ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL

EVALUATION OF THE EFFECTS OF BLANCHING AND ULTRASOUND PRE-TREATMENTS ON THE THIN LAYER DRYING KINETICS OF THYME

Ph.D. THESIS

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(506152508)

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Thesis Advisor: Assoc. Prof. Dr. Ebru FIRATLIGİL

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ISTANBUL TEKNİK ÜNİVERSİTESİ ★ LİSANSÜSTÜ EĞİTİM ENSTİTÜSÜ

HAŞLAMA VE ULTRASON ÖN İŞLEMLERİNİN KEKİK BİTKİSİNİN İNCE TABAKA KURUTMA KİNETİĞİ ÜZERİNDEKİ ETKİLERİNİN DEĞERLENDİRİLMESİ

DOKTORA TEZİ

Osman Yağız TURAN
(506152508)

Gıda Mühendisliği Anabilim Dalı

Gıda Mühendisliği Programı

Tez Danışmanı: Doç. Dr. Ebru FIRATLIGİL

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Osman Yağız TURAN, a Ph.D. student of ITU Graduate School student ID 506152508, successfully defended the thesis entitled "EVALUATION OF THE EFFECTS OF BLANCHING AND ULTRASOUND PRE-TREATMENTS ON THE THIN LAYER DRYING KINETICS OF THYME", which he prepared after fulfilling the requirements specified in the associated legislations, before the jury whose signatures are below.

Thesis Advisor :	**Assoc. Prof. Dr. Ebru FIRATLIGİL** Istanbul Technical University	…........................
Jury Members :	**Prof. Dr. Esra ÇAPANOĞLU GÜVEN** Istanbul Technical University	....
	Assist. Prof. Dr. Esra DOĞU BAYKUT Istanbul Medeniyet University	....
	Assoc. Prof. Dr. Aslı Can KARAÇA Istanbul Technical University	....
	Assoc. Prof. Dr. Zeynep TACER CABA Bahcesehir University	....

Date of Submission : 17 May 2025
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To my dearest spouse and parents,

FOREWORD

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Osman Yağız TURAN
(Food Engineer, M.Sc.)

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ABBREVIATIONS

ANOVA	**:** Analysis of Variance
CIE	**:** International Comission on Illumination
MR	**:** Moisture Ratio
RMSE	**:** Root Mean Square Error

SYMBOLS

$\mathbf{M_t}$	: Moisture content at process time t
$\mathbf{M_0}$	: Initial moisture content
$\mathbf{M_\infty}$	: Equilibrium moisture content
$\mathbf{A_1}$	: Geometric constant
$\mathbf{A_2}$	: Geometric constant
L	: Thickness
a	: Model constant
b	: Model constant
c	: Model constant
n	: Model constant
k	: Time constant
t	: Time
N	: Number of observations
z	: Number of constants in the model
c	: Model constant
$\mathbf{R^2}$	: Coefficient of determination
χ^2	: Reduced chi-square
$\mathbf{MR_{exp,i}}$	: Experimental moisture ratio
$\mathbf{MR_{pre,i}}$	: Predicted moisture ratio
$\mathbf{D_{eff}}$	: Effective moisture diffusivity
$\mathbf{D_0}$	: Arrhenius factor
$\mathbf{E_a}$	: Activation energy for diffusion
R	: Universal gas constant
L*	: CIE value, lightness
a*	: CIE value, redness
b*	: CIE value, yellowness
E*	: Total color
C*	: Chroma

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EVALUATION OF THE EFFECTS OF BLANCHING AND ULTRASOUND PRE-TREATMENTS ON THE THIN LAYER DRYING KINETICS OF THYME

SUMMARY

Drying is one of the mostly used preservation methods which involves the removal of moisture from a food product while a simultaneous heat and mass transfer takes place. It is applied to vegetables, fruits, herbs and other agricultural products to minimize microbial spoilage and deterioration reactions thus extending the shelf life. Hot air drying process is widely used in reducing the moisture content of agricultural products due to its low cost and ease of operation, but extended times of exposure to high temperatures causes significant decrease in the product quality. Therefore, it is required to control drying parameters in order to minimize chemical and physical changes in food material, and also to minimize nutritional losses. The drying rate during the operation is affected by the vapor pressures of air and food material, properties of air such as temperature and velocity, and properties of the food material such as thickness, surface area and moisture diffusion within the product.

There is an increasing interest in dried vegetables, herbs, and fruits, however recent researches have shown that, application of drying barely has some negative impacts on the quality of the products such as, color loss, structural changes, bioactive content and nutritional loses. Additionally, improving the drying rate and reducing the energy costs are important issues. Thus, there is a tendency for application of pre-treatments before drying. Pre-treatments improve drying efficiency, by reducing drying time and energy consumption and by minimizing nutritional and structural changes of the foods.

Mathematical models that describe drying processes are used for improving drying systems or designing new systems. Models provide an improved understanding of the drying process parameters. Information such as temperature and moisture are critical for drying process and equipment design, also for storage and handling procedures of the dried food material. These information can be provided by models that describe drying mechanisms. Thin layer drying models are widely used in describing drying characteristics of food products. These models are classified into three groups namely, theoretical, semi-theoretical and empirical models. Most widely applied thin layer models are semi-theoretical and empirical models as these models consider external resistance to moisture transfer between food material and air, thus providing a better prediction of drying behavior. Empirical models neglect the fundamentals of the process and cannot show a clear view of the drying process, still may express the drying curve for the conditions of the experiment. Semi theoretical models are derived by simplifying the general series solution of Fick’s second law and they do not need assumptions of geometry, diffusivity and conductivity of a food material. Widely used semi theoretical thin layer models are the Lewis model, the Page model, the Henderson and Pabis model, and the Logarithmic model.

Thyme (*Thymus vulgaris*) is an herb belonging to the *Lamiaceae* family, used as culinary and medicinal purposes. The plant has been used for medicinal purposes in

ancient times and currently being used for its flavor in cooking. Thyme also has antioxidative effects and its essential oils (thymol and carvacrol) shows antimicrobial and inflammatory properties. Thyme plant blooms in June to July and is cultivated all around the world. Preservation of herbs is performed by two operations in general: drying and irradiation. Drying process is used to store seasonal plants all year round, provides a great level of preservation.

The aim of this study was to reduce the adverse effects of conventional drying process on the chemical and physical properties of fruits and vegetables and increase the nutritional quality of dried products. During the studies, it was aimed to demonstrate the drying kinetics of pre-treated products and to determine the thin layer characteristics of the drying operations. The drying process was carried out with two different methods, namely air drying (60, 70 and 80°C) and microwave drying (30% - 390 W, 50% - 650 W, and 70% - 910 W). First of all, for the comparison, drying without pre-treatment was carried out with both methods and the kinetic data were modeled by investigating the compatibility of the moisture change ratios with the Lewis, Page, Henderson and Pabis, Midilli and Logarithmic models. In addition, effective diffusivity and drying activation energy were calculated using the moisture change ratio data. Finally, International comission on illumination (CIE) values were measured using a color measurement device in order to interpret the effect of the drying processes on the color of thyme leaves.

In this study, blanching and ultrasonication pre-treatments were applied to improve drying efficiency and increase the quality of the dried product before air and microwave drying. Samples were subjected to blanching pre-treatment for 1, 2 and 3 minutes and ultrasonication pre-treatment for 5, 10 and 15 minutes. The compatibility of moisture change ratios with Lewis, Page, Henderson and Pabis, Midilli and Logarithmic models was investigated and kinetic data were modeled. In addition, effective diffusivity and drying activation energy were calculated using moisture change ratio data. Finally, CIE values were measured using a color measurement device in order to interpret the effect of drying processes on the color of thyme leaves.

Consequently, the effects of two different pre-treatment applications on drying time for both drying methods were compared and interpreted. In addition, the color measurement results of un-treated fresh, dried without pre-treatment and dried after pre-treatment thyme leaf samples were compared and the effects of different drying methods and different pre-treatment applications on the quality of the product were interpreted.

According to the obtained data, pre-treatment applications before both drying types had an effect in a way that shortened the drying time. In this context, it was concluded that pre-treatment application could contribute to energy saving. For 60°C air drying, 2-minute blanching pretreatment was evaluated as a better option compared to others. Although ultrasonication application at this temperature shortened the drying time, no difference was observed between different application levels. 2-minute blanching application before air drying performed at 70°C emerged as the best option. When the results of samples to which ultrasonication was applied before air drying performed at the same temperature were examined, it was observed that 15-minute application gave better results than the others. Blanching application before air drying at 80°C gave better results than ultrasonication application, but since no difference was observed between application levels, it was concluded that the shortest pre-treatment applications could be preferred in terms of time saving.

When microwave drying results considered, it was observed that the drying time was 7 minutes when the boiling process was applied before drying at 30% power level, while it was observed that the drying time was 5 minutes when the ultrasonication process was applied. At 50% and 70% power levels, 15 minutes of ultrasonication application before microwave drying gave significantly better results than other applications.

Color values, L*, a* and b* measured after drying operations showed that blanching pre-treatment caused the darkness of samples to increase and greenness to decrease and this may be attributed to the loss of heat sesitive chlophyll content due to the high temperature applications.

HAŞLAMA VE ULTRASON ÖN İŞLEMLERİNİN KEKİK BİTKİSİNİN İNCE TABAKA KURUTMA KİNETİĞİ ÜZERİNDEKİ ETKİLERİNİN DEĞERLENDİRİLMESİ

ÖZET

Kurutma, eş zamanlı ısı ve kütle aktarımının gerçekleştiği bir gıda ürününden nemin uzaklaştırılmasını içeren, en çok kullanılan koruma yöntemlerinden biridir. Mikrobiyal bozulmayı ve bozulma reaksiyonlarını en aza indirerek raf ömrünü uzatmak için sebze, meyve, şifalı bitkiler ve diğer tarım ürünlerine uygulanır. Sıcak havayla kurutma işlemi, düşük maliyeti ve kullanım kolaylığı nedeniyle tarımsal ürünlerin nem içeriğinin azaltılmasında yaygın olarak kullanılmaktadır ancak yüksek sıcaklıklara uzun süre maruz kalması, ürün kalitesinde ciddi düşüşlere neden olmaktadır. Bu nedenle gıda maddesindeki kimyasal ve fiziksel değişimlerin en aza indirilmesi ve aynı zamanda besin kayıplarının en aza indirilmesi için kurutma parametrelerinin kontrol edilmesi gerekmektedir. Operasyon sırasında kuruma hızı, havanın ve gıda maddesinin buhar basınçlarından, havanın sıcaklık ve hız gibi özelliklerinden ve gıda maddesinin kalınlık, yüzey alanı ve ürün içindeki nem difüzyonu gibi özelliklerinden etkilenir.

Kurutulmuş sebze, bitki ve meyvelere ilgi giderek artmaktadır ancak son araştırmalar tek başına uygulanan kurutmanın ürün kalitesi üzerinde renk kaybı, yapısal değişiklikler, biyoaktif içerik ve besin kayıpları gibi olumsuz etkilerinin olduğunu göstermektedir. Ayrıca kurutma hızının arttırılması ve enerji maliyetlerinin düşürülmesi de önemli konulardır. Bu nedenlerden ötürü son yıllarda kurutmadan önce ön işlemlerin uygulanmasına yönelik bir eğilim vardır. Ön işlemler, kurutma süresini ve enerji tüketimini azaltarak ve gıdalardaki besinsel ve yapısal değişiklikleri en aza indirerek kurutma verimliliğini artturmaktadır.

Kurutma süreçlerini tanımlayan matematiksel modeller, kurutma sistemlerinin iyileştirilmesi veya yeni sistemlerin tasarlanması için kullanılmaktadır. Modeller kurutma işlemi parametrelerinin daha iyi anlaşılmasını sağlar. Sıcaklık ve nem gibi bilgiler, kurutma prosesi ve ekipman tasarımının yanı sıra kurutulmuş gıda malzemesinin saklanması ve taşınması prosedürleri için de kritik öneme sahiptir. Bu bilgiler kurutma mekanizmalarını açıklayan modeller tarafından sağlanabilir. İnce tabaka kurutma modelleri, gıda ürünlerinin kuruma özelliklerinin tanımlanmasında yaygın olarak kullanılmaktadır. Bu modeller teorik, yarı teorik ve ampirik modeller olmak üzere üç gruba ayrılmaktadır. En yaygın olarak uygulanan ince tabaka modelleri, yarı teorik ve ampirik modellerdir; çünkü bu modeller, gıda malzemesi ile hava arasındaki nem transferine karşı dış direnci dikkate alır ve böylece kuruma davranışının daha iyi tahmin edilmesini sağlar. Ampirik modeller prosesin temellerini ihmal eder ve kurutma prosesinin net bir görünümünü gösteremez, yine de deney koşulları için kuruma eğrisini ifade edebilir. Yarı teorik modeller, Fick'in ikinci yasasının genel seri çözümünün basitleştirilmesiyle türetilir ve bir gıda maddesinin geometrisi, yayınım ve iletkenliği varsayımlarına ihtiyaç duymazlar. Yaygın olarak

kullanılan yarı teorik ince katman modelleri Lewis modeli, Page modeli, Henderson ve Pabis modeli ve Logaritmik modeldir.

Kekik (Thymus vulgaris), Lamiaceae familyasına ait, mutfakta ve tıbbi amaçlarla kullanılan bir bitkidir. Bitki, eski çağlarda tıbbi amaçlarla kullanılmış olup, günümüzde lezzeti nedeniyle yemek pişirmede kullanılmaktadır. Kekiğin ayrıca antioksidatif etkileri vardır ve içindeki esansiyel yağlar (timol ve karvakrol) antimikrobiyal ve inflamatuar özellikler gösterir. Kekik bitkisi haziran-temmuz aylarında çiçek açar ve dünyanın her yerinde yetiştirilir. Bitkilerin muhafazası genel olarak iki işlemle gerçekleştirilir: kurutma ve ışınlama. Mevsimlik bitkileri tüm yıl boyunca depolamak için uygulanan kurutma işlemi, büyük düzeyde koruma sağlar.

Bu çalışmanın amacı geleneksel kurutma işleminin meyve ve sebzelerin kimyasal ve fiziksel özellikleri üzerindeki olumsuz etkilerini azaltmak ve kurutulmuş ürünlerin besin kalitesini arttırmaktır. Çalışmalar sırasında ön işlem görmüş ürünlerin kurutma kinetiğinin ortaya konulması ve kurutma işlemlerinin ince tabaka özelliklerinin belirlenmesi amaçlanmıştır. Kurutma işlemi hava kurutması (60, 70 ve 80°C) ve mikrodalga kurutması (30% - 390 W, 50% - 650 W, and 70% - 910 W) olmak üzere iki farklı yöntem ile gerçekleştirilmiştir. Öncelikle karşılaştırma amacıyla her iki yöntem ile de ön işlemsiz kurutma gerçekleştirilmiş ve zamana karşı olarak ölçülen nem değişim oranlarının Lewis, Page, Henderson ve Pabis, Midilli ve Logaritmik modellerine olan uyumluluğu araştırılarak kinetik veriler modellenmiştir. Buna ek olarak, zamanla nem değişim oranı verilerinden yararlanılarak efektif difüzivite ve kurutma aktivasyon enerjisi hesaplanmıştır. Son olarak, kurutma işlemlerinin kekik yapraklarının rengi üzerindeki etkisini yorumlayabilmek amacıyla renk ölçüm cihazı kullanılarak CIE değerleri ölçülmüştür.

Bu çalışmada hava ve mikrodalga kurutmalarının öncesinde kurutma verimini geliştirmeye ve kurutulmuş ürünün kalitesini arttırmaya yönelik olarak haşlama ve ultrasonikasyon ön işlemleri uygulanmıştır. Numuneler haşlama ön işlemine 1, 2 ve 3 dakika süresince, ultrasonikasyon işlemine ise 5, 10 ve 15 dakika süresince tabi tutulmuştur. İki farklı yöntem ile belirtilen üç farklı seviyede kurutma işlemlerinin gerçekleştirilmesi sırasında zamana bağlı olarak ölçülen nem değişim oranlarının Lewis, Page, Henderson ve Pabis, Midilli ve Logaritmik modellerine olan uyumluluğu araştırılarak kinetik veriler modellenmiştir. Buna ek olarak, zamanla nem değişim oranı verilerinden yararlanılarak efektif difüzivite ve kurutma aktivasyon enerjisi hesaplanmıştır. Son olarak, kurutma işlemlerinin kekik yapraklarının rengi üzerindeki etkisini yorumlayabilmek amacıyla renk ölçüm cihazı kullanılarak CIE değerleri ölçülmüştür.

Sonuç olarak, her iki kurutma yöntemi için de iki farklı ön işlem uygulamasının kurutma süresi üzerine olan etkisi karşılaştırılarak yorumlanmıştır. Buna ek olarak, hiç işlem görmemiş taze, ön işlem uygulanmadan kurutulan ve ön işlem uygulaması sonrası kurutulan kekik yaprağı numunelerinin renk ölçümü sonuçları kıyaslanarak farklı kurutma yöntemlerinin ve farklı ön işlem uygulamalarının ürünün kalitesi üzerine etkileri yorumlanmıştır.

Elde edilen verilere göre, her iki kurutma türünde de ön işlem uygulamaları kurutma süresini kısaltacak şekilde etki etmiştir. Bu bağlamda, ön işlem uygulamasının enerji tasarrufuna katkıda bulunabileceği sonucuna varılmıştır. 60°C'de gerçekleştirilen hava kurutmasında 2 dakikalık haşlama ön işleminin diğerlerine göre daha iyi bir seçenek olduğu değerlendirilmiştir. Bu sıcaklıkta ultrasonikasyon uygulaması kurutma süresini kısaltsa da farklı uygulama seviyeleri arasında değişiklik gözlemlenmemiştir. 70°C'de gerçekleştirilen hava kurutması öncesinde 2 dakikalık haşlama uygulaması en iyi seçenek olarak ortaya çıkmaktadır. Aynı sıcaklıkta gerçekleştirilen hava kurutması öncesinde ultrasonikasyon uygulaması yapılan numunelerin sonuçlarına bakıldığında 15 dakikalık uygulamanın diğerlerine göre daha iyi sonuç verdiği gözlemlenmiştir. 80°C'de hava kurutması öncesinde yapılan haşlama uygulaması ultrasonikasyon uygulamasından daha iyi sonuç vermekte fakat uygulama seviyeleri arasında herhangi bir fark gözlemlenmediğinden zaman tasarrufu açısından en kısa süreli ön işlem uygulamalarının tercih edilebileceği souncuna varılmıştır. Mikrodalga kurutması sonuçlarına bakıldığında, %30 güç seviyesinde kurutma öncesinde haşlama işlemi uygulanması durumunda kurutma süresinin 7 dakika olduğu görülürken, ultrasonikasyon işlemi uygulandığında kurutma süresinin 5 dakika olduğu gözlemlenmiştir. %50 ve %70 güç seviyelerinde ise mikrodalga kurutması öncesinde 15 dakika ultaronikasyon uygulaması diğer uygulamalara göre anlamlı şekilde daha iyi sonuç vermiştir.

Kurutma işlemleri sonrasında ölçülen L*, a* ve b* renk değerleri, haşlama ön işleminin numunelerin koyuluğunu artırdığını, yeşilliğini ise azalttığını göstermektedir ve bu durum yüksek sıcaklık uygulamalarına bağlı olarak ısıya duyarlı klorofil içeriğindeki kayba bağlanabilir.

1. INTRODUCTION

1.1 Significance of the Thesis

In this study, several drying pre-treatment applications were applied to thyme leaves; physical, chemical and nutritional quality of the dried food material and the kinetics of the processes were determined. Single drying type and single method studies about pre-treatments are present in literature, which mostly cover the comparison of one method with conventional drying methods. In this study, two different pre-treatment operations were applied to thyme leaves and subjected to comparison against each other and two different conventional drying methods regarding the physical, chemical, and nutritional effects and drying kinetics. Such comprehensive investigation concerning the effects and utilization of pre-treatments is unavailable in the literature and would be a substantial addition.

1.2 Purpose and Scope of the Thesis

- To reduce the adverse effects of conventional drying processes on the chemical and physical properties of thyme leaves.
- To increase the nutritional quality of dried product.
- To demonstrate the drying kinetics of pre-treatments and to determine the thin layer characteristics of the drying operations.

1.3 Unique Aspect

Fruits and vegetables are dried to obtain shelf stable and versatile food products. Drying operations conducted in industry are mostly based on simple hot-air convection methods. These applications are both adversely affecting the quality of the product and high-energy consuming. Long duration heat treatment on plant originated foods which have high production and exportation rates in Turkey, such as apricot, fig, grape, tomato and eggplant; causes aroma/color changes, essential amino acid and vitamin losses. Heat and mass transfer, and phase transitions during drying increases the energy consumption. Overcoming the adverse effects of conventional drying is mostly difficult. Simplicity, low equipment and installation costs, and traditional operation know-how makes these technologies highly accepted. Instead of removing these technologies completely, improvements regarding the energy efficiency and product quality would be an important addition.

This thesis suggests that application of pre-treatments prior to conventional drying would increase the drying efficiency and lower the duration of the process, therefore energy consumption and adverse effects of heat transfer would be minimized. Pre-treatments are independent from the actual drying process, so they can be installed as integrated with a drying equipment or on an existing drying equipment. For this purpose, two different pre-treatments, namely; ultrasound and blanching treatments were applied to thyme leaves prior to both air and microwave drying.

Thin layer drying kinetics regarding the pre-treatment – drying combination of the dried products were calculated and discussed. Chemical, physical and nutritional quality of the products were determined by measurement of color, shrinkage, and sensory analysis. Consequently, effective pre-treatment – drying combination for thyme leaves were determined in terms of chemical, physical and nutritional quality and process duration and kinetics.

1.4 Impact

Turkey is one of the biggest food and agriculture producers of the world, and the premier producer of certain dry foods. Plant originated foods which have high production and exportation rates in Turkey, such as apricot, fig, grape, tomato and eggplant are conventionally dried with hot-air. The integration of novel pre-treatments into existing dryers would improve energy efficiency and product quality in dry food industry. Outcome of this thesis can potentially be utilized in the food industry towards process improvement.

2. LITERATURE REVIEW

Drying is one of the oldest preservation methods used for centuries all around the world. The basic principle is to reduce moisture content of the food material that inhibits the deteriorative reactions. Most important methods used for dying foods are traditional methods like solar and hot air dying. Solar dying is the oldest drying method where food material is subjected to sun on an open surface such as concrete or soil. It is a low temperature air-drying method taking long time, hence susceptible to environmental deterioration, labor consuming and primitive. Convective hot air drying on the other is rapid but uses high temperatures and energy consuming. Main setback of this method is the adverse effects of heat treatment on the quality of the food material and high rate of energy consumption. Nevertheless, these methods are the primary drying methods used in industry currently.

2.1 Drying Pre-Treatments

The idea of using pre-treatments is to enhance the drying operation of heat-sensitive materials. Main assumption of these operations is to reduce drying time without increasing the operation temperature and energy consumption (Musielak, Mierzwa and Kroehnke, 2016). Thus, these methods should minimize the adverse effects of heat treatment in an energy efficient manner. Preeminent methods used for this purpose are ultrasound, blanching, freezing, solution immersion and osmotic drying.

2.1.1 Ultrasound

Sonication is a promising technology used in food processes to induce desired chemical and physical changes. Ultrasound application can support several processes such as drying, extraction, emulsification, freezing and suchlike summarized in Figure 2.1 (Ren *et al.*, 2018). Ultrasound mechanism can be expressed as the generated acoustic waves in a medium (mostly water) promote expansion and compression of the subjected material, leading to formation of microchannels and leakage of intercellular liquid (Nowacka *et al.*, 2012).

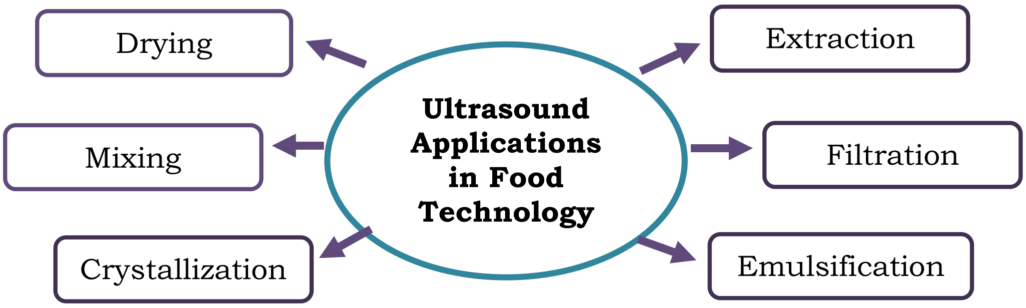

Figure 2.1: Applications of ultrasound.

The acoustic intensity applied to food materials in a liquid is generated by a transducer, which is connected to an energy-supplying device. Sonic wave frequency used in ultrasound applications may be between 20 kHz to 100 MHz; food applications require low frequencies as higher frequencies increase the energy absorption by the solution, therefore waves does not penetrate deep in the solid (Cárcel *et al.*, 2007).

2.1.2 Blanching

Blanching is a thermal treatment used before several preservation methods (drying, freezing, canning) in order to inactive enzymes, prevent color changes, alter microbial reactions and prolong shelf life. Typical hot water blanching is applied by immersion of food product into hot water (70-100°C) for several minutes (Wang *et al.*, 2017). A typical hot water blanching application on almonds is shown in Figure 2.2.

Figure 2.2: Industrial blanching application on almonds at Blue Diamond Growers, CA (Mikell Knights, 2014).

Blanching is applied before drying treatment to depolymerize pectic polymers that involved in cell adhesion thus softening the plant tissue. Change in the cell wall structure in terms of strength and composition results in alteration of dehydration kinetics in a preferred manner to decrease drying time (Ando *et al.*, 2016). Hot water blanching reported to reduce drying time up to a certain operation time, however similar to other thermal operations, blanching may affect the concentration of some bioactive compounds in vegetables (Ren *et al.*, 2018).

2.1.3 Freeze-thawing

Freezing is used as a preservation method for food products. Materials are freezed at below 0°C temperatures to hinder deterioration reactions. As an adverse effect of this operation, water in fruit and vegetable cells expand in solid state and injures cells. Later when the material thawed, water from internal sections remain unbound and diffuses out. It is suggested that, drying of food materials may be accelerated by using this mechanism as a pre-treatment. Studies indicate that freezing treatment alters water and ion permeability of cell membranes by forming ice crystals (Ando *et al.*, 2016) and breaks cell walls allowing water to transport easily (Tatemoto *et al.*, 2016), thus leading to an increase in the drying rate.

2.1.4 Solutions pre-treatment

Drying operations are drastically limited by the physicochemical properties of food materials to be dried. Most food materials subjected to drying have a kind of skin or protective surface layer. Drying of fruits is limited to an external waxy layer that interrupts mass transfer and reduces the drying rate. Physical pre-treatments are suggested for whole fruits, which have waxy skins such as grape, cherry, plum, tomato, apricot and suchlike. Most commonly used chemical pretreatment used for drying of whole fruits is dipping the fruits into a solution of olive oil and potassium carbonate (K_2CO_3) or fatty acid ethyl esters (e.g. ethyl oleate) and K_2CO_3 (Vásquez-Parra, Ochoa-Martínez and Bustos-Parra, 2013) (shown in Figure 2.3).

Figure 2.3: Chemicals used in dipping pretreatment; ethyl oleate, olive oil and potassium carbonate, left to right.

Surface treatment of grapes or other waxy cuticle materials by ethyle oleate increase the drying rate by altering the waxy layer on the surface and reducing the resistance to water diffusion. Ethyl oleate acts on the skin and dissolves the waxy layer of the fruit that results to decrease of mass transfer resistance during drying, still higher dipping times or high concentrations may cause adverse effects on the subjected food material (Doymaz and Pala, 2002). Also, Vasquez-Parra et. al. states that using olive or sunflower oils increase the drying rate of gooseberries when applied at the concentration of 5-10% with the addition of K_2CO_3 (2013). This is explained by the loss of fruit firmness and subsequent shrinkage, and reduction of fruit porosity.

2.1.5 Osmotic dehydration

Osmotic dehydration is a water removal process applied to fruits and vegetables to obtain minimally processed foods or used as a pre-treatment to drying removing the moisture content up to 50%. Moisture content of a food material is reduced by immersion in a hypertonic solution where the solution provides osmotic pressure that promotes the diffusion of water from the food material to the solution (Silva, Fernandes and Mauro, 2014). Schematic representation of osmotic dehydration is given in Figure 2.4. below.

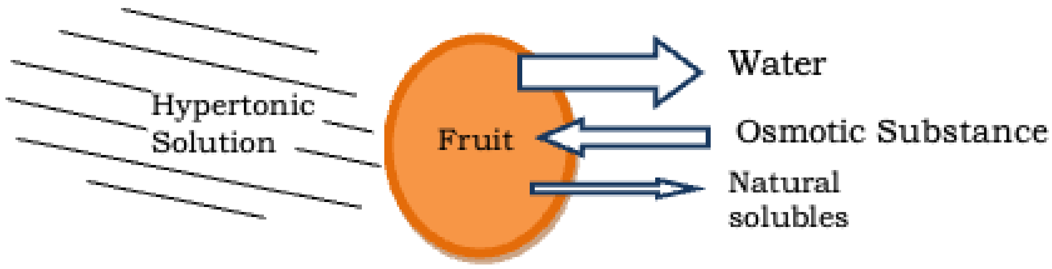

Figure 2.4: Schematic representation of osmotic dehydration.

Characteristic of the osmotic agent used in the process affects the water loss and solid gain during the process. However, the type of solution may affect the sensory and nutritional quality of the product. Sucrose, glucose and calcium salts are the main agents used in osmotic dehydration (Silva, Fernandes and Mauro, 2014). Considering studies, osmotic dehydration causes partial removal of water and influences the shortening of drying time. However, drying time may also be extended due to the increase in the internal resistance to mass transfer depending on the solid uptake of the material during osmotic dehydration (Nowacka *et al.*, 2017). Therefore, osmotic dehydration parameters should carefully be determined in order to restrict solid uptake and increase the subsequent drying efficiency.

2.2 Sorption Characteristics

Moisture migration in food materials plays an important role in physical and chemical changes, thus affecting the shelf life of the product. It is difficult to characterize moisture migration in dry foods, due to the reason it depends on a number of moisture transfer mechanisms and material microstructure. Moisture sorption isotherms are useful tools in predicting potential changes during storage and provides information about the changes related to thermodynamics of the system. Empirical and semi-empirical models such as GAB, BET, Oswin, Handerson, Caurie, Lewicki and Peleg are proposed to describe the moisture sorption characteristics of foods (Yang *et al.*, 2017). Sorption isotherm for a typical food material is given below in Figure 2.5.

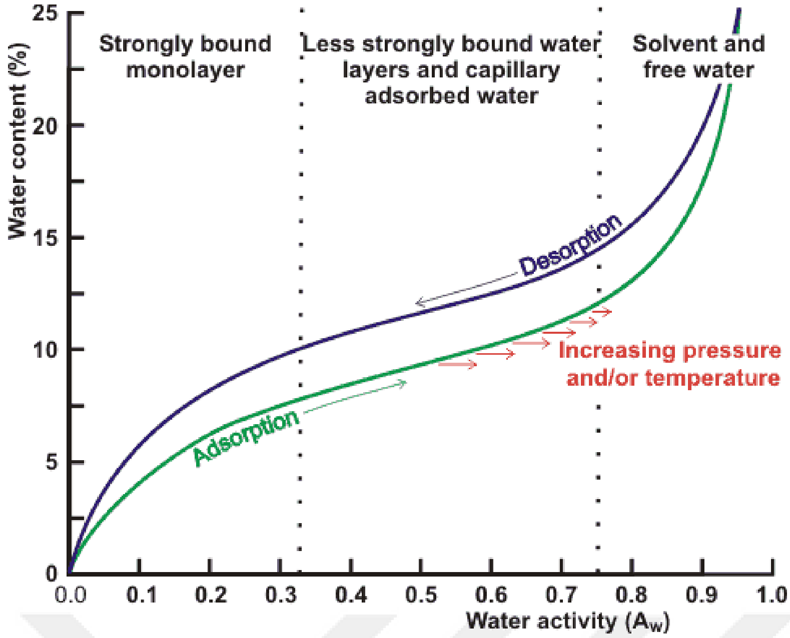

Figure 2.5: Sorption isotherm for a typical food material (Chaplin, 2016).

Typically, moisture sorption isotherms describe the relationship between water activity and the moisture content expressed in dry basis of a product at a given temperature. These isotherms are widely used to predict shelf life stability or to optimize drying processes and packaging. Most common method to build a sorption isotherm is to store samples in desiccators or dynamic sorption devices until equilibrium reached. Consequently, water activity and moisture content are assessed, and the moisture sorption isotherm is plotted (Dupas-Langlet *et al.*, 2016).

2.3 Thin Layer Drying Kinetics

Drying kinetics of food materials is a complex subject and requires dependable models to describe drying behavior. Experimental studies are conducted and drying models are applied to predict drying behavior (Pardeshi, Arora and Borker, 2009). Thin layer equation are used to describe drying phenomena in a united way and being used to estimate drying time and generalize drying curves for several food products (Kadam, Goyal and Gupta, 2011). Gravimetric methods are used to measure drying kinetics and several simplified or complex models are used to describe drying behavior such as Lewis, Page, Henderson & Pabis, and Logarithmic models (Table 2.1).

Table 2.1: Mathematical models used to describe drying behavior.

Model	Mathematical Formulation	Reference
Lewis	$MR=exp(-kt)$	(Lewis, 1921)
Page	$MR=exp(-kt^n)$	(Lewis, 1921)
Handerson and Pabis	$MR=a\ exp(-kt))$	(Henderson and Pabis, 1961)
Logarithmic	$MR=a\ exp(-kt)+c$	(Yaldiz, Ertekin and Uzun, 2001)

A typical drying curve is given below to illustrate the food drying operation kinetics (Figure 2.6). As seen from the curve, there are three phases in process of drying; first falling drying rate period (C-D), second falling rate period (D-E), and constant drying rate period (B-C) (Naewbanij and Thepent, no date).

In the constant rate period, water migrates to the surface of the material and evaporaion takes place in high rate constantly until reaching critical moisture content. When the migration from the inner parts of the material to the surface is slower than the evaporation from the surface, the falling rate period starts. During this period the drying rate decreases dramatically, surface temperature incrases and the removal of water takes longer times. At the equlibrium moisture content, drying rate reduces to zero (Barbosa-Canovas and Vega-Mercado, 1996).

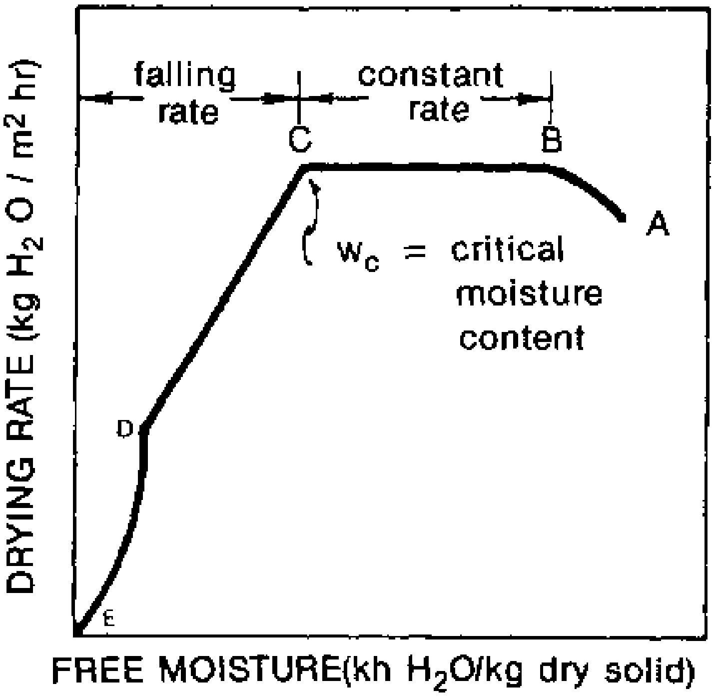

Figure 2.6: Illustration of constant and falling rate drying periods on a drying curve (Naewbanij and Thepent, no date).

2.4 Energy Efficiency

The growth of global population increased the demand for food production and consumption. Rising energy costs and continuous energy consumption by industrial production of every kind of goods leads the industries to search for methods to decrease energy consumption. European Commission is working towards environmental safety with emphasis on energy efficiency and provides customers with information that allows them to choose more energy efficient products (Minetto, Rossetti and Marinetti, 2017). Drying operations are highly energy dependent processes, thus increasing the efficiency of these processes and decreasing the energy consumption is highly essential.

3. MATERIALS AND METHODS

3.1 Materials

Fresh thyme (*Thymus vulgaris*) samples used in this work were purchased from a local farmer’s market in Istanbul, where goods were sold directly after harvest. Samples were not stored, experiments initiated right after receiving. Initial moisture content was determined by using standard method at 70°C under vacuum for 12 hours until constant weight is achieved and calculated as 89.8% (AOAC, 2002). Fresh samples were firstly washed in a centrifugal vegetable washer-drier and excess washing water removed. Remaining washing water was removed by gently wiping the sample at room temperature. After cleaning up process, leaves of thyme samples were picked up from its branches to obtain the edible portion. 10 ± 0.5 grams of thyme leaves were spread on glass petri dishes with 10 cm diameter, right before drying operation. Thickness of the material should be reduced to dimensions that will provide uniform distribution of drying air and temperature over the material thus making lumped parameters suitable for thin layer drying concept (Onwude *et al.*, 2016). Due to the natural thickness of thyme leaves, when spread as one layer on dishes, it can be considered as a slab having moisture distributed uniformly.

3.2 Methods

3.2.1 Drying procedure

The air drying process was conducted in a conventional drying oven (Memmert UNB 400) at four different temperatures (50, 60, 70, and 80 °C). Three replicates of samples were prepared for each temperature. Dishes were placed in pre-heated oven and weight differences were measured by particular time intervals. Measurements were made in 10-minute time intervals for the first 150 minutes of drying. After 2.5 hours of drying, measurements were made in 30-minute time intervals until constant weight was obtained in three consecutive measurements for each replicate.

The microwave drying process was conducted in a 1300 W microwave oven (Arçelik) at three different power levels (30% - 390 W, 50% - 650 W, and 70% - 910 W). Three replicates of samples were prepared for each temperature. Dishes were placed in microwave oven and weight differences were measered by 30-second time intervals until constant weight was obtained in three consecutive measurements for each replicate.

3.2.2 Drying kinetics

Drying kinetics were characterized during dehydration of pre-treated or non-treated samples and compared. Drying curves were plotted as a function of moisture ratio and time. Moisture ratio (MR) is a dimensionless water content function calculated as following:

$$MR = \frac{M_t - M_\infty}{M_0 - M_\infty} \quad (3.1)$$

Where M_t is the moisture content at process time t, M_0 is the initial moisture and M_∞ is the equilibrium moisture content (Ricce *et al.*, 2016).

Obtained drying curves were evaluated using appropriate drying models. Best fitting model determined for each treatment by evaluating different models which are excessively used in the literature. Five following models were evaluated and fitted to data, namely, Lewis (Newton), Page, Henderson and Pabis, Logarithmic, and Midilli models.

The Henderson-Pabis model is the first term of the general series solution of Fick's second law of diffusion (Henderson and Pabis, 1961). The analytical solution of Fick's second law for infinite slab or sphere is given below:

$$MR = A_1 \sum_{i=1}^{\infty} \frac{1}{(2i-1)} exp\left[-\frac{(2i-1)^2 \pi^2 D_{eff}^{t}}{A_2}\right] \quad (3.2)$$

Where D_{eff} is the effective diffusivity (m^2/s), A_1 and A_2 are the geometric constants.

The first term of the series solution can be used for sufficiently long drying times (Erbay and Icier, 2010) and food drying is especially very complex because of the differential structure of products. In practice, a food dryer is considerably more complex than a device that merely removes moisture, and effective models are necessary for process design, optimization, energy integration, and control. Although modeling studies in food drying are important, there is no theoretical model which neither is practical nor can it unify the calculations. Therefore, the experimental studies prevent their importance in drying and thin layer drying equations are important tools in mathematical modeling of food drying. They are practical and give sufficiently good results (Erbay and Icier, 2010). Geometric constants have different values depending on the product geometry, which are given in Table 3.1.

Table 3.1: Geometric constants according to the product geometry.

Product geometry	A_1	A_2
Infinite slab	$8/\pi^2$	$4L^2$
Sphere	$6/\pi^2$	$4r^2$

L – thickness of the slab when the drying occurs on one side

The first term of the equation can be written in a simplified form as below, which is referred to as the Henderson and Pabis model:

$$MR = a\exp(-kt) \tag{3.3}$$

Where *a* is the model constant (dimensionless), *k* is the drying constant (h^{-1}), *t* is the time (h).

The Lewis model is a special case of the Henderson and Pabis model where the food material is assumed to be thin enough and the air conditions are kept constant during operation (Lewis, 1921). The model equation is given below:

$$MR = \exp(-kt) \tag{3.4}$$

The Page model is an empiric modification of the Lewis model, where the constant *n* was added to the equation to increase accuracy (Page, 1949):

$$MR = \exp(-kt^n) \tag{3.5}$$

The logarithmic model is the modified form of the Henderson and Pabis model with the addition of an empirical constant (Yagcioglu *et al.*, 1999):

$$MR = a\exp(-kt) + c \tag{3.6}$$

where *c* is the dimensionless model constant.

The Midilli model is given below, where *b* is the dimensionless model constant (Nayak *et al.*, 2021):

$$MR = aexp^{-(kt^n)} + bt \tag{3.7}$$

The model parameters were evaluated by non-linear regression analysis with the Levenberg-Marquardt procedure in Statistica software (StatSoft Inc., USA). The goodness of fit of the curves was evaluated by using the coefficient of determination (R^2) primarily. Other criteria used to determine the most suitable drying model were root mean square error (*RMSE*) and reduced chi-square ($\chi 2$). These values were calculated by using the equations below. The higher R^2 values and lower $\chi 2$ and *RMSE* values indicate the goodness of fit (Doymaz, 2006).

$$\chi^2 = \frac{\sum_{i=1}^{N}(MR_{exp,i}-MR_{pre,i})^2}{N-z} \tag{3.8}$$

$$RMSD = \left[\frac{1}{N}\sum_{i=1}^{N}\left(MR_{exp,i} - MR_{pre,i}\right)^2\right]^{1/2} \tag{3.9}$$

Where N is the number of observations, z is the number of constants in the model, $MR_{exp,i}$ is the experimental moisture ratio, $MR_{pre,i}$ is the predicted moisture ratio at the i^{th} observation.

3.2.3 Calculation of effective diffusivity and activation energy

Diffusion of moisture in solids during drying is a complex process. Surface diffusion, capillary flow, molecular diffusion and/or Knudsen flow takes place along with drying. So, these phenomena are combined into one term, with a lumped parameter concept, namely effective moisture diffusivity (D_{eff}) (Erbay and Icier, 2010). The first term of the diffusion equation is used to calculate the effective diffusivity for a slab:

$$\boldsymbol{MR = \frac{8}{\pi^2} exp\left[-\frac{\pi^2 D_{eff} t}{4L^2}\right]} \tag{3.10}$$

Ln(MR) versus time curve is plotted according to the equation, and the effective diffusivity can be calculated via the slope of the curve (k) as below:

$$k = -\frac{\pi^2 D_{eff}}{4L^2} \tag{3.11}$$

Temperature has a significant effect on the effective diffusivity. This effect can be described by an Arrhenius equation:

$$D_{eff} = D_0 \exp\left(-10^3 \frac{E_a}{R(T+273.15)}\right) \tag{3.12}$$

where, D_0 is the Arrhenius factor, E_a is the activation energy for diffusion (kJ/mol) and R is the universal gas constant (kJ/kmol.K). The slope of the ln(D_{eff}) versus [1/(T+273.15)] straight line is equal to ($-10^3 \times E_a/R$) according to the equation.

3.2.4 Pre-treatments

3.2.4.1 Ultrasound treatment

Ultrasound treatments were applied using a 40 kHz bath sonicator (Digital Pro) with 240 W ultrasonic power for 5, 10, and 15 minutes. Three replicates were conducted for each level. Air drying or microwave drying was applied after removing the excess water on the samples.

3.2.4.2 Blanching treatment

Firstly, samples were exposed to boiling water for 1, 2, and 3 minutes and then rapidly cooled by placing them in ice water. Three replicates were conducted for each level. Air drying or microwave drying was applied after removing the excess water on the samples.

3.2.5 Color measurements

Color measuremets of thyme leaves were conducted by using a Hunter colorimeter (Konica Minolta). Before the mesurements colorimeter was adjusted with a standard white surface (L* = 97.52, a* = -0.03, b = 1.91). Where L* is the lightness, a*/-a* is the redness/greenness, and b*/-b* is the yellowness/blueness. The experiments were conducted at room temperature with three replicate. Total color (E*) and chroma (C*) were calculated by following formula;

$$\boldsymbol{E}^{*} = \sqrt{\boldsymbol{L}^{*2} + \boldsymbol{a}^{*2} + \boldsymbol{b}^{*2}} \tag{3.13}$$

$$\boldsymbol{C}^{*} = \sqrt{\boldsymbol{a}^{*2} + \boldsymbol{b}^{*2}} \tag{3.14}$$

3.2.6 Statistical analysis

All experiments were conducted as triplicate sets unless it was mentioned otherwise. The results were expressed as mean±standard deviation. The data were statistically analyzed and the differences among experimental groups were investigated by one-way analysis of variance (ANOVA) at α = 0.05 significance level, and the means were compared with the Tukey test, using Minitab (Minitab, 2019) program.

4. RESULTS AND DISCUSSION

4.1 Thin Layer Drying of Thyme Leaves

4.1.1 Air drying

Thin layer drying operations were carried out in four different temperatures (50 – 80 °C), moisture loss values were calculated after constant weight obtained. Change of moisture ratio with time and the change of drying rate with moisture ratio were expressed. Moisture ratio and drying rate change during drying period of thyme leaves were given in Figures 4.1 and 4.2. As can be seen on figures, drying rate increases significantly as the temperature rises from 50 to 80 °C, and the drying time declines proportionally. It is also seen that the constant rate drying period does not exist. Drying rate continuously decreases with time. Similar results were reported for agricultural products such as red pepper (Akpinar, Bicer and Yildiz, 2003), mint leaves (Doymaz, 2006) and pumpkin (Hashim, Daniel and Rahaman, 2014). The falling rate behavior of drying rate indicates that, diffusion is the dominant physical mechanism for the movement of moisture during drying process (Onwude *et al.*, 2016). Moisture ratio and drying rate deviations along three replicates were given as error bars in the figures.

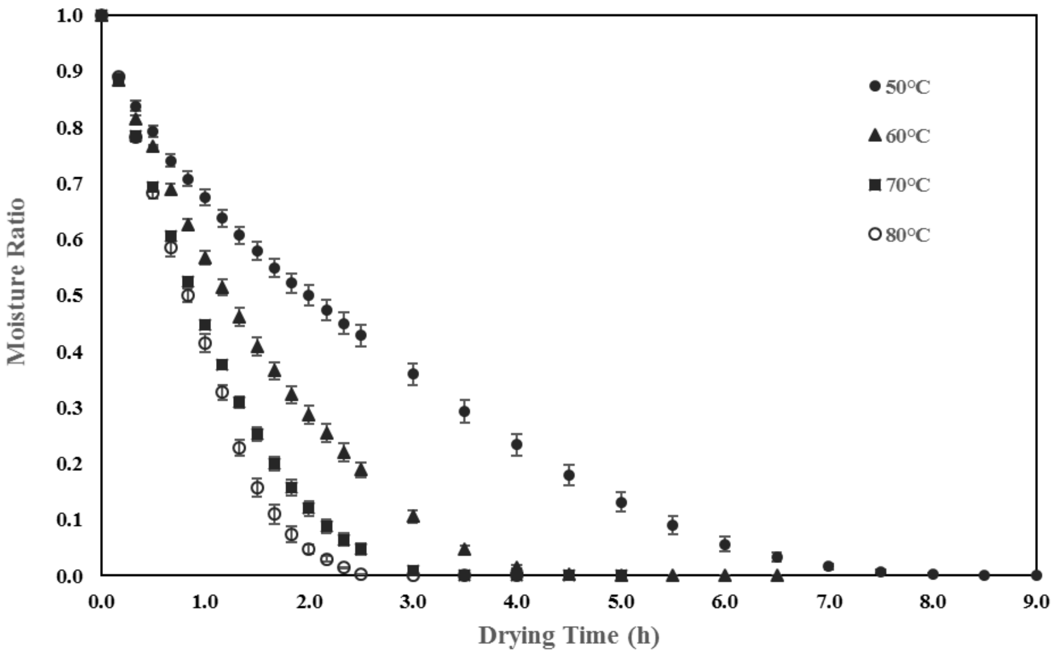

Figure 4.1: Change of moisture ratio with time at different temperatures.

As seen on Figure 4.2, drying rate increases with the increase of drying temperature and the highest rate of drying was achieved at 80 °C as expected. Also the drying rate decreases with time as the moisture ratio drops. Duration of the drying operation to reduce the moisture content to a desired level is directly dependent on drying temperature. Many researchers suggested corresponding results for food materials such as gundelia (Evin, 2011), araticum (Botrel *et al.*, 2016) and pumpkin (Hashim, Daniel and Rahaman, 2014).

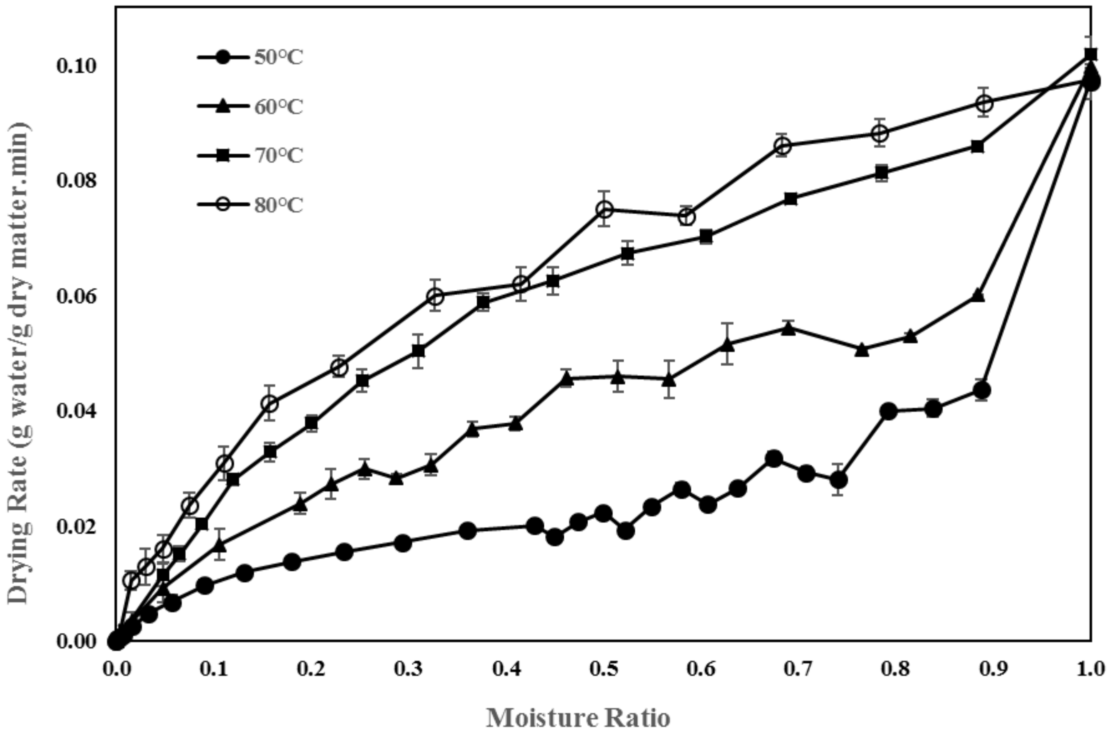

Figure 4.2: Change of drying rate as a function of moisture ratio.

4.1.2 Modelling of drying curves

It is a matter of great importance to effectively model the drying behavior of thyme leaves in order to determine the moisture content as a function of drying time. Widely used semi-theoretical thin layer drying model equations were fitted to the experimental data to achieve the most accurate model. Coefficient of determination values were calculated to evaluate the goodness of fit. Statistical criteria to evaluate the goodness of fit; coefficient of determination (R^2), root mean square error (RMSE) and chi-square (χ^2), and the drying model constants; *k*, *a*, *n* and *c* were given in Table 4.1.

The drying models presented high coefficient of determination values (R^2), ranging from 0.9644 to 0.9976. So, it can be said that all the models applied in this work can describe the characteristics of drying. The least values were observed at 80 °C, and with the Lewis model.

As seen on Table 4.1, the values of R^2 obtained from models Page and Logarithmic were higher than the others. Logarithmic model shows higher R^2 values at lower temperatures (50 and 60 °C) while the Page model have higher values of R^2 at higher temperatures (70 and 80 °C). Comparing the RMSE and the chi square values, identical conclusions can be made as R^2 values. As stated above, the Page model shows a higher χ^2 values at 50 °C and the logarithmic model shows a higher χ^2 value at 80 °C, which means a deviation from the model. Experimental moisture ratios were compared to the calculated values obtained from the models and shown in Figures 4.3 and 4.4 to visualize the fit of the models. Comparison of the calculated values of the Page model with the experimental data shown in Figure 4.3, and the comparison of the calculated values of the logarithmic model with the experimental data shown in Figure 4.4. As seen on the figures, both models show a good fit to the experimental values. Considering higher temperatures, the Page model shows a better fit to actual data while the Logarithmic model shows a slightly better fit at lower temperatures. Consequently, it can be said that the Page and the logarithmic models can present the thin layer drying behavior of thyme leaves.

Table 4.1: Statistical criteria and model constants for different models.

Model Name	Temperature (°C)	Model Constants *k*	*a*	*n*	*c*	Statistical parameters R^2	RMSE	χ^2
Lewis	50	0.37883				0.9879	0.0335	0.00117
	60	0.62701				0.9892	0.0323	0.00109
	70	0.90250				0.9836	0.0401	0.00169
	80	1.03823				0.9644	0.0618	0.00403
Henderson and Pabis	50	0.36892	0.97974			0.9886	0.0327	0.00115
	60	0.64781	1.02972			0.9902	0.0306	0.00102
	70	0.95418	1.05813			0.9872	0.0354	0.00139
	80	1.12155	1.08788			0.9722	0.0547	0.00334
Page	50	0.36691		1.03353		0.9883	0.0330	0.00117
	60	0.57377		1.17399		0.9957	0.0204	0.00045
	70	0.84466		1.27706		0.9976	0.0155	0.00026
	80	0.97639		1.46334		0.9954	0.0221	0.00055
Logarithmic	50	0.28559	1.05839		-0.10993	0.9966	0.0178	0.00035
	60	0.55690	1.07554		-0.06567	0.9959	0.0199	0.00045
	70	0.83065	1.09909		-0.05898	0.9928	0.0266	0.00083
	80	0.90416	1.15851		-0.09706	0.9840	0.0414	0.00204

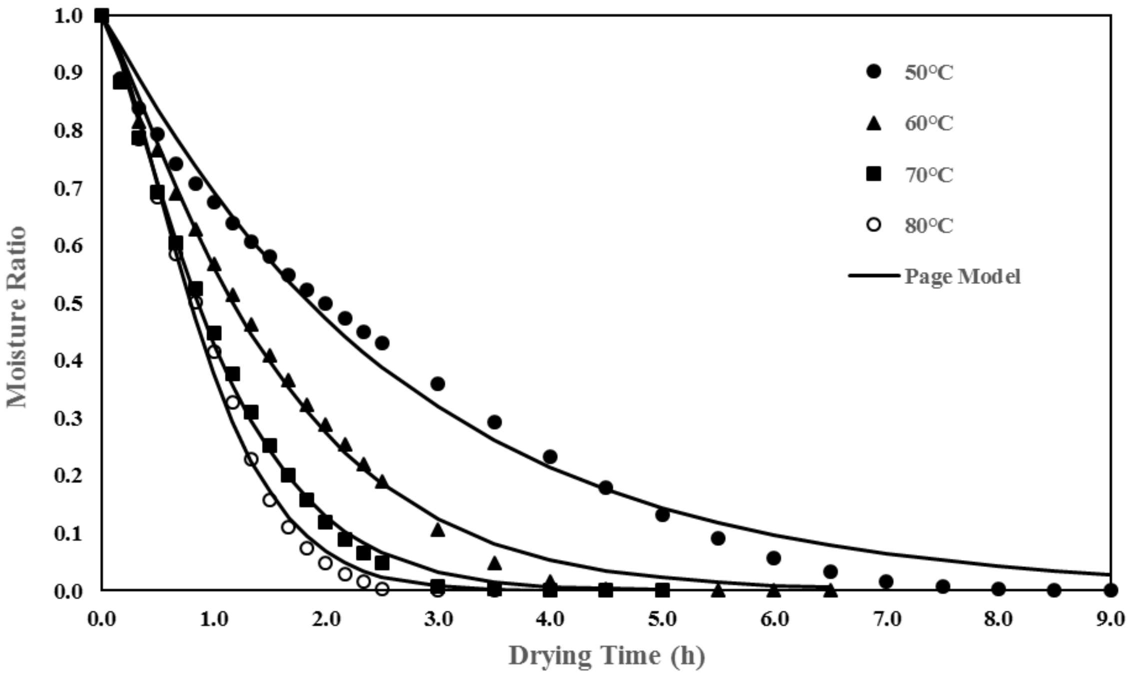

Figure 4.3: Comparison of experimental data and calculated moisture ratios via Page model.

Various studies show the goodness of fit of the Page model and the logarithmic for different food materials. In a study conducted on pumpkin showed that both Page model, and the logarithmic model have a good fit to the experimental values; where also the Page model had a better fit at higher temperatures (60 and 70 °C) and the logarithmic model had a slightly better fit at lower temperatures (40 and 50 °C) considering the R^2 values (Guiné, Pinho and Barroca, 2011). Also, the Page model shows a good fit for the drying of olive cake at different temperatures except at 50 °C (Akgun and Doymaz, 2005) and both Page and logarithmic models were found to be suitable for the drying of banana slices (Doymaz, 2010).

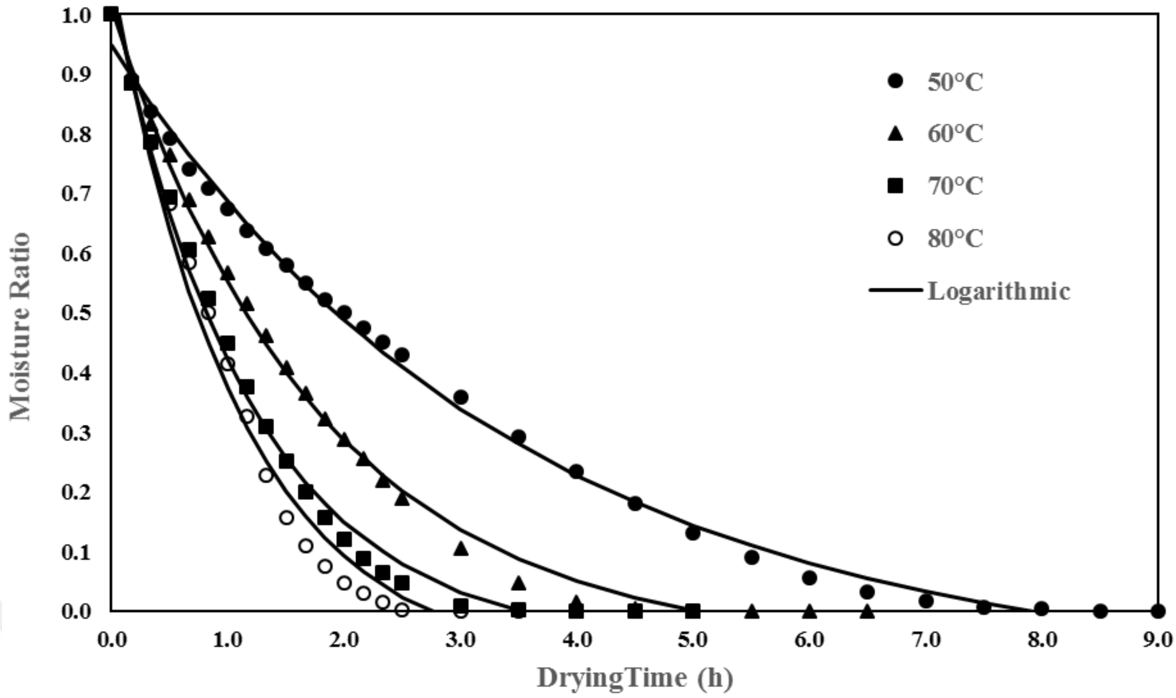

Figure 4.4: Comparison of experimental data and calculated moisture ratios via logarithmic model.

4.1.3 Effective diffusivity and activation energy

Effective diffusivities were calculated for the thin layer drying of thyme leaves and given in Table 4.2. Values of D_{eff} ranges between 2.59×10^{-9} to 1.28×10^{-8} m^2/s for 50 to 80 °C. Studies conducted on the thin layer drying kinetics of fruits and vegetables show that effective diffusivity varies between 10^{-12} to 10^{-6} m^2/s, and for the most samples (80%), D_{eff} value is in the region 10^{-11} to 10^{-8} m^2/s (Onwude *et al.*, 2016) . As seen on Table 4.2, D_{eff} values increased highly with increasing temperature, as literature suggests. An increase in the effective diffusivity with increasing temperature were observed in various studies conducted on different food materials, such as carrot (Kumar, Sarkar and Sharma, 2012), jackfruit (Saxena and Dash, 2015) and green peas (Pardeshi, Arora and Borker, 2009).

Table 4.2: Effective diffusivity values for drying of thyme leaves at different temperatures.

Temperature (°C)	Effective diffusivity (m^2/s)
50	2.59×10^{-9}
60	5.18×10^{-9}
70	7.69×10^{-9}
80	1.28×10^{-8}

As seen above, temperature has a significant effect on the effective diffusivity and this effect is described by an Arrhenius equation (Equation 3.10). From this statement, the activation energy for thin layer drying of thyme was calculated as 21.40 kJ/mol. The activation energy shows the sensibility of effective diffusivity to temperature, where higher E_a indicates more sensibility to air temperature. The activation energy for the drying of thyme is found to be lower than most of the other food products studied, such as basil (33.21 kJ/mol)(Kadam, Goyal and Gupta, 2011), mint (82.93 kJ/mol) (Jin Park, Vohnikova and Pedro Reis Brod, 2002), and pumpkin (35.6 kJ/mol) (Perez and Schmalko, 2009).

4.2 Thin Layer Drying of Thyme Leaves with Pre-treatments

4.2.1 Drying kinetics of air drying

Drying operations were conducted at 60, 70 and 80 °C air temperatures; moisture ratio change over time during 60 °C drying following blanching and ultrasound pre-treatments are given in Figure 4.5. 2 and 3 minutes of blanching both showed a similar and linear moisture ratio decrease during dehrydration, also showed significant reduction on drying time with respect to 1 minute of blanching. However, while ultrasound treatment had an impact on reducing drying time, level of treatment did not show a significant influence. Total dehydration required approximately 4.5 hours for non-teated samples at 60 °C while blanching and ultrasound pre-treatments present 44% and 33% reduction in drying time, respectively, until final moisture ratio.

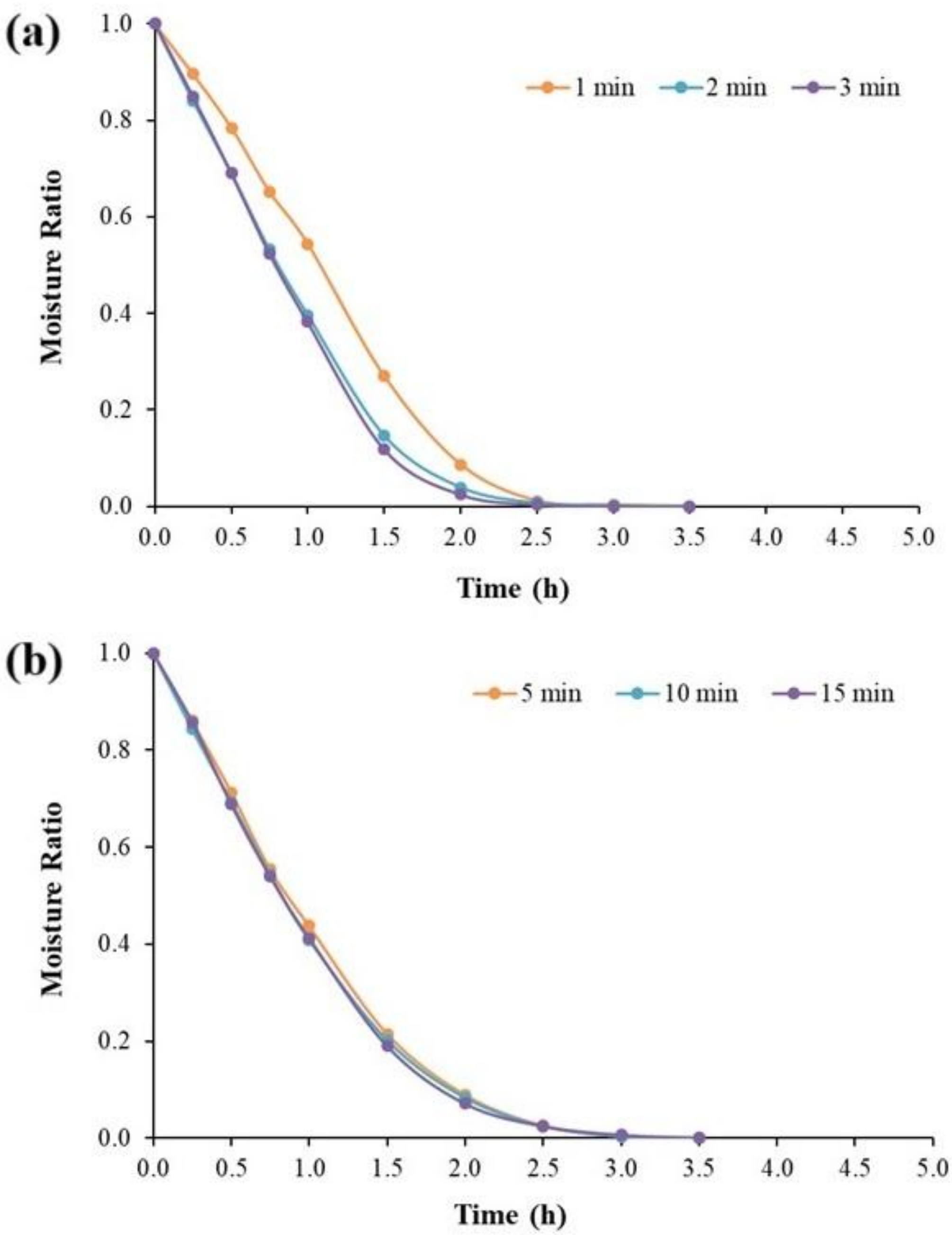

Figure 4.5: Change of moisture ratio with time at 60°C air drying (a) with blanching pre-treatment (b) with ultrasound pre-treatment.

Figure 4.6 represnts the change of moisture ratio over time at 70 °C air drying subsequent to pre-treatments. 2 and 3 minutes of blanching pre-treatments show a significant reduction in time compared to 1 minute of blanching, while 3 minute of blanching applied samples showed a paced dehydration through the beginning stage but considering the full period, these two applications did not have a considerable difference. Ultrasound pre-treatment also presents high reduction on drying time, where 15 minutes of treatment had a clear otcome related to other applications. Drying time was determined as 3.5 hours without any pre-treatments. Blanching and ultrasound treatments showed a 43 to 57 % and 37 to 54 % reduction on dyring time, respectively, a decline to approaching 1.5 hours.

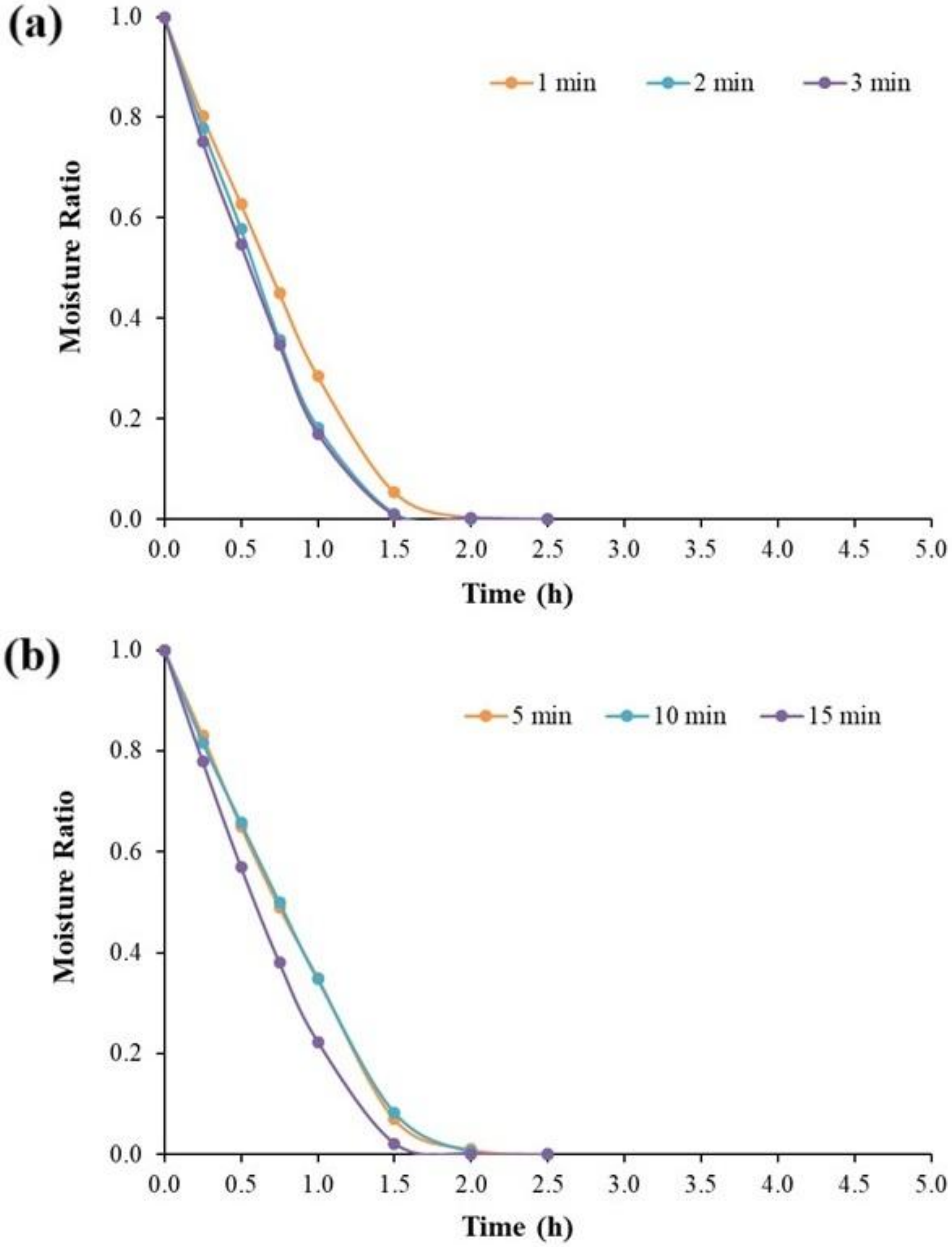

Figure 4.6: Change of moisture ratio with time at 70°C air drying (a) with blanching pre-treatment (b) with ultrasound pre-treatment.

Pre-treatments at 80 °C significantly reduces the drying time of thyme leaves from 2.5 hours to 1.5 hours, which corresponds to 40% lower drying durations. Effects of pre-treatments on drying time were given in Figure 4.7, where the levels of pre-treatments have not showed a significant difference. This may be due to the very high level of temperature, where the rate of mass transfer is substantially extreme and the alterations of plant texture as a result of pre treatments would not have a critical difference to one another to match the momentum of moisture transfer.

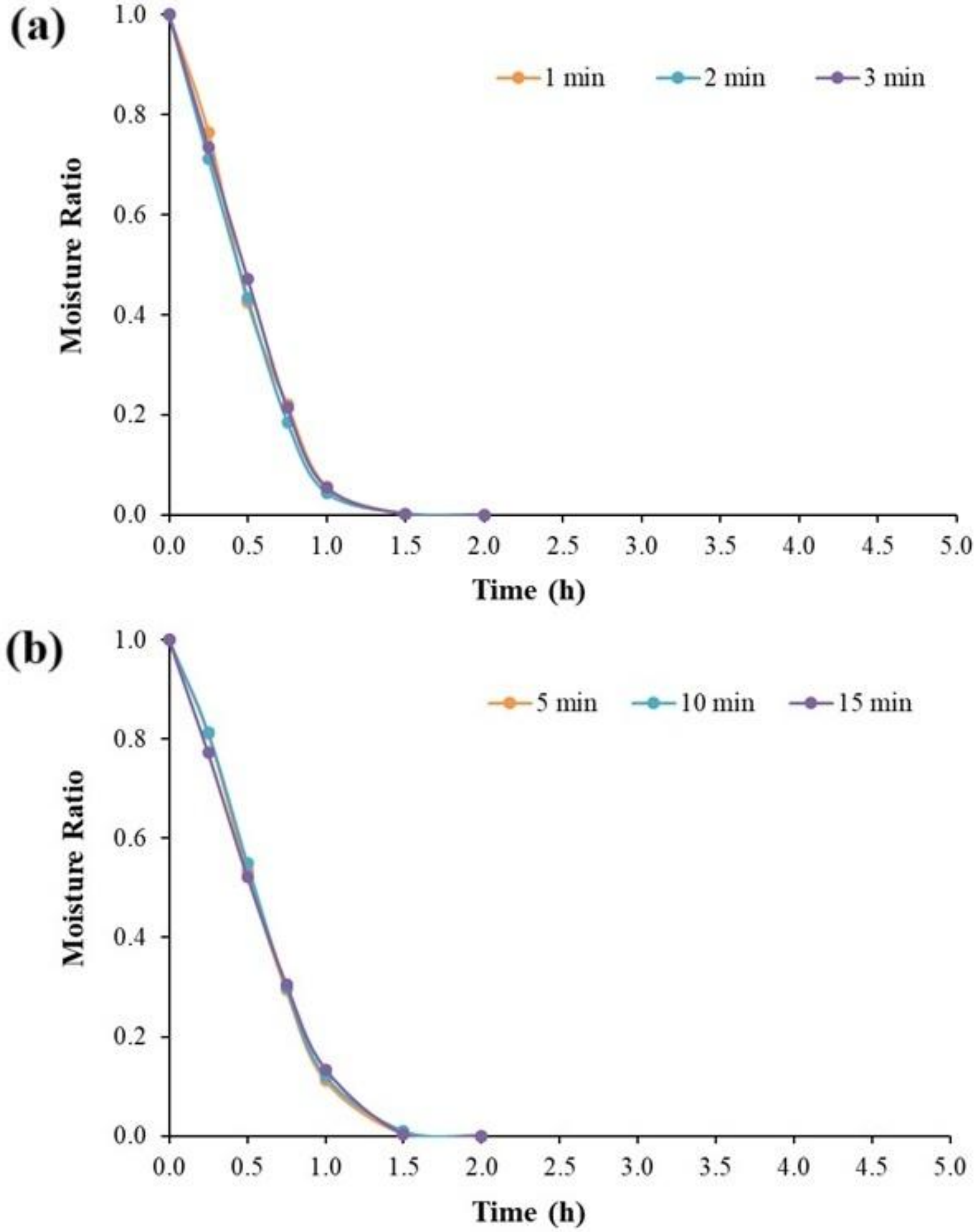

Figure 4.7: Change of moisture ratio with time at 80°C air drying (a) with blanching pre-treatment (b) with ultrasound pre-treatment.

4.2.1.1 Modelling of drying curves

Dehydration behaviour of food materials are modelled to effectively show the mathematical estimation of moisture ratio as a function of time. Semi-empirical models were developed in order to represent this relation of moisture ratio and time. Such models were previously used in this study, as Lewis, Henderson and Pabis, Page, and logarithmic models. These were succesfully implemented to demonstrate the drying behaviour of non-treated thyme leaves. For the dehydration of pre-treated samples, constant ‘c’ of logarithmic model was mostly calculated as zero, so the model was reduced to Henderson and Pabis model. Therefore, Midilli model was used instead of logarithmic model, which has a higher complexity than other three models and logarithmic model.

Widely used four models studied to fit the drying curves of pre-treated samples, model constants and statistical coefficents were calculated. Statistical coefficients and model constants were given in Table 4.3 for 60°C air drying, Table 4.4 for 70°C and Table 4.5 for 80°C. Best pre-treatment levels were selected for each temperature, experimental data points were plotted and predicted moisture ratios were represented as model lines in figures. Fitting of models at 60 °C air drying are shown in Figure 4.8 (a) for 2 minutes of blanching and (b) for 5 minutes of ultrasound pre-treatments. Coefficent of determination (R^2) for models ranged from 0.9802 to 0.9979 (Table 4.3). Highest level of deviation from observed values is seen at Henderson-Pabis model for blanching pre-treatment. Both Page and Midilli models had a strong fit for pre-treatments where R^2 values are over 0.99**.**

Table 4.3: Comparison of best suited models for thin layer drying of thyme leaves with 60 °C air drying.

Pre-Treatment	Blanching 2 min				Ultrasound 5 min			
Model	**Lewis**	**Henderson**	**Page**	**Midilli**	**Lewis**	**Henderson**	**Page**	**Midilli**
R^2	0.9838	0.9802	0.9977	0.9979	0.9886	0.9845	0.9990	0.9991
RMSE	0.0627	0.0566	0.0184	0.0171	0.0565	0.0497	0.0122	0.0115
χ^2	0.0049	0.0040	0.0004	0.0004	0.0040	0.0031	0.0002	0.0002
k	1.0062	1.0769	1.0021	0.9747	0.9105	0.9821	0.8787	0.8618
a		1.0672		0.9793		1.0669		0.9875
n			1.4743	1.5302			1.4004	1.4297
b				0.0000				0.0000

Table 4.4: Comparison of best suited models for thin layer drying of thyme leaves with 70 °C air drying.

Pre-Treatment	Blanching 2 min				Ultrasound 15 min			
Model	**Lewis**	**Henderson**	**Page**	**Midilli**	**Lewis**	**Henderson**	**Page**	**Midilli**
R^2	0.9803	0.9772	0.9971	0.9972	0.9843	0.9813	0.9971	0.9972
RMSE	0.0638	0.0595	0.0205	0.0197	0.0578	0.0538	0.0204	0.0199
χ^2	0.0054	0.0047	0.0006	0.0005	0.0045	0.0039	0.0006	0.0005
k	1.4345	1.5124	1.6704	1.6594	1.3830	1.4530	1.5479	1.5357
a		1.0566		0.9847		1.0516		0.9867
n			1.5153	1.5557			1.4335	1.4646
b				0.0000				0.0000

Table 4.5: Comparison of best suited models for thin layer drying of thyme leaves with 80 °C air drying.

Pre-Treatment	**Blanching 1 min**				**Ultrasound 5 min**			
Model	**Lewis**	**Henderson**	**Page**	**Midilli**	**Lewis**	**Henderson**	**Page**	**Midilli**
R^2	0.9748	0.9720	0.9990	0.9990	0.9703	0.9657	0.9994	0.9994
RMSE	0.0701	0.0657	0.0116	0.0116	0.0795	0.0735	0.0095	0.0093
χ^2	0.0069	0.0060	0.0002	0.0002	0.0089	0.0076	0.0001	0.0001
k	1.8594	1.9549	2.5882	2.5883	1.5651	1.6684	2.0769	2.0762
a		1.0586		0.9996		1.0713		0.9945
n			1.6323	1.6333			1.7116	1.7277
b				0.0000				0.0000

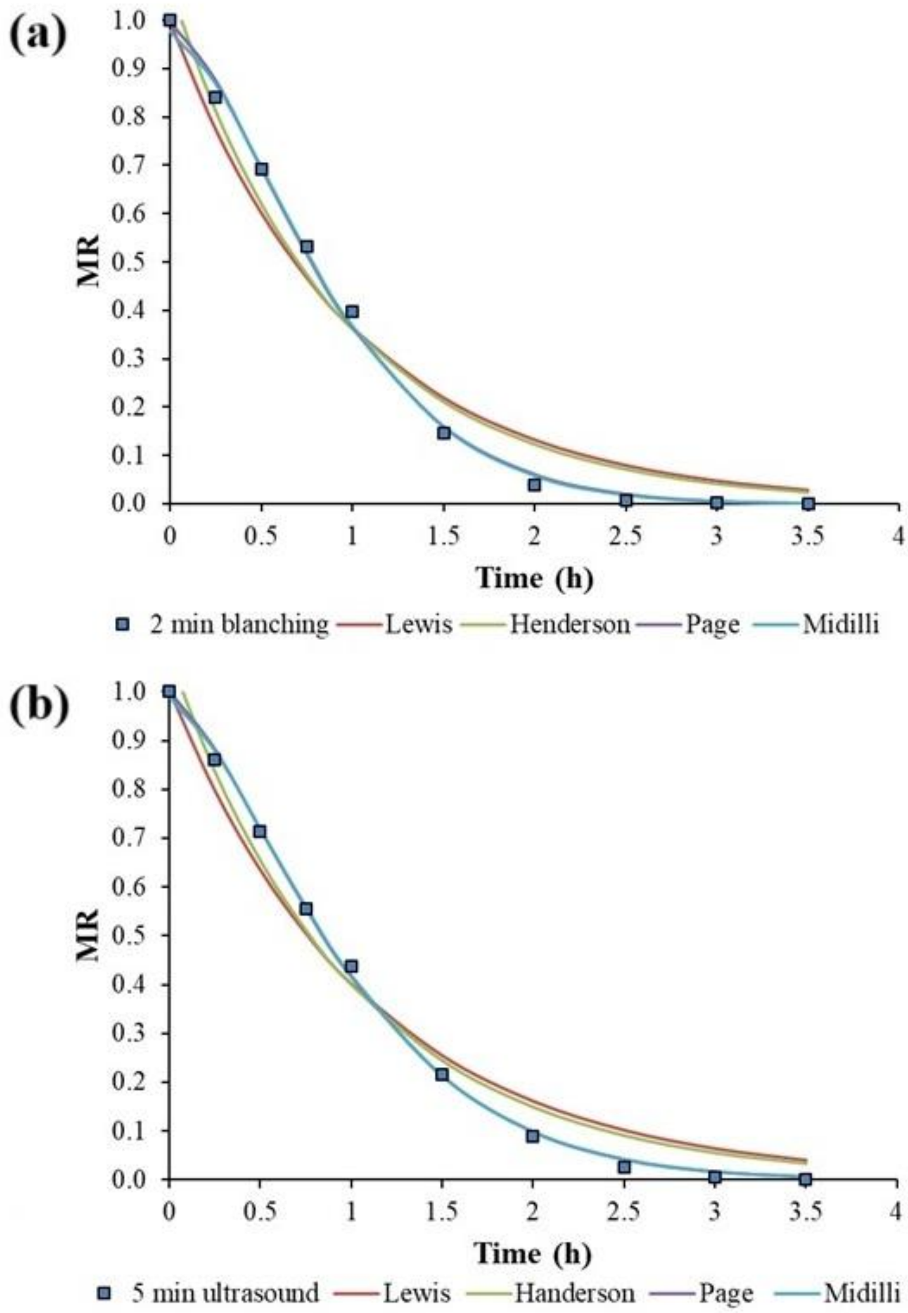

Figure 4.8: Model equation fittings for 60°C air drying data (a) 2 min blanching pre-treatment (b) 5 min ultrasound pre-treatment.

Model fitting coefficients for pre-treated samples air dried at 70 °C can be seen in Table 4.4 and representation of fitting in Figure 4.9 (a) for 2 minutes of blanching and (b) for 15 minutes of ultrasound treatment. R^2 values for these models were calculated in between 0.9772 to. 0.9972. Lowest degree of fitting were seen for Henderson-Pabis model for 2 minutes of blanching followed by Lewis model where R^2 are 0.9772 and 0.9802, respectively. Highest fit were seen for Page and Midilli models with R^2 of 0.9971 and 0.9972.

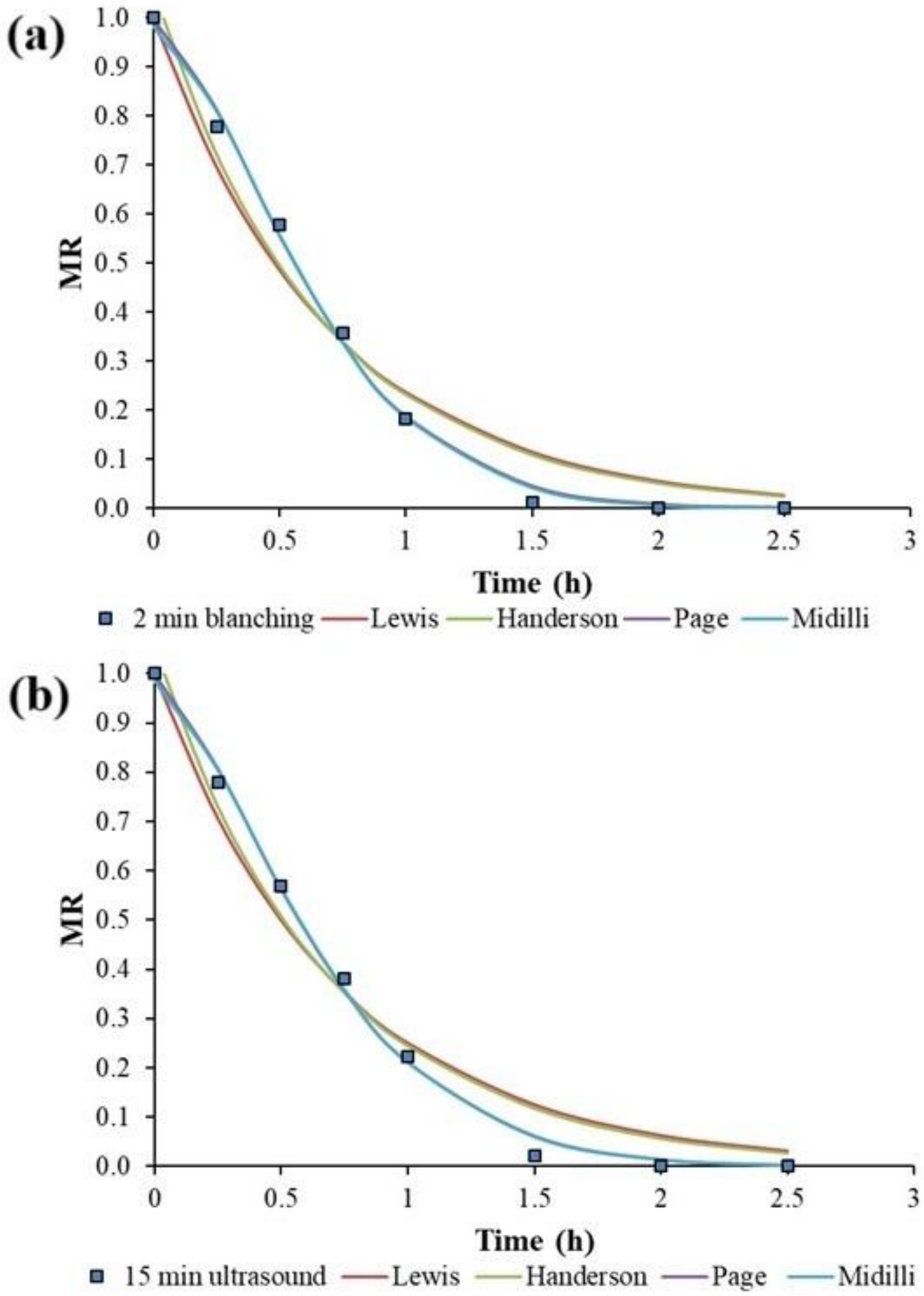

Figure 4.9: Model equation fittings for 70°C air drying data (a) 2 min blanching pre-treatment (b) 15 min ultrasound pre-treatment.

Model equations exhibited a fit similar to previous calculations for 1 minute blanching and 5 minutes ultrasound pre-treated samples air-dired at 80 °C. Representation of the goodness of fit was shown in Figure 4.10. Page model had the closest along with Midilli model with a R^2 of 0.9990 for blanching pre-treatment. Lewis and Henderson-Pabis models had lower fits (0.9703 and 0.9657, respectively) while Page and Midilli models had the closest fits with the R^2 of 0.9994 for 5 minutes of ultrasound pre-treatment. For all treatments, Page and Midilli models showed the highest fit where

Page model is less complex than the Midilli model. Coefficient b of the Midilli model was mostly calculated as zero, thus the model was reduced to $MR = a * e^{-(kt^{n})}$. As instance, for 5 min. of ultrasound treatment, the Page model is $MR = \exp(-2.0769 * t^{1.7116})$ where the Midilli model is $MR = 0.9945 * \exp(-)(2.0762 * t^{1.7277})$.

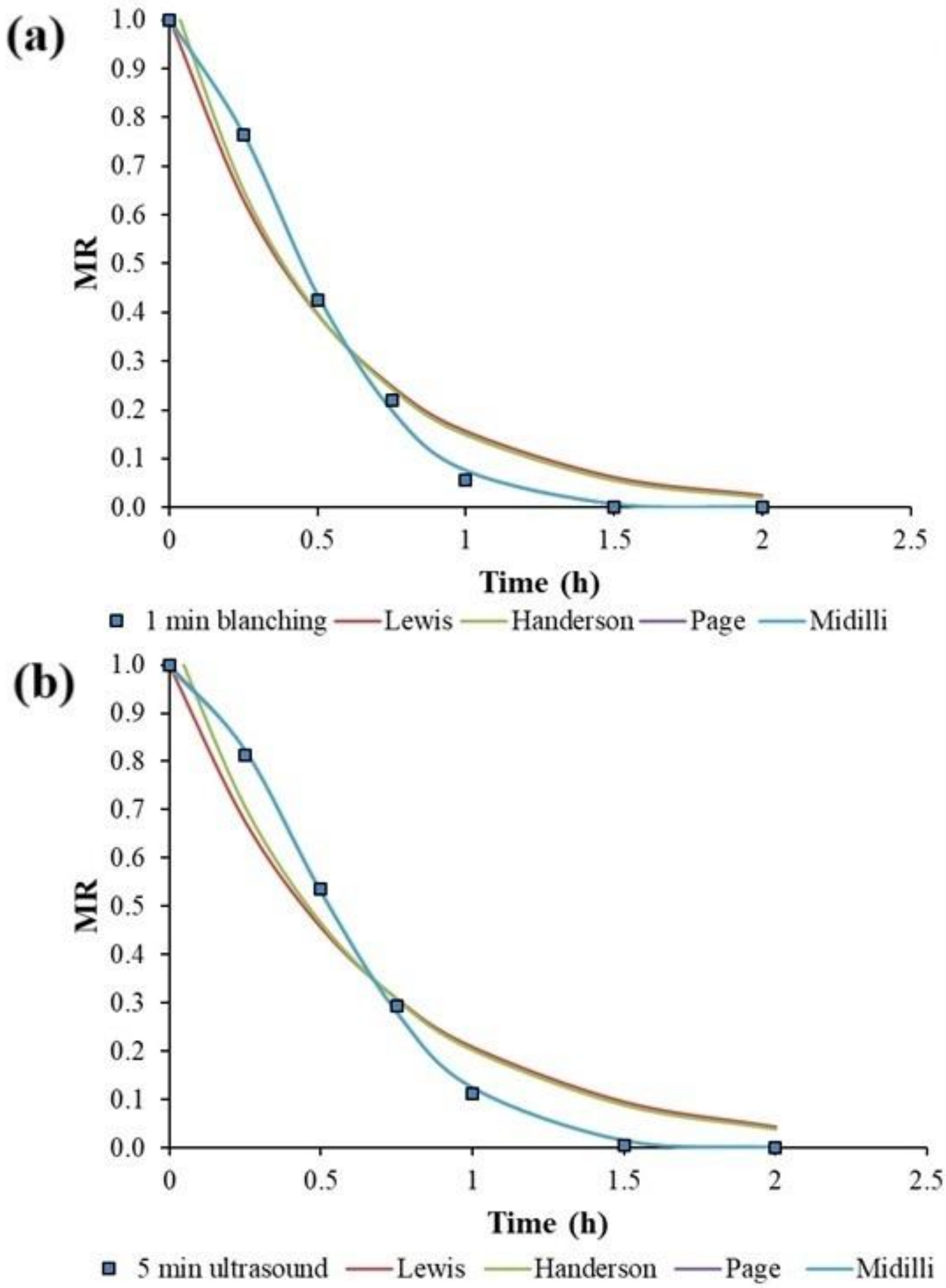

Figure 4.10: Model equation fittings for 80°C air drying data (a) 1 min blanching pre-treatment (b) 5 min ultrasound pre-treatment.

4.2.2 Drying kinetics of microwave drying

Drying kinetics of microwave drying subsequent to pre-treatments have been evaluated at three different levels of microwave power. Levels of power is shown as percentage on microwave ovens, which are 10, 30, 50,70, 100%. In this study 30, 50 and 70% are used which corresponds to 390, 650 and 910 watts on a 1300 W maximum power microwave oven operates at 2450 Mhz. Change of moisture ratio over time for pre-treated samples are given in figures. In figure 4.11, moisture ratio change over time for blanching (a) treated and ultrasound (b) treated samples dried at 30% microwave power are given. Dehydration of thyme samples took 7 minutes for blanched samples and 5 minutes for ultrasound treated samples. 2 and 3 minutes of blanching pre-treatment had the same effect, which were slightly quicker than 1 minute treated samples. On the other hand, ultrasound treated samples did not have any significant difference between each other.

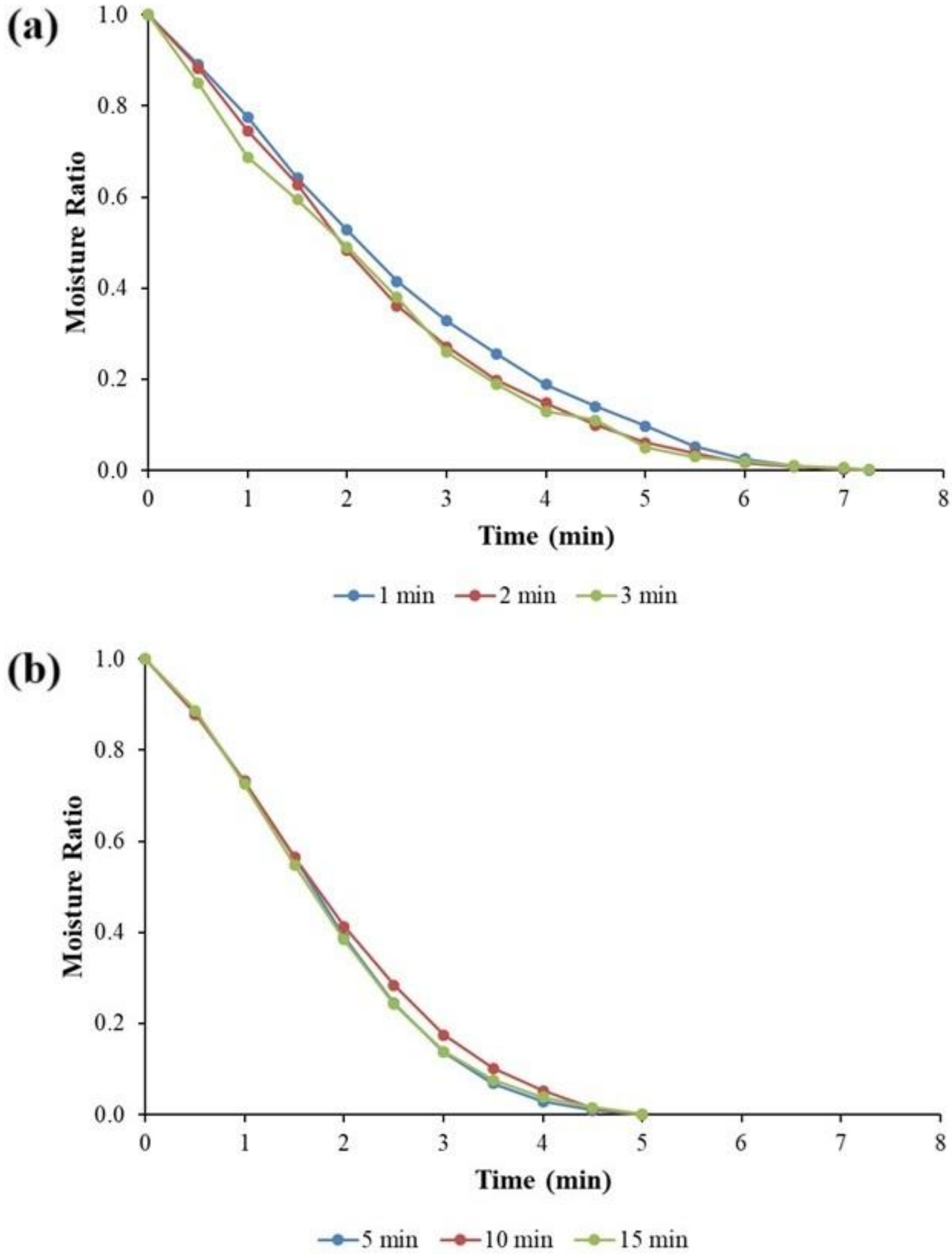

Figure 4.11: Change of moisture ratio with time at 30% microwave drying (a) with blanching pre-treatment (b) with ultrasound pre-treatment.

Moisture ratios of samples dried at 50% microwave power are given in Figure 4.12 for blanching (a) and ultrasound (b) pre-treatments. Complete dehydration took 4 minutes for blanched samples and 3.5 minutes for ultrasound treated samples. Similar to of 30% power, blanched samples at 50% power had an almost identical behavior where 2 and 3 minutes applications are same and slightly swift than 1 minutes of treatment. On the other hand, ultrasound treated samples showed a more adequate behaviour, where 15 minutes of treatment demonstrates a faster dehydration then respectively 10 and 5 minutes of treatments.

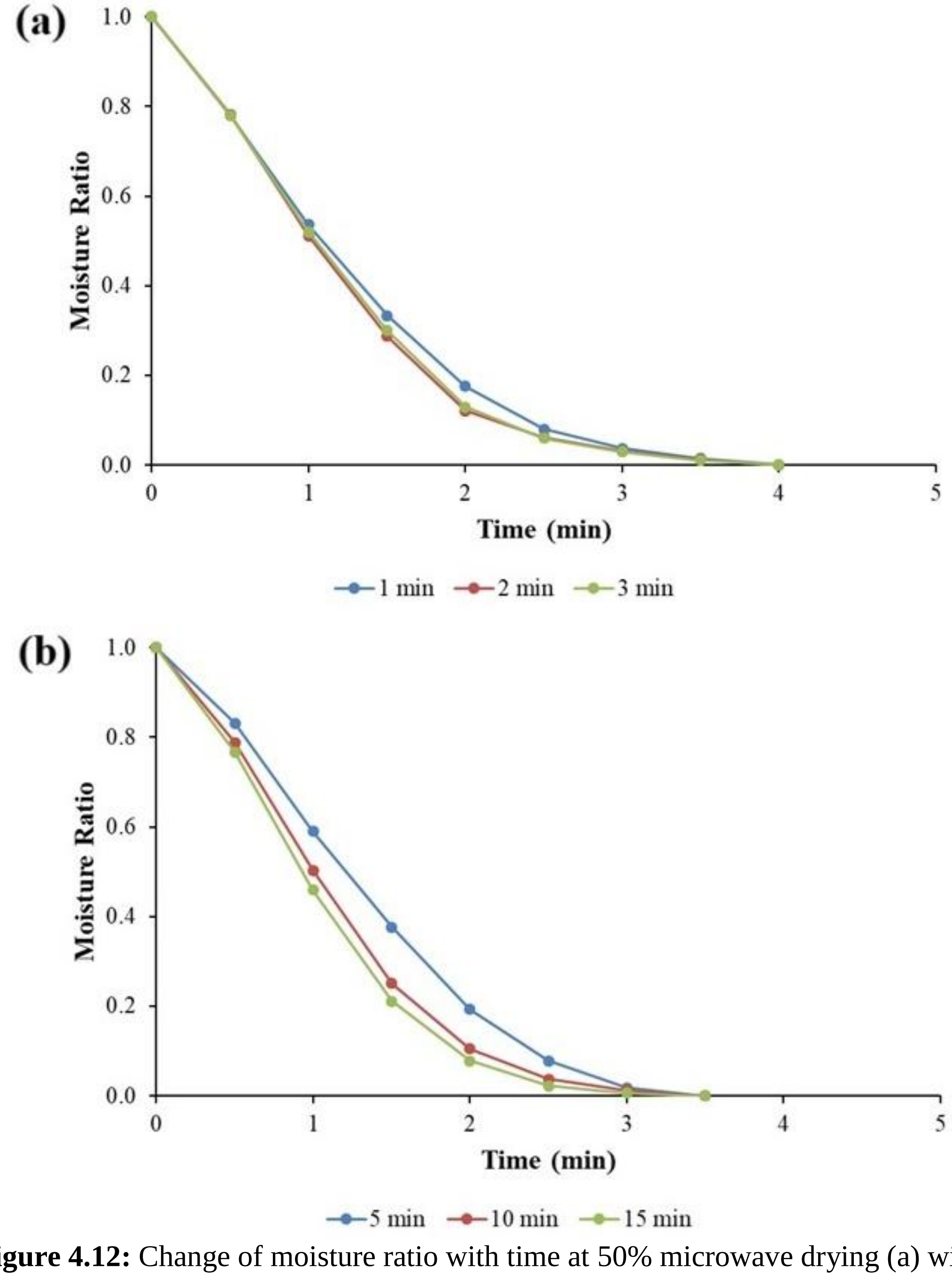

Figure 4.12: Change of moisture ratio with time at 50% microwave drying (a) with blanching pre-treatment (b) with ultrasound pre-treatment.

Drying of blanching pre-treated samples at 70% microwave power displays a non-significant profile among each other (Figure 4.13a). M

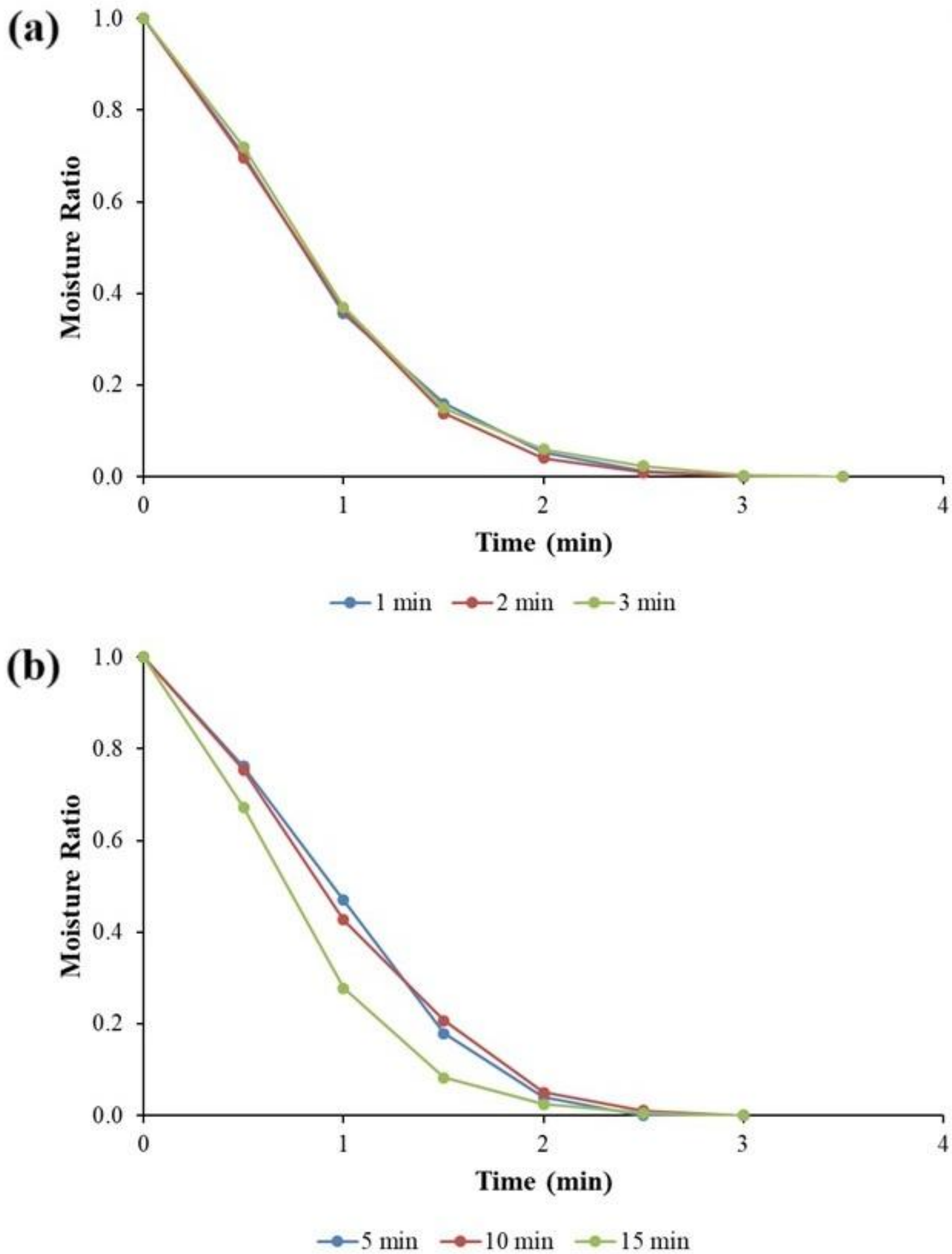

Figure 4.13: Change of moisture ratio with time at 70% microwave drying (a) with blanching pre-treatment (b) with ultrasound pre-treatment.

4.2.2.1 Modelling of drying curves

Semi-emprical drying models namely; Lewis, Henderson-Pabis, Page and Midilli models were used to demonstrate the microwave drying behavior of blanching and ultrasound pre-treated samples. Comparison of model parameters and constants have been given in Table 4.6 for 30% power, Table 4.7 for 50% power and Table 4.8 for 70% microwave power. Coefficient of determination for microwave drying were between 0.9603 and 0.9999, where the least fit was for Henderson-Pabis model and best fit was Page and Midilli models for each microwave power.

Model fittings at 30% microwave power is displayed in Figure 4.14 for 2 minutes of blanching (a) and 5 minutes of ultrasound (b) treatments. Best fit are seen for Page and Midilli models, the model lines aligns with experimental data points and overlap as seen in the figures. R^2 for 2 minutes of blanching treatment were calculated as 0.9993 for both Page and Midilli models, and also show good fit for 5 minutes of ultrasound pre-treatment as seen in Table 4.6. Lewis and Henderson-Pabis models display a lower fit with R^2 of 0.9687 and 0.9603, respectively.

Table 4.6: Comparison of best suited models for thin layer drying of thyme leaves with 30% microwave drying.

Pre-Treatment	**Blanching 2 min**				**Ultrasound 5 min**			
Model	**Lewis**	**Henderson**	**Page**	**Midilli**	**Lewis**	**Henderson**	**Page**	**Midilli**
R^2	0.9876	0.9829	0.9993	0.9993	0.9687	0.9603	0.9989	0.9991
RMSE	0.0556	0.0475	0.0096	0.0089	0.0861	0.0761	0.0130	0.0115
χ^2	0.0035	0.0026	0.0001	0.0001	0.0091	0.0071	0.0002	0.0002
k	0.4257	0.4586	0.2756	0.2647	0.5223	0.5711	0.2983	0.2796
a		1.0867		0.9880		1.1062		0.9820
n			1.4194	1.4460			1.7106	1.7659
b				0.0000				0.0000

Table 4.7: Comparison of best suited models for thin layer drying of thyme leaves with 50% microwave drying.

Pre-Treatment	**Blanching 2 min**				**Ultrasound 15 min**			
Model	**Lewis**	**Henderson**	**Page**	**Midilli**	**Lewis**	**Henderson**	**Page**	**Midilli**
R^2	0.9803	0.9769	0.9995	0.9995	0.9747	0.9710	0.9999	0.9999
RMSE	0.0626	0.0575	0.0077	0.0076	0.0716	0.0668	0.0046	0.0045
χ^2	0.0050	0.0043	0.0001	0.0001	0.0068	0.0059	0.0000	0.0000
k	0.8293	0.8743	0.6830	0.6797	0.9331	0.9822	0.7968	0.7931
a		1.0653		0.9971		1.0658		0.9972
n			1.5387	1.5472			1.6464	1.6531
b				0.0002				0.0000

Table 4.8: Comparison of best suited models for thin layer drying of thyme leaves with 70% microwave drying.

Pre-Treatment	**Blanching 1 min**				**Ultrasound 15 min**			
Model	**Lewis**	**Henderson**	**Page**	**Midilli**	**Lewis**	**Henderson**	**Page**	**Midilli**
R^2	0.9832	0.9812	0.9999	0.9999	0.9768	0.9749	0.9999	0.9999
RMSE	0.0560	0.0530	0.0040	0.0040	0.0647	0.0621	0.0031	0.0027
χ^2	0.0042	0.0037	0.0000	0.0000	0.0059	0.0054	0.0000	0.0000
k	1.0806	1.1212	1.0164	1.0169	1.2419	1.2840	1.2709	1.2779
a		1.0476		1.0004		1.0448		1.0004
n			1.5180	1.5173			1.6677	1.6745
b				0.0000				0.0010

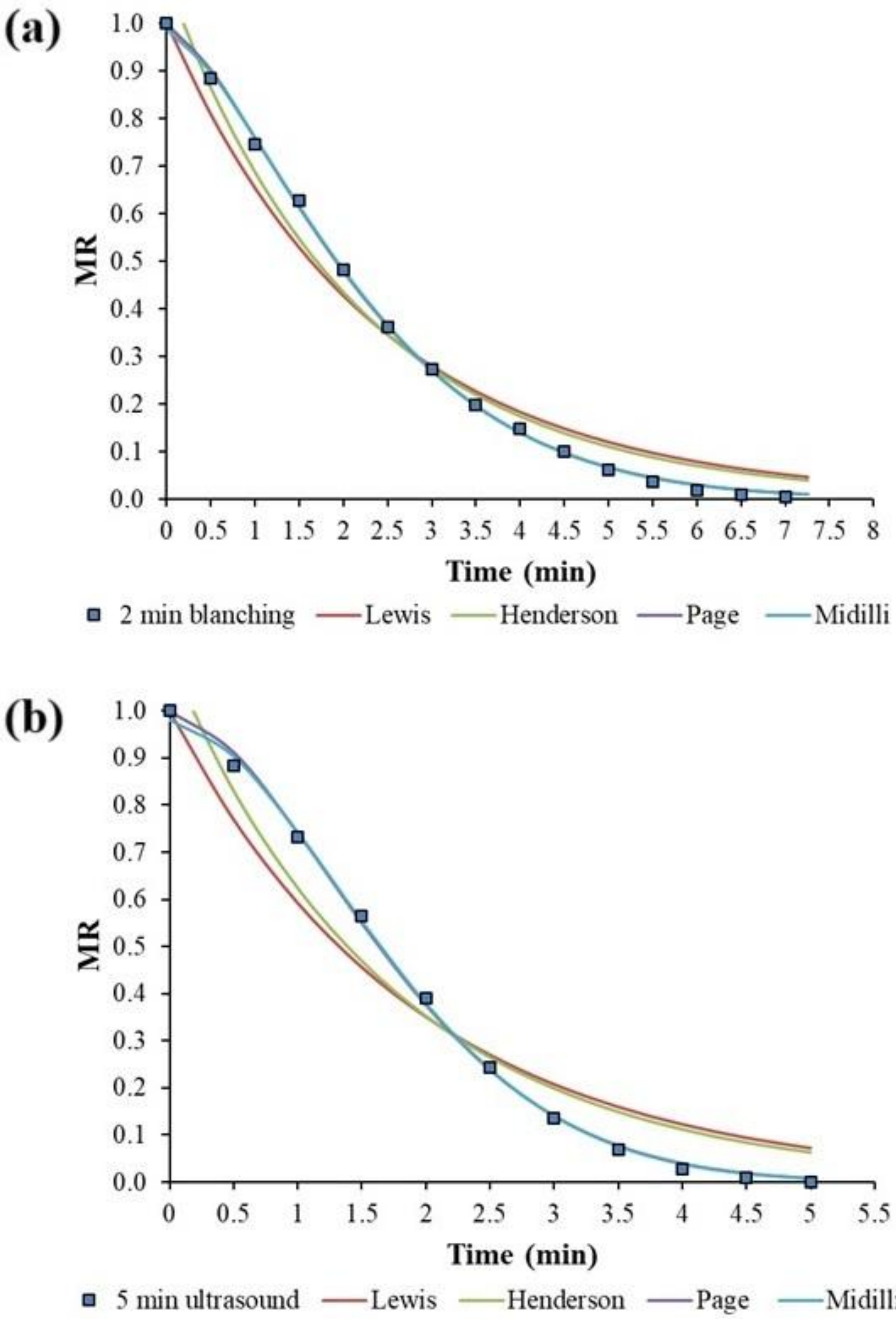

Figure 4.14: Model equation fittings for 30% microwave drying data (a) 2 min blanching pre-treatment (b) 5 min ultrasound pre-treatment.

For 50% microwave power, 2 minutes of blanching and 15 minutes of ultrasound treatments were selected to implement model fitting. As seen on Figure 4.15, overlapping Page and Midilli models had the best fit. Those models both had the R^2 of 0.9995 for blanching and 0.9999 for ultrasound pre-treatments.

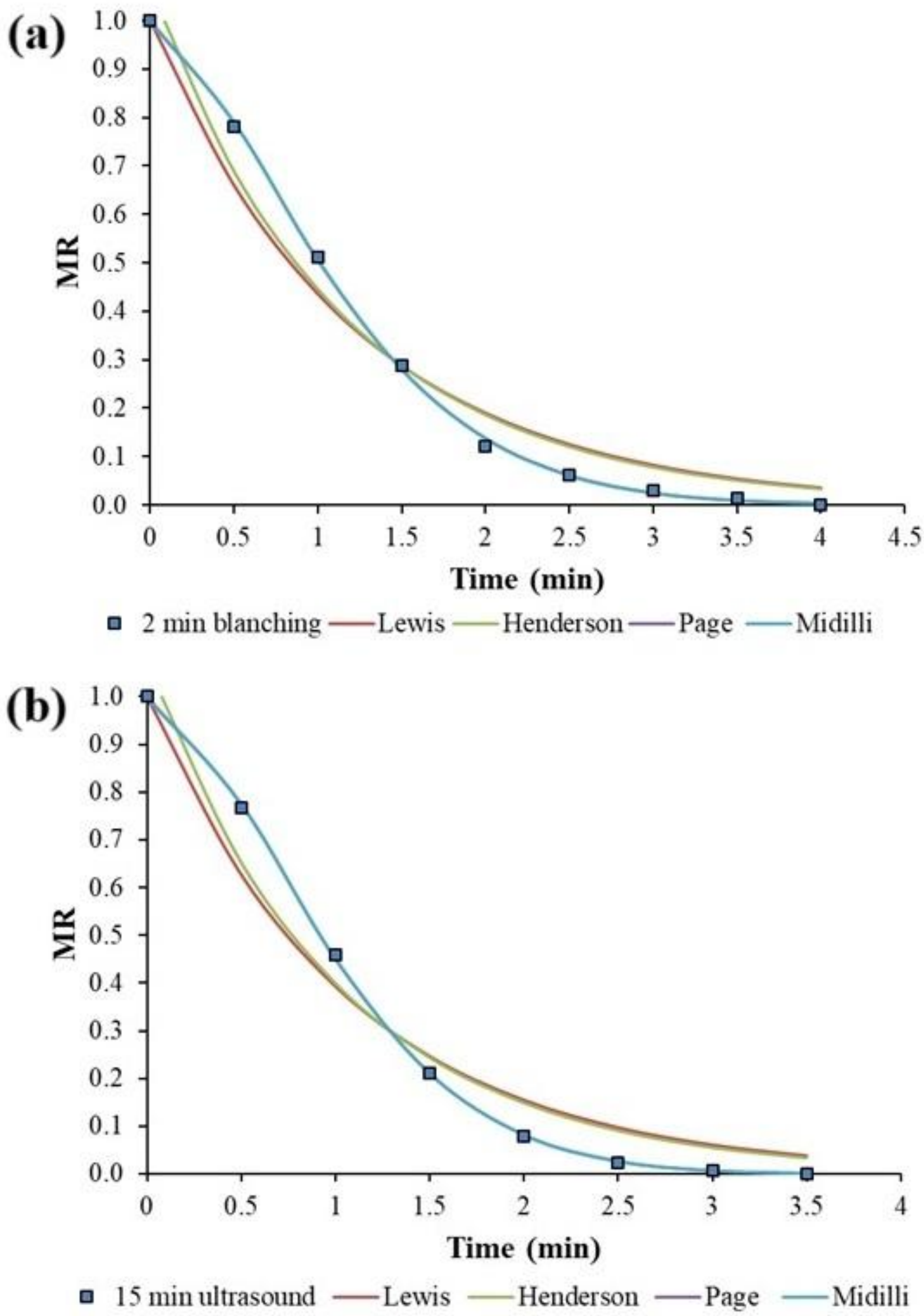

Figure 4.15: Model equation fittings for 50% microwave drying data (a) 2 min blanching pre-treatment (b) 15 min ultrasound pre-treatment.

Page and Midilli models had the most ideal fit for the 1 minute blanching and 15 minutes ultrasound pre-treatments at 70% microwave power with a R^2 of 0.9999. as seen on Figure 4.16, Page and Midilli model lines overlaps on exact experimental data points. On the other hand Lewis and Henderson-Pabis models showed a slightly lower fit, where the R^2 values are 0.9768 and 0.9749 respectively, for 15 minutes of ultrasound pre-treatment at 70% microwave power.

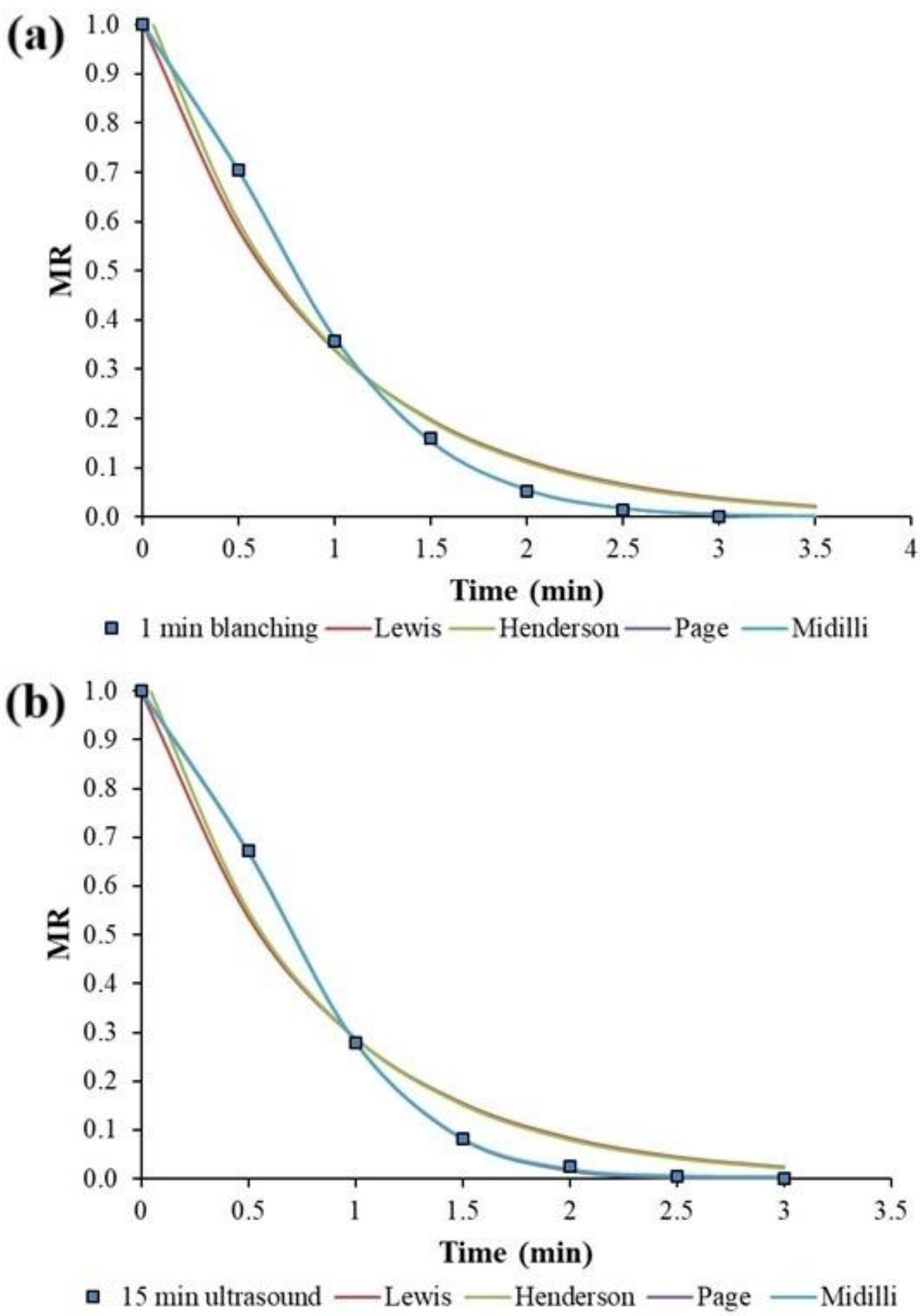

Figure 4.16: Model equation fittings for 70% microwave drying data (a) 1 min blanching pre-treatment (b) 15 min ultrasound pre-treatment.

4.3 Influence of Pre-treatments and Drying Conditions on the Color of Thyme Leaves

Consumer acceptance of food products is considerably affected by the color of that food and products like vegtables and dried herbs have distinctive color attributes. In this study, Hunter color values L*, a* and b* of thyme leaves before and after pre-treatments have been measured and examined. Color values of blanching and ultrasound treated samples subjected to air drying is given in Table 4.9, and microwave drying in Table 4.10. Color values L*, a* and b* of fresh leaves were measured as 41.9, -11.5, and 14.5. Based on CIE color standards L* values represent dakness to lightness where L*=0 is black and L*=100 is white. Considering the measurements of pre-treated dried samples, blanched thyme leaves had substantially lower L* values, which indicates that these samples have darker color. Both microwave and air dried samples after blanching treatment display the same behaviour. On the other hand L* values tend to increase after ultasound treatment. As seen on Table 4.9, ultrasound treatments influence the darkness/lightness of the air dried product, where L* values measured between 40.9 to 49.6.

Table 4.9: Effect of pre-treatments on color values of air dried thyme leaves.

Drying Condition	Pre-treatment	Condition (min)	L*	a*	b*	E*	C*
		5	49.6	-9.9	23.1	55.61	25.1
60 °C		10	48.2	-10.1	25.2	55.28	27.2
		15	47.0	-8.9	21.7	52.53	23.4
		5	44.8	-7.5	19.5	49.41	20.9
70 °C	Ultrasound	10	41.9	-8.3	23.1	48.58	24.5
		15	44.8	-9.1	20.2	50.01	22.1
		5	41.8	-5.3	15.8	44.99	16.7
80 °C		10	40.9	-8.1	18.9	45.81	20.6
		15	45.7	-6.1	16.0	48.81	17.2
		1	28.4	-7.0	12.5	31.85	14.4
60 °C		2	27.1	-6.4	10.6	29.74	12.4
		3	26.3	-6.8	11.9	29.71	13.8
		1	30.7	-7.6	11.9	33.80	14.1
70 °C	Blanching	2	30.1	-7.1	13.2	33.60	15.0
		3	28.6	-7.5	14.5	32.89	16.3
		1	25.8	-5.6	10.5	28.39	11.9
80 °C		2	26.0	-6.9	11.8	29.42	13.7
		3	31.9	-4.3	7.2	33.02	8.4

Table 4.10: Effect of pre-treatments on color values of microwave dried thyme leaves.

Drying Condition	Pre-treatment	Condition (min)	L*	a*	b*	E*	C*
30% (540 w)	**Ultrasound**	5	51.8	-13.7	19.7	57.06	24.0
		10	39.8	-10.7	19.6	45.58	22.3
		15	37.1	-9.4	17.0	41.85	19.4
50% (720 w)		5	42.7	-12.1	19.1	48.28	22.6
		10	41.9	-11.6	18.5	47.29	21.8
		15	36.2	-9.7	16.7	41.04	19.3
70% (900 w)		5	46.9	-13.7	20.7	53.02	24.8
		10	43.0	-9.9	15.0	46.57	18.0
		15	40.7	-10.7	16.9	45.33	20.0
30% (540 w)	**Blanching**	1	32.6	-7.6	15.0	36.71	16.8
		2	28.2	-7.2	12.7	31.72	14.6
		3	32.1	-5.3	10.6	34.23	11.8
50% (720 w)		1	41.9	-9.8	15.6	45.82	18.5
		2	40.4	-10.6	16.8	45.01	19.8
		3	35.2	-8.8	14.6	39.15	17.1
70% (900 w)		1	36.7	-8.8	14.8	40.57	17.3
		2	37.6	-8.3	14.1	41.01	16.3
		3	28.3	-6.7	10.3	30.88	12.3

Visual images of samples were given in Figure 4.17 where dried samples after blanching (A) and ultrasound (B) pre-treatments are shown. It can be clearly seen that blanching treatment causes thyme leaves to attain darker color. In Figure 4.18, visual comparison of pre-treated samples at different levels to fresh and non-treated dried samples were given. Noticably, blanched samples attain a darker color while ultrasound treated samples have a lighter color than fresh samples.

Figure 4.17: Visual comparison of (A) blanching and (B) ultrasound pre-treated 60 °C air dried samples.

In regard to CIE color space a* values represent greenness to redness of the measured sample, where negative (-) a* values are green and positive (+) a* values are red. Therefore increased –a* values on an absoulte unit basis mean the greenness increase. While greenness of fresh samples have a high value, -12.9, pre-treated samples have lower values. Blanching treated samples have lower a* values, ranging between -3.71 to -10.5, while ultrasound treated samples have relatively higher values, -5.22 to -15.1. Greenness of plants is attributed to chlorophyll content, therefore lower a* values may be the result of degredation of plant chlorophyll. It can be said that a high temperature treatment such as blanching diminishes the heat sensitive pigment molecules like chlorphylls. This response can also be seen when microwave and air drying are compared with respect to a* values, where air dried samples have considerably lower values than microwave dried samples.

Figure 4.18: Visual comparison of fresh, blanching and ultrasound pre-treated, and non-treated 60 °C air dried samples.

5. CONCLUSIONS AND RECOMMENDATIONS

The aim of this study was to reduce the adverse effects of conventional drying process on the chemical and physical properties of fruits and vegetables and increase the nutritional quality of dried products. During the studies, it was aimed to demonstrate the drying kinetics of pre-treated products and to determine the thin layer characteristics of the drying operations. The drying process was carried out with two different methods, namely air drying (60, 70 and 80°C) and microwave drying (30% - 390 W, 50% - 650 W, and 70% - 910 W). First of all, for the comparison, drying without pre-treatment was carried out with both methods and the kinetic data were modeled by investigating the compatibility of the moisture change ratios with the Lewis, Page, Henderson and Pabis, Midilli and Logarithmic models. In addition, effective diffusivity and drying activation energy were calculated using the moisture change ratio data. Finally, CIE values were measured using a color measurement device in order to interpret the effect of the drying processes on the color of thyme leaves.

In this study, blanching and ultrasonication pre-treatments were applied to improve drying efficiency and increase the quality of the dried product before air and microwave drying. Samples were subjected to blanching pre-treatment for 1, 2 and 3 minutes and ultrasonication pre-treatment for 5, 10 and 15 minutes. The compatibility of moisture change ratios with Lewis, Page, Henderson and Pabis, Midilli and Logarithmic models was investigated and kinetic data were modeled. In addition, effective diffusivity and drying activation energy were calculated using moisture change ratio data. Finally, CIE values were measured using a color measurement device in order to interpret the effect of drying processes on the color of thyme leaves.

Consequently, the effects of two different pre-treatment applications on drying time for both drying methods were compared and interpreted. In addition, the color measurement results of un-treated fresh, dried without pre-treatment and dried after pre-treatment thyme leaf samples were compared and the effects of different drying methods and different pre-treatment applications on the quality of the product were interpreted.

According to the obtained data, pre-treatment applications before both drying types had an effect in a way that shortened the drying time. In this context, it was concluded that pre-treatment application could contribute to energy saving. For 60°C air drying, 2-minute blanching pretreatment was evaluated as a better option compared to others. Although ultrasonication application at this temperature shortened the drying time, no difference was observed between different application levels. 2-minute blanching application before air drying performed at 70°C emerged as the best option. When the results of samples to which ultrasonication was applied before air drying performed at the same temperature were examined, it was observed that 15-minute application gave better results than the others. Blanching application before air drying at 80°C gave better results than ultrasonication application, but since no difference was observed between application levels, it was concluded that the shortest pre-treatment applications could be preferred in terms of time saving.

When microwave drying results considered, it was observed that the drying time was 7 minutes when the blanching process was applied before drying at 30% power level, while it was observed that the drying time was 5 minutes when the ultrasonication process was applied. At 50% and 70% power levels, 15 minutes of ultrasonication application before microwave drying gave significantly better results than other applications.

Color values, L*, a* and b* measured after drying operations showed that blanching pre-treatment caused the darkness of samples to increase and greenness to decrease and this may be attributed to the loss of heat sesitive chlophyll content due to the high temperature applications.

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CURRICULUM VITAE

Name Surname : Osman Yağız TURAN

EDUCATION

- **B.Sc.** : 2011, Istanbul Technical University, Faculty of Chemical and Metallurgical Enginnering, Food Engineering Department
- **M.Sc.** : 2015, Istanbul Technical University, Graduate School, Food Engineering Department

PROFESSIONAL EXPERIENCE:

2013-2023, Research Assistant at Istanbul Technical University, Chemical and Metallurgical Enginnering, Food Engineering Department

PUBLICATIONS ON THE THESIS:

- **Turan, O.Y., Fıratlıgil, E.,** 2019. Modelling and characteristics of thin layer convective air-drying of thyme (*Thymus vulgaris*) leaves. *Czech Journal of Food Sci*ences, 37: 128-134. DOI: https://doi.org/10.17221/243/2017-CJFS

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