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Investigation of Black Mulberry Drying Kinetics Applying Different Pretreatments

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ABSTRACT

Common drying systems, including hot air convection, infrared, vacuum and IR-convective were employed to investigate and analyze the drying process of mulberry fruits. To evaluate the effect of pretreatment on the drying phenomenon, samples were pretreated by being subjected to: microwaves, chemical preparation, mechanical as well as blanching approaches. Results revealed that the microwave pretreatment, integrated with IR-convective dryer required the lowest time needed for drying mulberries. Furthermore, the experimental data were fitted to semi empirical as well as theoretical models to achieve the most suitable function governing mulberry drying process. Eventually, Page Model proved to perform best with regard to its high coefficient of determination, low value of χ^2 and root mean square of error.

Keywords: Black mulberry, Dryer, Drying, Pretreatment.

INTRODUCTION

Three most known kinds of mulberry are: white mulberry (*Morus alba L.*), black (*Morus nigra L.*), and red (*Morus rubra L.*). The fruit contains a high level of moisture at harvest. Because of the short harvesting season and the sensitivity to storage, drying is often employed as a preservation method. In addition, mulberry is used in mulberry pekmez, juices, paste, marmalade and as well in wine production (Doymaz, 2004).

Drying is rated as an important post-harvest process for foods and fruits despite of high consumption of energy accompanied by quality concerns. The final and major purpose is to minimize moisture to reduce microbial and chemical reactions. Sun drying is still used for drying fruits and vegetables in spite of considerable such drawbacks as long drying time, pollutions, product deterioration and other unwanted damages. These drawbacks have motivated researchers and industrialists to move towards new technologies in drying (Doymaz, 2005). Since the products dried through hot air convection dryers, as the most common methods, are highly prone to surface burning, shrinkage and undesirable color, the need to use technologies which obviate such problems is highlighted. Infrared (IR) energy, inciting the molecular structure of the tissue, raises the temperature of the material and thermal gradient within a short period of time (Umesh Hebbar *et al.,* 2004). All this happens with no heating up of the sample surroundings, resulting in reduced energy consumption as compared with hot air convection systems (Motevali *et al.,* 2011b). However, IR can be integrated with other types of dryers to result in higher efficiency (Ratti and Mujumdar,

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1995) and better quality of the fruit (Navari *et al.,* 1992). It is observed that this integration leads to lower time and energy need as compared with when either IR or hot air convection is individually employed (Umesh Hebber *et al.,* 2004). Vacuum drying, as another method, is a unit operation in chemical and engineering processes, in which moist material is dried under sub-atmospheric pressures (Fernando and Thangavel, 1987). Vacuum drying is proved to have preserved the fruit quality and sensory attributes (Jaya and Das, 2003). In spite of the advantages of the mentioned systems, a pretreatment operation in advance of drying can facilitate a faster moisture removal from the samples. There have been several pretreatment methodologies introduced from among which few of them are more common. Microwave heating as a pretreatment, due to its selective heating characteristics, is reported to reduce the energy and time needed to be consumed in drying (Motevali *et al.,* 2010b). In addition, chemical pretreatment and blanching present similar effects (Doymaz, 2004). Ultrasound energy has also proved to be advantageous in improving the drying phenomenon through frequent and fast contractions *vs.* expansions which in turn cause the tissue to be made spongy (Fernandes, 2007). The pressure gradient influenced by using ultrasound vibrations increases the evaporation rate and as a result of the raised intensities of energy, cavitations appear in water molecules. Consequently, drying would be carried out at a high rate (Mulet *et al.,* 2003) and mass transfer would be enhanced (Garcia-Perez *et al.,* 2006a).

Numerous researches have conducted studies on the drying of agricultural products including sweet cherry (Doymaz and Ismail, 2011), sour cherry (Motevali *et al.,* 2011a), pomegranate arils (Motevali et al., 2001b), Quercus (Tahmasebi *et al.,* 2011), Soybean (Rafiee *et al.,* 2009), Apple (Mousavi and Javan, 2009), Cumin (Zomorodian and Moradi, 2010). Yet, few studies are reported on the comparison of pretreatment methods in the drying of fruits and vegetables. The major objective of this paper is to analyse the effect of pretreatment methodologies including microwave, chemical (ethyl oleate), mechanical (ultrasound) and blanching (hot water) on drying kinetics of mulberry fruits, using hot air convection, IR, IR-convective as well as vacuum dryers.

MATERIALS AND METHODS

Preprocessing

Samples of fresh mulberry fruits were layered on the dryer tray and dried up to an approximate moisture content of 10% wb (kg moisture kg^{-1} dry matter) in which the variations in weights of samples at different successive intervals were settled and unchanged. The mean ambient temperature was 27±3°C and the relative humidity of the air measured to be at 25±5% throughout the experiments. Experiments were carried out for five treatments of: control, blanching, microwave, ethyl oleate as well as ultrasonic. Also drying experiments were carried out by means of four dryers including convective, IR, IR-convective as well as vacuum. The parameters and their variable levels designed for the dryers are detailed in Table 1.

Moisture content was found by air drying of the representative samples of fruits at 100°C for 4 to 5 hours (AOAC, 1980). The initial moisture contents of mulberries stood at 78% (wb). Three levels of temperature (40, 50 and 60°C) along with three levels of air velocity (0.3, 0.7 and 1 m s^{-1}) were chosen for the case of hot air convection drying.

In order to facilitate the vacuum drying, a vacuum dryer (VS-1202 v5, Korea) equipped with vacuum pump (Platinum, 3055jxhrl-4205, USA) was employed. Air parameters of temperature and velocity were measured and adjusted using thermometers (Lutron, TM-925, Taiwan) and an anemometer (Anemometer, Lutron-YK, 80AM, Taiwan). Microwave (SAMSUNG, 75DK300036V, Model: M945, Korea) was utilized for preheating the samples at 200W

Table 1. Operational parameters of dryers.

Dryer	Parameters	Levels		
Hot air convection	Temperature (°C)	40	50	60
	Air velocity (m s^{-1})	0.3	0.7	1
Infrared	Radiation (W cm^{-2})	0.22	0.31	0.49
	Air velocity (m s^{-1})	0.3	0.7	1
IR-convective	Radiation (W cm^{-2})	0.22	0.31	0.49
	Temperature (°C)	40	50	60
	Air velocity (m s^{-1})	0.3	0.7	1
Vacuum	Temperature (°C)	40	50	60
		70	80	90

power radiation for 10 minutes. Additionally, ultrasound pretreatment was carried out using a 240W ultrasound source (Hielscher ultrasonic GmbH, UP400S, POWER 400W, Frequency 24 kHz, USA) for 10 minutes. Chemical preprocessing of samples was achieved by immersing the fruits in the solution of 2% ethyl oleate and 5% potassium carbonate (K_2CO_3) for 1 minute (Doymaz, 2004). Samples were immersed in hot water at 80°C for 10 seconds proceeding with water soaking at the ambient temperature to yield blanching.

Modeling the Drying Process

Kinetic models are commonly stated based on the Moisture Ratio (MR) normally characterized by centering attribute and better illustration (4). *MR* is defined as Equation (1):

$$MR = \frac{M_t - M_e}{M_o - M_e} \quad (1)$$

Where, M_t is the moisture content (kg $_{water}$ kg$^{-1}_{dry\ matter}$) and M_e and M_o are equilibrium and initial moisture contents, respectively. Considering a negligible value of M_e with respect to M_o and M_t, the error of eliminating M_e from Equation (1) would be ignorable permissible. Thus, Equation (1) can be rewritten as Equation (2):

$$MR = \frac{M_t}{M_0} \quad (2)$$

MATLAB 2007, curve fitting toolbox environment was employed to run standard drying curve fitting (Table 2) to the experimental data. Statistical criteria of R^2, χ^2 and *RMSE* (Equations (3), (4) and (5)) were considered for the selection of the optimized model.

$$R^2 = 1 - \left[\frac{\sum_{i=1}^{N} (MR_{pre,i} - MR_{\exp,i})^2}{\sum_{i=1}^{N} (\overline{MR}_{pre} - MR_{\exp,i})^2} \right] \quad (3)$$

$$\chi^2 = \frac{\sum_{i=1}^{N} (MR_{\exp,i} - MR_{pre,i})^2}{N - m} \quad (4)$$

$$RMSE = \left[\frac{1}{N} \sum_{i=1}^{N} (MR_{pre,i} - MR_{\exp,i})^2 \right]^{\frac{1}{2}} \quad (5)$$

Where, $MR_{exp,i}$ stands for *i*th experimental *MR*, $MR_{pre,i}$ is the predicted *i*th *MR*, *N* standing for the number of observations and *m* for the number of constants (Akpinar *et al.,* 2003; Babalis *et al.,* 2005).

Results and discussion

Hot Air Convection

Figure 1 illustrate the drying time at the air velocities of 0.3, 0.7 and 1 m s^{-1}.

From the Figures, it can be deduced that the thermal gradient resulting from temperature increase at the constant velocities might be

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Table 2. Standard models reported in the literature used for drying of agricultural products.

No.	Model	Mathematical Function	Ref.
1	Wang and Singh	$MR = at^2 + bt + c$	Wang and Singh (1978)
2	Verma *et al.*	$MR = a\exp(-kt) + (1-a)\exp(-gt)$	Verma *et al.* (1985)
3	Henderson and Pabis	$MR = a\exp(-kt)$	Chhinnan (1984)
4	Logaritmic	$MR = a\exp(-kt) + c$	Dandamrongrak *et al.* (2002)
5	Modified Page	$MR = \exp(-(kt)^n)$	Wang *et al.* (2007)
6	Two term	$MR = a\exp(-k_0 t) + b\exp(-k_1 t)$	Diamente and Munro (1991)
7	Approximation of diffusion	$MR = a\exp(-kt) + (1-a)\exp(-kbt)$	Ertekin and Yaldiz (2004)
8	page	$MR = \exp(-kt^n)$	Simal *et al.* (2005)
9	Modified Henderson and Pabis	$MR = a\exp(-kt) + b\exp(-gt) + c\exp(-ht)$	Sharma *et al.* (2005)
10	Newton	$MR = \exp(-kt)$	Ayensu (1997)
11	Midili et al.	$MR = a\exp(-kt^n) + bt$	Menges and Ertekin (2005)
12	Two term exponential	$MR = a\exp(-k\ t) + (1-a)\exp(-k\ at)$	Motevali *et al.* (2010)

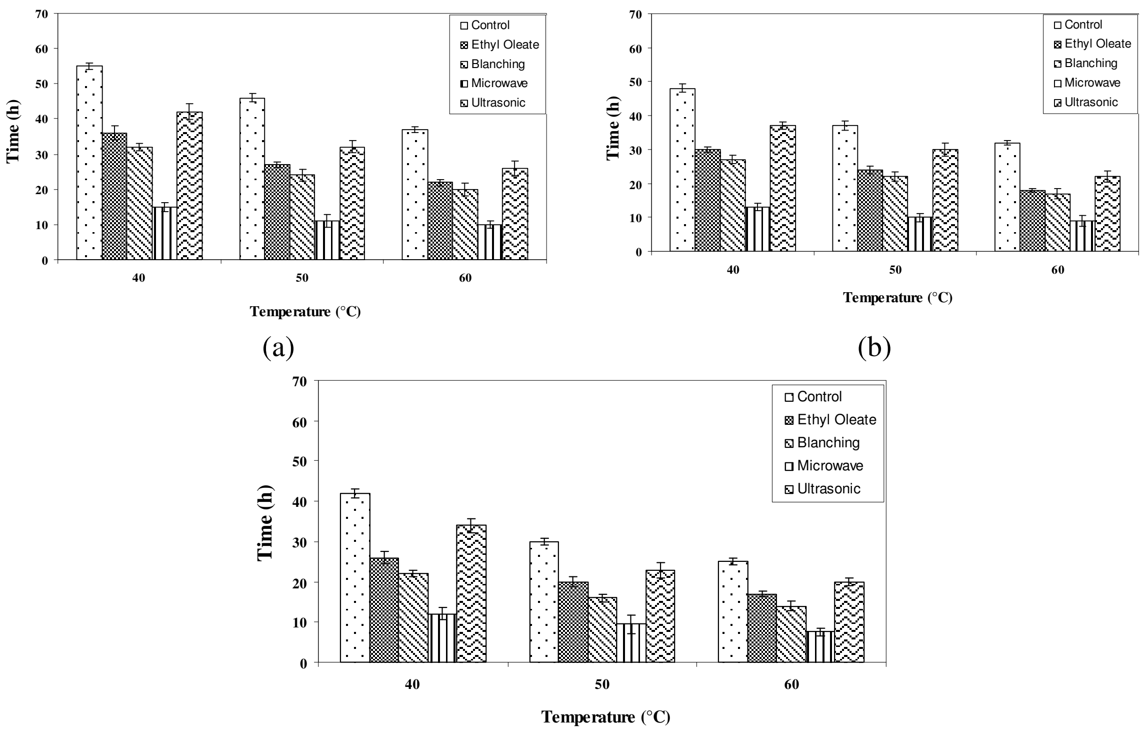


Figure 1. Drying time values for hot air convective drying at the air velocity of (a) 0.3 m s^{-1} (b) 0.7 m s^{-1} and (c) 1 m s^{-1}.

the main reason for diminishing the drying time. Air velocity has a similar effect in the sense that the ambient air pressure decreases with the velocity of air leading to the reduced inhibition for water transfer from the material body. Researches already conducted for pomegranate arils (Motevali *et al.,* 2010, 2011b) and sweet cherries (Doymaz and Ismail, 2011) have confirmed such a result. Also it can be observed that using all types of pretreatment reduced the drying time compared to when no pretreatment was applied. The lowest drying time was recorded for microwave pretreatment. This may be stated by the selective heating of microwave energy along with the volume tempering. Moreover, there happens a slight destruction in the outer layer of the fruit which in turn facilitates the removal of moisture using microwave energy (Motevali *et al.,* 2010, 2011a). The effect of chemical preparation on the drying time is also shown in Figure 1. Removal of waxy layer integrated with fine pores on the surface of the fruit may be originated directly from application of ethyl oleate. The consequence would be higher moisture diffusivity and faster water transfer from the fruit, which is clearly proved by some researchers (Doymaz, 2004; Doymaz and Pala, 2002). Thermal shocks originated from using blanching (hot water) can also significantly reduce the drying time. Numerous cracks were observed after this pretreatment which can be reasonable justification of such effect (Doymaz, 2004, 2010). Ultrasonic effect is described as a reason for pressure gradient in the sample resulting in a spongy shaped material with no sensible change in temperature. This makes the evaporation process faster. Additionally, deformation of microscopic channels and pores in fruit, influenced by ultrasound energy, reduced the diffusion boundary layer leading in turn to the mass transfer increase and hence decreased drying time (Fernandes *et al,* 2008; Fernandes and Rodrigues, 2007; Fuente-Blanco and Sarabia, 2007; Tarleton and Wakeman, 1998).

Infrared Energy

Figure 2 presents the required drying time for mulberries when the predefined pretreatment methods and infrared dryer were applied. The radiation intensity and air velocity are the major parameters to be controlled in IR dryers. Unlike hot air convection dryers, here, the drying time didn't increase with the air velocity which can be justified by the cooling effect of air in IR dryers. Also the effect of radiation

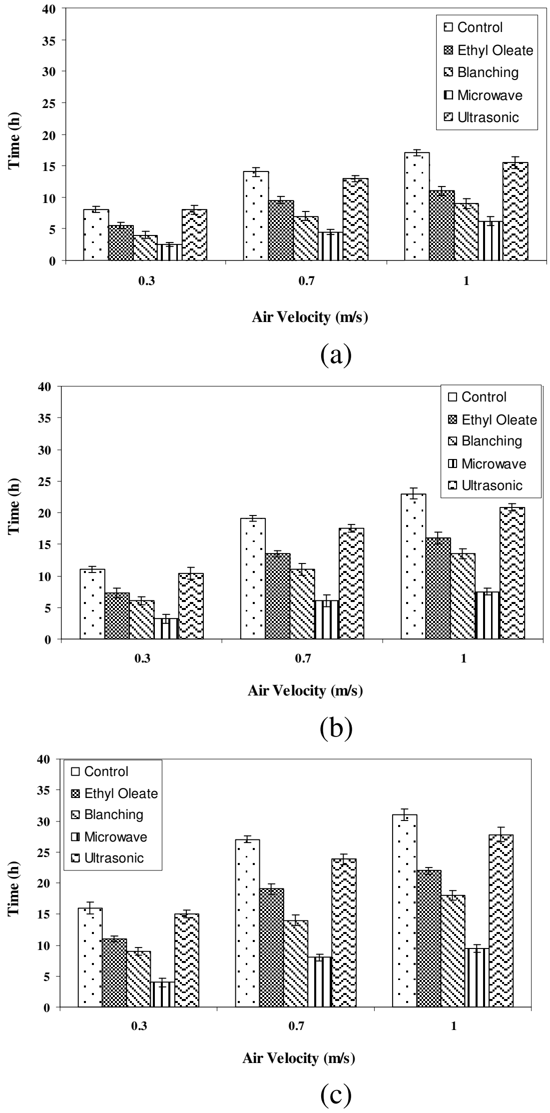


Figure 2. Drying time values for infrared drying at the radiation of (a) 0.49, (b) 0.31, and (c) 0.22 W cm^{-2}.

intensity is illustrated. As predicted, intense radiations can raise heat gradient leading to faster evaporation of water and reduced drying time. IR drying preceded by ultrasound pretreatment showed the maximum drying time among the treatments. Again, microwave pretreatment was the most effective approach to reduce drying time as shown in Figure 2. Statistical analysis of the results showed that the drying time was 2.74 to 4 times lower in microwave; 0 to 1.13 times lower in ultrasound, 1.40 to 1.54 times lower in ethyl oleate, and 1.70 to 2 times lower in blanching pretreatments when compared to the untreated state. This trend is frequently reported by researchers in other agricultural products as grape (Celma *et al.,* 2009), olive husk (Celma *et al.,* 2008), onion (Sharma *et al.,* 2005) and blueberries (Shi *et al.,* 2008). Furthermore, similar claim is reported for grape chemical preprocessing in an IR dryer (Caglar *et al.,* 2009). Comparison of drying time for mulberries in IR and convection dryers showed that the time needed for drying a given amount of the sample is longer in convection dryers due to low thermal conductivity of the fruit. Moreover, since the heating phenomenon begins from the outer layer of the fruit, hardening happens causing decrease in diffusivity. The hard layer inhibits the mass transfer through the body and hence increases drying time. In IR drying, however, the absorption of infrared wavelengths by water molecules provides a roughly selective heating and prevents surface hardening. This is confirmed for the case of potato drying (Afzal and Abe, 1999).

Integrated Combination of IR and Convection Dryers

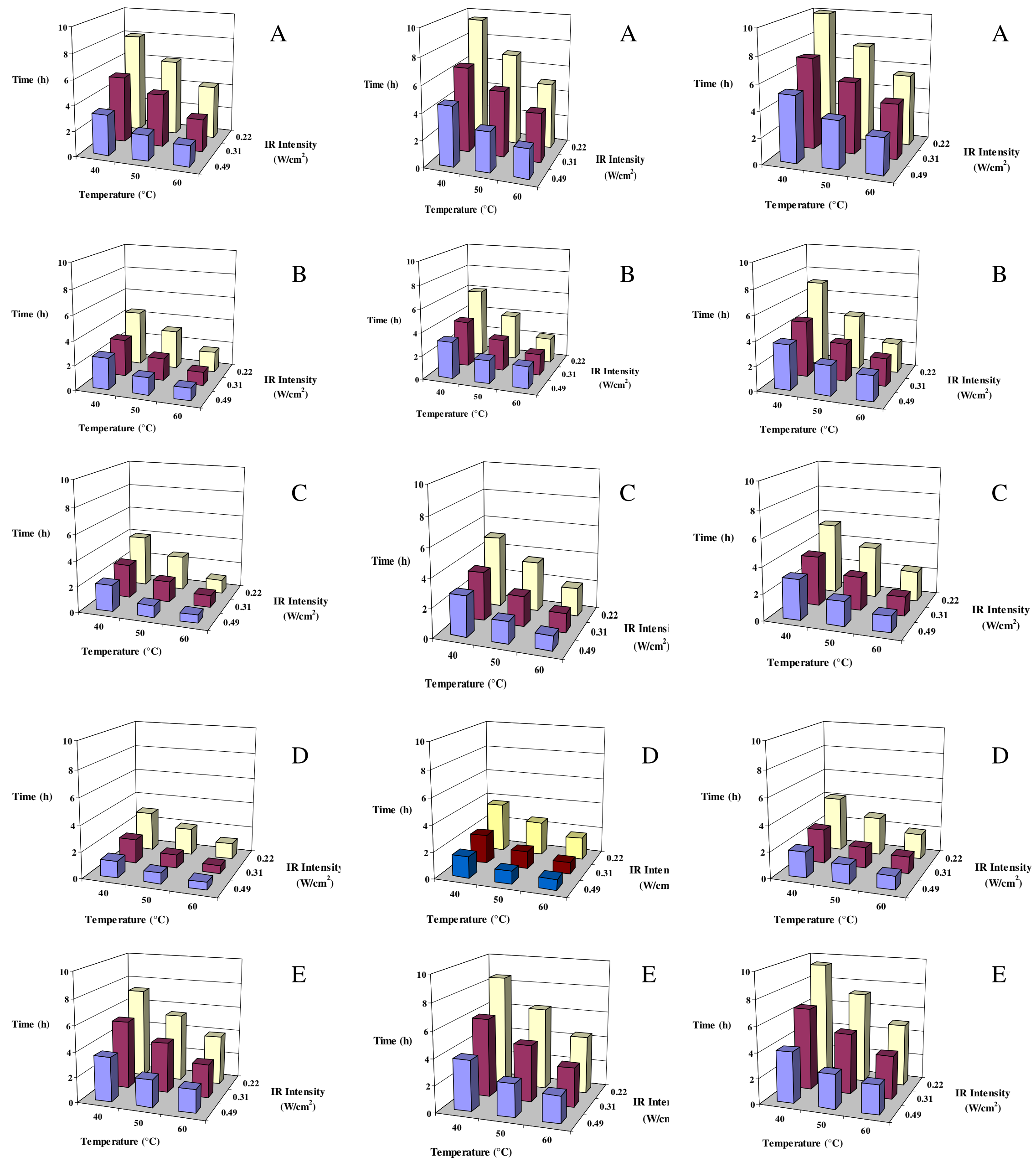
A simultaneous heat transfer by infrared energy and hot air convection takes place using an IR-convective dryer. Figure 3 illustrate that the drying time is reduced with radiation intensity and temperature. Also increasing air velocity increases the drying time. This may be due to the cooling of the product surface through air flow, so that with increasing air flow rate, the product surface becomes cooler and the thermal gradient inside the product is decreased, resulting in increased drying time of the product. Considerable thermal gradient caused by the mentioned parameters can be the major reason for this effect. The integration of two thermal sources was proved to be much more efficient comparing the application of either individually. (Jaturonglumlert and Kiatsiriroat, 2010; Pathare *et al.,* 2006; Pawar *et al.,* 2008; Afzal *et al.,* 1999; Umesh Hebbar *et al.,* 2004). In addition, statistical evaluation of data revealed a 256 to 445% decrease of drying time using microwave pretreatment for 0.7 m s^{-1}. This decrease was obtained to be 150 to 227% in ethyl oleate, 160 to 285% in blanching and 107 to 126% in ultrasound pretreatment for 0.7 m s^{-1}.

Vacuum Dryer

Figure 4 presents the effect of temperature and pretreatment on the drying of mulberry in a vacuum dryer. Air temperature and indoor pressure are the major parameters dominating the drying process in vacuum dryers. As seen in the figure, the time required for drying fruits decreased with temperature which is predictable based on the experience on other dryers. The main reason lies on the fact that the thermal gradient increases directly with air temperature resulting in an improved drying. Drying of pumpkin (Arevalo-Pinedo and Fernanda, 2007); mango (Jaya and Das, 2003) and white radish (Lee and Kim 2009) is assessed using vacuum dryers. The effect of applying microwave pretreatment was found to be more significant (244 to 500% reduction in drying time).

Modeling and Curve Fitting

Primary moisture content of fruits was



(*a*) (*b*) (*c*)

Figure 3. Drying time values for hot air-infrared drying at the air velocity of (*a*) 0.3 m s^{-1} (*b*) 0.7 m s^{-1} and (*c*) 1m s^{-1} in different temperatures and IR intensities: (A) Control; (B) Ethyl Oleate; (C) Blanching; (D) Microwave, and (E) Ultrasonic.

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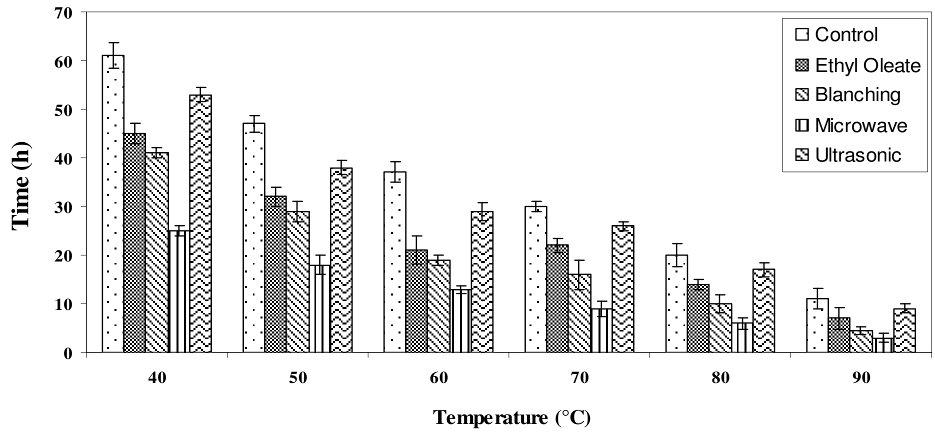

Figure 4. Drying time values for vacuum dryer.

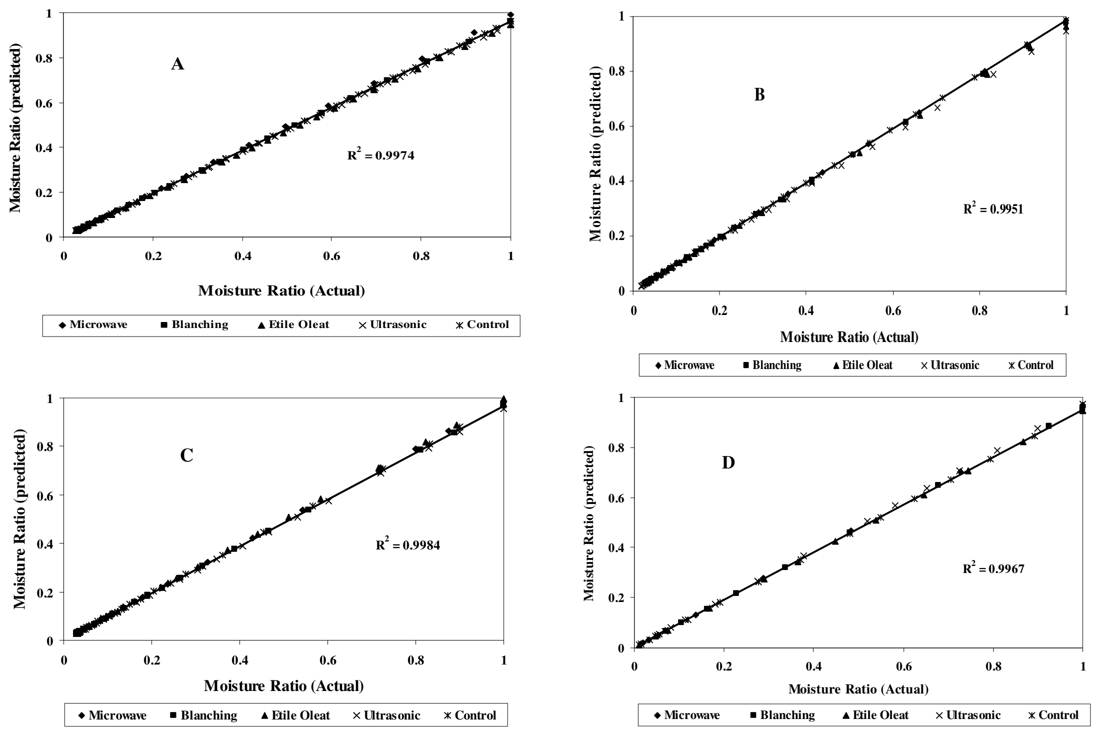

Figure 5. Comparison of experimental moisture ratio with predicted moisture ratio from the Page Model in: (A) Convection; (B) IR; (C) Convection-IR, and (D) Vacuum dryer.

measured to be 78 % wb. Moisture was monitored at time intervals and *MR* was calculated by Equation (2). Real moisture ratios were fitted to the introduced models in Table 2 and accuracy of fitting was evaluated by R^2, χ^2 and *RMSE* of the correlation. Results showed that Page model provided the best accuracy in fitting to the experimental data of mulberry drying. The results of other models are fully detailed in Table 3.

Figure 5 demonstrates the comparison between measured *MR* and predicted *MR* with page model. Validation of the determined model was established by comparing the experimental data, for drying curve (in different drying method), with the values predicted by the Page model and the

Table 3. Optimum statistical criteria obtained from fitting experimental data to the predefined models.

Statistical Analyses	Model Number 1	2	3	4	5	6	7	8	9	10	11	12
Hot air drying (60°C, 0.7 m s^{-1}, Microwave pretreatment)												
R^2	0.9859	0.9851	0.9871	0.9817	0.9941	0.9863	0.9889	0.9994	0.9909	0.9718	0.9545	0.9172
χ^2	0.0016	0.0018	0.0016	0.0021	0.0019	0.0016	0.0013	0.0004	0.0012	0.0024	0.0021	0.0017
RMSE	0.0410	0.0426	0.0398	0.0457	0.0435	0.0401	0.0357	0.0314	0.0319	0.0534	0.0508	0.0469
IR (0.49 W cm^{-2}, 0.3 m s^{-1}, Ethyl oleate pretreatment)												
R^2	0.9182	0.9631	0.9819	0.9906	0.9944	0.9923	0.9872	0.9989	0.991	0.9878	0.9918	0.9906
χ^2	0.0089	0.0043	0.0015	0.0011	0.0012	0.00097	0.0015	0.0008	0.0011	0.0015	0.0009	0.0011
RMSE	0.0943	0.0657	0.0462	0.0326	0.0323	0.0312	0.0383	0.0334	0.0325	0.0388	0.0301	0.0332
IR-Hot air (0.49 W cm^{-2}, 60°C, Microwave pretreatment)												
R^2	0.9839	0.9884	0.9986	0.9906	0.9930	0.9923	0.9872	0.9995	0.991	0.9878	0.9918	0.9906
χ^2	0.0017	0.0011	0.00015	0.0011	0.0011	0.00097	0.0015	0.0002	0.0011	0.0016	0.001	0.0011
RMSE	0.0415	0.0338	0.0124	0.0333	0.0329	0.0312	0.0393	0.0135	0.0338	0.0404	0.0319	0.0332
Vacuum (70°C, Blanching pretreatment)												
R^2	0.9906	0.9914	0.9923	0.9859	0.9985	0.987	0.9817	0.9991	0.9992	0.9839	0.9884	0.9986
χ^2	0.0011	0.0011	0.00097	0.0015	0.0017	0.0015	0.0019	0.0008	0.0009	0.0017	0.0011	0.0001
RMSE	0.0333	0.0329	0.0312	0.0392	0.0418	0.0392	0.0446	0.0427	0.0097	0.0415	0.0338	0.0124

results are plotted in Figure 5. The data points are banded around a 45° straight line, demonstrating the suitability of the model in describing the thin-layer drying behavior of the mulberry.

CONCLUSIONS

Drying process of mulberry fruits was comprehensively investigated in this research. Several pretreatments and common types of dryers were employed to achieve a precise idea of mulberry drying. It was found that using pretreatment, microwave in special, is an efficient way to reduce the drying time. The integration of infrared energy and convective drying proved to yield the most suitable condition of drying with regard to the required drying time. Furthermore, Page Model fitted best to the experimental data as compared with the other standard models reported in drying of agricultural products.

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بررسی خشک کردن توت سیاه با استفاده از پیش تیمارهای مختلف

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چکیده

در این تحقیق از پیش تیمارهای مختلف برای بررسی فرآیند خشک کردن توت سیاه با بکارگیری از روشهای مختلف خشک-کردن از جمله جریان هوای داغ، خلا و فروسرخ و جریان هوای داغ-فروسرخ بررسی شد. آزمایشات با استفاده از پیش تیمارهای مایکروویو، شیمیایی (اتیل اولئات و کربنات پتاسیم)،

مکانیکی)آلتراسونیک) و بلانچینگ (آب گرم (و شرایط بدون پیش تیمار انجام شد .فرآیند خشک شدن توت سیاه برحسب تابعی از متغیرهای مختلف در خشک کن های مختلف بررسی شد. نتایج حاصل از آزمایشات نشان داد که بیشترین و کمترین زمان خشک شدن به ترتیب در شرایط بدون استفاده از پیش تیمار (Control) و با استفاده از پیش تیمار مایکروویوو در خشک کن ترکیبی جریان هوای داغ- فروسرخ بود. همچنین میزان برازش داده های تجربی با انواع مدلهای نیمه نظری و تجربی ارزیابی شد و نهایتاً از میان مدلهای مورد برازش، مدل پیج بدلیل دارا بودن بالاترین R^2 و کمترین x^2 و RMSEبه عنوان مناسب ترین مدل انتخاب شد.

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