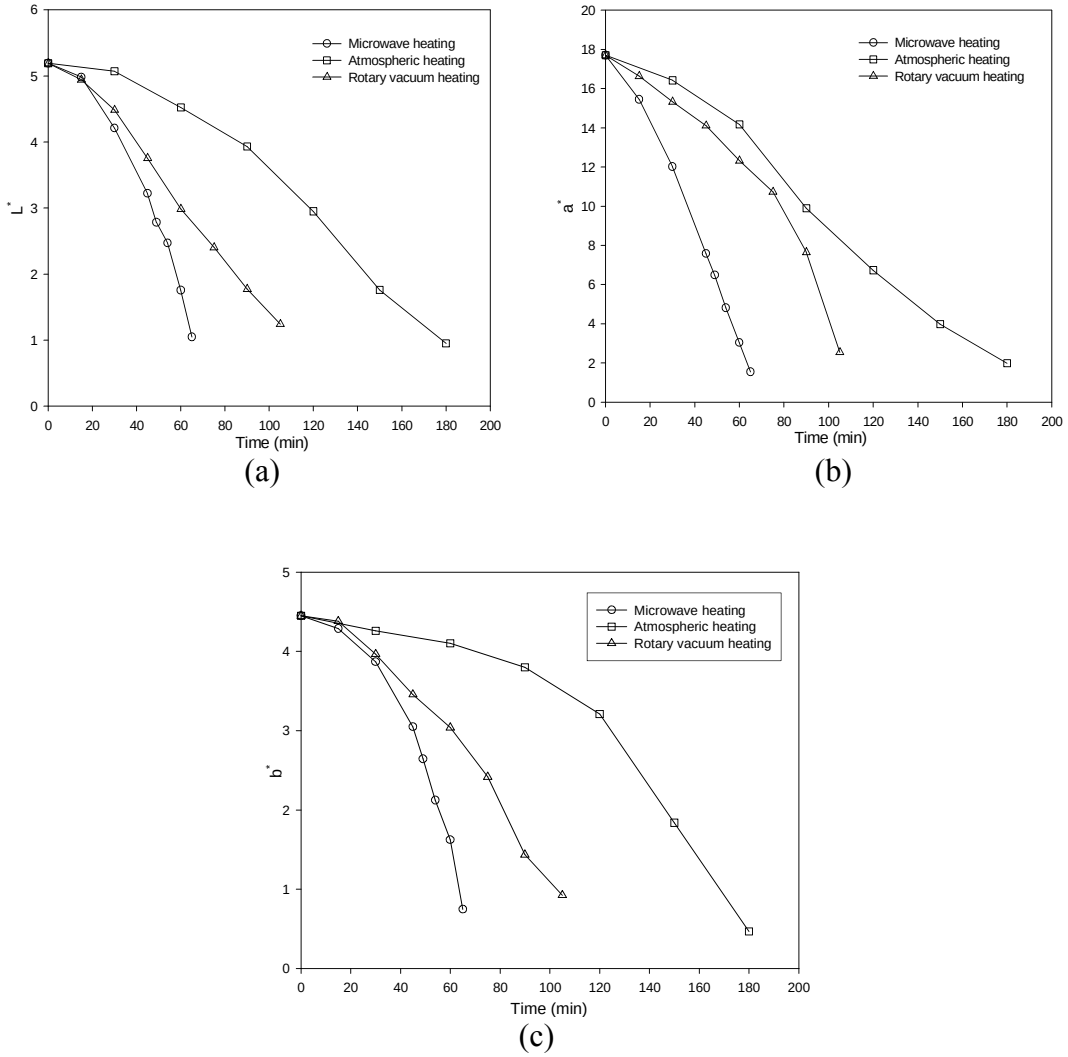

Figure 3.27. Changes of CIELAB color parameters, (a) L^*, (b) a^* and (c) b^* with drying time at different temperature

CHAPTER IV
CONCLUSION

1. The increasing the air temperature in hot air drying or the use of microwave energy instead of air drying reduced the drying time.
2. Moisture diffusivity can not remain constant throughout the drying, one or more rate periods can occur.
3. Shrinkage must be considered during the estimation of effective moisture diffusivity of shrinkable samples; it is more affective on D_{eff}.
4. Mulberry is a highly shrinkable fruit.
5. The increasing temperature does not cause a standard effect on the extent of shrinkage, it depends on the properties of dried sample.
6. Drying causes significant changes on the color of mulberry and microwave drying increases deterioration rate but causes less deteriorative effect.
7. Drying at 80^{o}C can be recommended for hot air drying with respect to color, shrinkage and drying time.
8. The rotary vacuum heating can be used to produce good mulberry juice concentrates with respect to color, nevertheless MW heating reduced significantly the concentration time.

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APPENDICES

A. ANOVA for Percent Shrinkage within the 60–70–80 ^{o}C Temperature Ranges for (*M. Alba)*

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	50.739	2	25.370	3.620	.070
Within Groups	63.080	9	7.009		
Total	113.819	11			

Post Hoc Tests
Homogeneous Subsets

VAR00002

Duncan

	N	Subset for alpha = .05	
VAR00001	1	2	1
80.00	4	78.8200	
70.00	4	82.8600	82.8600
60.00	4		83.4450
Sig.	.059	.762	

Means for groups in homogeneous subsets are displayed.
a Uses Harmonic Mean Sample Size = 4.000.

B. ANOVA for change of L^* value with time at T=60°C for (*M. Alba)*.

ANOVA

L^*value

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	2027.363	11	184.306	1072.555	.000
Within Groups	4.124	24	.172		
Total	2031.487	35			

Post Hoc Tests
Homogeneous Subsets

L*value

	N	Subset for alpha = .05										
Time	1	2	3	4	5	6	7	8	9	10	11	1
600	3	24.39										
540	3		25.19									
480	3		25.75									
420	3			28.41								
360	3				29.85							
300	3					31.33						
240	3						33.41					
180	3							35.74				
120	3								39.28			
60	3									43.84		
30	3										44.91	
0	3											45.63
Sig.		1.00	.10	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00

Duncan
Means for groups in homogeneous subsets are displayed.
a Uses Harmonic Mean Sample Size = 3.000.

C. ANOVA for change of a* value with time at T=60°C for (*M. Alba)*.

a* value

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	87.598	11	7.963	514.604	.000
Within Groups	.371	24	.015		
Total	87.970	35			

Post Hoc Tests
Homogeneous Subsets

a* value

Duncan

	N	Subset for alpha = .05						
Time	1	2	3	4	5	6	7	1
.0000	3	3.920000						
30.0000	3		4.323333					
60.0000	3			5.133333				
120.0000	3				6.236667			
180.0000	3					7.250000		
240.0000	3						7.470000	
300.0000	3							8.130000
480.0000	3							8.160000
360.0000	3							8.163333
600.0000	3							8.186667
540.0000	3							8.190000
420.0000	3							8.196667
Sig.		1.000	1.000	1.000	1.000	1.000	1.000	.569

Means for groups in homogeneous subsets are displayed.

D. ANOVA for change of b^* value with time at T=60°C for (*M. Alba)*.

b^*value

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	257.884	11	23.444	1634.916	.000
Within Groups	.344	24	.014		
Total	258.228	35			

Post Hoc Tests
Homogeneous Subsets

b^*value

Duncan

	N	Subset for alpha = .05								
Time	1	2	3	4	5	6	7	8	9	1
480	3	14.27								
600	3	14.28								
540	3	14.28								
420	3	14.29								
360	3		14.87							
300	3			15.28						
240	3				16.19					
180	3					18.14				
120	3						19.36			
60	3							20.32		
30	3								20.73	
0	3									21.05
Sig.		.87	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00

Means for groups in homogeneous subsets are displayed.
a Uses Harmonic Mean Sample Size = 3.000.

E. ANOVA for Percent Shrinkage with different drying methods (Hot-air Drying (60 ^{o}C) – MW Drying (350W)) for (*M. Alba)*.

PercentShrinkage

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	.525	1	.525	.032	.865
Within Groups	99.804	6	16.634		
Total	100.330	7			

F. ANOVA for ΔE with time during MW Drying (350W) (*M. Alba)*

ANOVA

ΔE^*

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	352.208	7	50.315	3207.565	.000
Within Groups	.125	8	.016		
Total	352.334	15			

Post Hoc Tests
Homogeneous Subsets

ΔE^*

Duncan

	N	Subset for alpha = .05				
VAR00001	1	2	3	4	5	1
.00	2	.0000				
5.00	2		7.5450			
10.00	2			11.3600		
15.00	2				13.5300	
20.00	2				13.6800	
30.00	2					14.0610
25.00	2					14.0700
35.00	2					14.2700
Sig.		1.000	1.000	1.000	.265	.148

Means for groups in homogeneous subsets are displayed.
a Uses Harmonic Mean Sample Size = 2.000.

G. ANOVA for ΔE in different drying methods (Hot-air Drying (60 ^{o}C) – MW Drying (350W)) for (*M. Alba)*.

ANOVA

DE

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	121,140	1	121,140	1348,500	,000
Within Groups	,359	4	,090		
Total	121,500	5			

H. ANOVA for Shrinkage within the 60–70–80 ^{o}C Temperature Ranges for (*M. nigra).*

Shrinkage

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	148.937	2	74.468	3.830	.063
Within Groups	174.990	9	19.443		
Total	323.926	11			

Post Hoc Tests
Homogeneous Subsets

Duncan

	N	Subset for alpha = .05	
Factors	1	2	1
60.00	4	72.4150	
70.00	4	79.3575	79.3575
80.00	4		80.3250
Sig.	.053	.763	

Means for groups in homogeneous subsets are displayed.
a Uses Harmonic Mean Sample Size = 4.000.