Effect of pre-treatments on drying characteristics of Chinese jujube (*Zizyphus jujuba* Miller)

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Abstract: Chinese jujube is delicious and nourishing fruit. However, fresh Chinese jujube is liable to rot and drying is a necessary process. Traditional drying is a time-consuming task due to the thick cuticle of Chinese jujube. To improve its drying efficiency, fresh Chinese jujube was pretreated with nine different methods prior to hot-air drying. Among these methods, dipping in 2% ethyl oleate plus 5% K_2CO_3 for 10 min (alkaline emulsion of ethyl oleate, AEEO) was recommended for its time-saving effect, which was found more significant at lower drying temperatures. The beneficial effect was considered based on its cuticle destruction by AEEO pre-treatment. In the meantime, the drying process was divided into three stages; each of them obeyed the first order reaction kinetics. Activation energies for the first, second and third stages of control over jujube drying were 41.45 kJ/mol, 35.24 kJ/mol and 49.52 kJ/mol, and reduced by 20.9%, 22.1% and 29.0%, respectively, after AEEO pre-treatment, and the drying process was well predicted by Midilli et al. model. In view of browning during drying at higher temperatures, AEEO pretreated jujube was suggested to be dried at 60°C. This finding was considered to be helpful to the industrial drying of Chinese jujube.

Keywords: Zizyphus jujuba, Chinese jujube, pre-treatment, hot-air drying, activation energy, mathematical modelling
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1 Introduction

Zizyphus jujuba Miller is a deciduous tree (Rhamnaceae) and indigenous in China, where it has been cultivated for more than 4 000 years. As the largest jujube producer, China has approximately 98% share of the total production of the world. Its harvested area was about 10 000 hectares and production quantity was 147 600 tons in 2010[1]. Its fruit, Chinese jujube or Chinese date, is consumed for its excellent taste and abundant nutrition, can be eaten fresh, dried and preserved, or added as a base in meals or in the manufacture of candy, tea, juice, vinegar, wine and powder. In traditional Chinese medicine, it is also used to treat anorexia, asthenia, loose stool and climacteric syndrome[2].

Fruit rot after harvesting has always been a serious problem for Chinese jujube, leading to short shelf life, inferior quality, and poor economic benefit. Drying is the best choice to solve this problem for most cultivars except those particularly well suited for fresh eating, such as "Dongzao"[3]. Traditionally, sun drying is applied to jujube drying, which has the advantage of no professional equipment and easy operation. However, sun drying is also easily influenced by weather condition, long drying time and danger of contamination. Zhang et al.[4] found that hot-air drying could improve the quality of the dried jujube fruit. Due to the more significant benefits of

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hot-air drying, such as the controllability of drying process, high productivity, and uniform and hygiene product[5], it has been increasingly applied to the industrial production of Chinese jujube in practice. In recent years, researchers have conducted several studies on the drying characteristics of jujube[6-10]. Microwave drying and vacuum drying were also applied to jujube drying[11-14]. The researches are helpful for our understanding, predicting and guiding jujube drying.

Jujube is covered with a thick layer of cuticle[15], usually serving as barrier against fungal pathogens, avoiding leakage of nutrition and mechanical damage, and maintaining water[16]. However, this cuticle also hinders the moisture loss during drying, leading to low moisture diffusivity and drying rate, and time-consuming drying process[17-19]. To overcome this obstacle, many methods have been developed, such as heat shock and/or chemical pre-treatments in aqueous solutions of sodium hydroxide (NaOH), potassium hydroxide (KOH), potassium carbonate (K_2CO_3) and ethyl oleate (EO) etc.[17-24]. However, no information is available about pre-treatment effect on jujube drying in literature. In this study, therefore, fresh jujube was subjected to several pre-treatments prior to hot-air drying, and the drying characteristics and mathematical modelling of the jujube pretreated with the suggested method were investigated in comparison with the untreated control. The mechanism underlying this pre-treatment was also discussed.

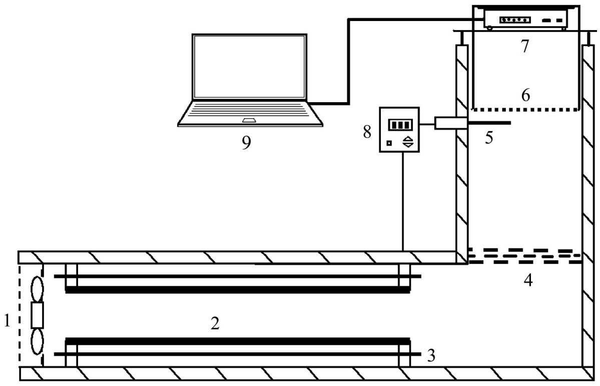
2 Materials and methods

2.1 Experimental material

Fresh jujube (*Ziziphus jujuba* ‘Yuanling’) was collected in the middle of September, 2011 and 2012, from the same jujube orchard located in the southern hilly region of Jinan, Shandong Province, China, and stored in a refrigerator at 4°C prior to the experiments. Jujube of uniform size (average diameter was (2.5 ± 0.2) cm and average weight was (10.0±0.5) g) was used for the experiments. The initial moisture content ((2.93±0.03) g water/g dry matter) was determined by drying the samples at 105°C for 24 h.

2.2 Experimental dryer

The dryer consists of an axial fan, a heating chamber, a drying chamber and a weighing system (Figure 1). The chamber is made of galvanized sheet insulated by silicate material. Air supplied by the fan was heated to the required temperature in the heating chamber by using four far infrared lamps (total output power, 4 kW). The hot air enters into the drying chamber, and then exhausts at its top. Drying temperature was automatically controlled by a proportional-integral-derivative (PID) controller. Air velocity was measured using an anemometer and adjusted by changing the revolving speed of the axial fan by an electron speed regulator. A sample basket (25 cm × 25 cm), made out of stainless steel mesh, was suspended from an electronic balance (0.01 g). Sample weight was automatically recorded by a computer through communication with the balance at an interval of 0.5 h or 1.0 h.



1. Axial fan; 2. Far infrared lamp; 3. Reflecting shade; 4. Perforated floor; 5. Temperature senor; 6. Sample basket; 7. Electronic balance; 8. PID controller; 9. Computer

Figure 1 Schematic diagram of the experimental dryer

2.3 Experimental procedure

The fresh jujube was taken out of refrigerator and acclimated to room temperature for 2 h, and then pretreated by dipping in tap water (25°C, 10 min) as control, cutting into halves, partly peeling, or dipping in hot water (80°C, 1 min), 5% K_2CO_3 or 2% EO (ethyl oleate) (25°C, 10 min), 2% EO plus 5% K_2CO_3 (alkaline emulsion of ethyl oleate, AEEO) or its 8-fold dilution (25°C, 10 min), 1% NaOH or KOH (60°C, 10 min). At the beginning of each experiment, the dryer was adjusted to the indicated air velocity and temperature (1.0 m/s and 60°C). The pretreated jujube was distributed into the sample basket in a thin layer and dried until the weight loss was about 50%. Relative humidity and temperature of the environment during the experiments were set at (40±5)% and (25±2)°C, respectively.

To obtain the drying characteristics of jujube, the control or AEEO pretreated jujubes were dried at air temperatures of 60, 70, 80 and 90°C and air velocity of 1.0 m/s. Each drying experiment was repeated twice and the average of drying data was used for data analysis.

2.4 Scanning electron microscopy

The control and pretreated jujubes were dried at 60°C for 2 h, and the skin was cut (about 1 mm thick) and freeze-dried in liquid nitrogen. After sputtered with gold, the coated samples were subsequently examined under a scanning electron microscope (SEM, JEOL JSM-6700F).

2.5 Data analysis

The moisture change of the jujube sample during the drying process was expressed by moisture ratio (*MR*), and calculated as follows[25-32]:

$$MR = \frac{M_t - M_e}{M_0 - M_e} \tag{1}$$

where, M_t, M_0 and M_e are moisture content (kg water/kg dry matter) at a given time, initial and equilibrium moisture content, respectively. The value of M_e is relatively small compared to M_t or M_0 for a long time drying; hence, the *MR* is simplified to:

$$MR = \frac{M_t}{M_0} \tag{2}$$

Drying behaviour of biomaterials can be described by Fick's second law with the assumptions of diffusion-controlled and constant moisture migration, and negligible shrinkage. At a given temperature, the solutions of Fick's second law for spherical samples can be obtained according to the following equation[25,32]:

$$MR = \frac{6}{\pi^2}\sum_{n=1}^{\infty}\frac{1}{n^2}\exp(-\frac{n^2\pi^2 D_{eff} t}{r^2}) \tag{3}$$

where, D_{eff} is the effective diffusivity (m^2/s); r is sample radius (m); t is the drying time (s) and n is a positive integer. For long drying periods, Equation (3) can be further simplified to a straight line equation in the following form[32-36]:

$$\ln(MR) = \mathrm{Ln}(\frac{6}{\pi^2}) - \frac{\pi^2 D_{eff} t}{r^2} \tag{4}$$

Plotting ln(*MR*) versus drying time according to Equation (4) will give a straight line with a slope of k:

$$k = \frac{\pi^2 D_{eff}}{r^2} \tag{5}$$

The effect of temperature on effective diffusivity is usually described by Arrhenius equation[25-36]:

$$D_{eff} = D_0 \exp(\frac{-Ea}{RT}) \tag{6}$$

where, D_0 is the pre-exponential factor (m^2/s); E_a is the activation energy (J/mol); T is the temperature (K) and R is the gas constant (J/(mol·K)).

Mathematical model is an effective means to understand moisture distribution and predict food processing and production. In this study, twelve drying models (Table 1), commonly used in literature to describe the drying characteristics of biological materials, were selected to fit jujube drying data at 60°C using non-linear regression solved by a Levenberg-Marquardt numerical algorithm. The goodness of the fit was evaluated by means of coefficient of determination (R^2), the reduced chi-square (χ^2), the root mean square error (*RMSE*) and the mean relative error modulus (*P*). The model with high R^2 and low *P*, *RMSE* and χ^2 was chosen as the best model for describing the thin-layer drying characteristics of jujube. These parameters were calculated as follows[32,35]:

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}\left(MR_{\exp,i} - MR_{pre,i}\right)^2\right]^{1/2} \tag{7}$$

Table 1 Mathematical models applied to fit jujube drying data

Model	Mathematical expression [a]
Lewis	$MR = \exp(-kt)$
Henderson and Pabis	$MR = a\exp(-kt)$
Logarithmic	$MR = a\exp(-kt) + c$
Page	$MR = \exp(-kt^n)$
Wang and Singh	$MR = 1 + at + bt^2$
Midilli et al.	$MR = a\exp(-kt^n) + bt$
Two term	$MR = a\exp(-k_0t) + b\exp(-k_1t)$
Two term exponential	$MR = a\exp(-kt) + (1-a)\exp(-kat)$
Modified Henderson and Pabis	$MR=a\exp(-kt)+b\exp(-gt)+c\exp(-ht)$
Approximation of diffusion	$MR = a\exp(-kt) +(1-a)\exp(-kbt)$
Verma	$MR = a\exp(-kt) +(1-a)\exp(-gt)$
Weibull distribution	$MR = a - b\exp[-(gt^n)]$

Note: [a] *a*, *b*, *c*, *g*, *h* and *n* are drying constants in models and *k* is the drying rate constant.

$$\chi^2=\frac{\sum_{i=1}^{N}\left(MR_{\exp,i}-MR_{pre,i}\right)^2}{N-z} \qquad (8)$$

$$P(\%)=\frac{100}{N}\sum_{i=1}^{N}\frac{\left|MR_{\exp,i}-MR_{pre,i}\right|}{MR_{\exp,i}} \qquad (9)$$

where, $MR_{exp,i}$ is the moisture ratio derived from the experimental data; $MR_{pre,i}$ is the moisture ratio predicted by a given model; N is the number of observations and z is the number of constants.

3 Results and discussion

3.1 Effects of pre-treatments on jujube drying

The effects of different pre-treatments on drying time were quite different as shown in Figure 2. It took 18.5 h to dry the control jujube to the extent of MR = 0.3 at 60°C. Cutting into halves and partly peeling were found to be two most effective pre-treatment methods, which respectively saved 70.3% and 64.9% of drying time compared with control. However, cutting or peeling makes the resulting jujube lose its intrinsic shape, colour and texture, and the product is hard to be accepted by the consumers. From another point of view, the result confirmed that the outer cuticle is the main barrier for water removal from the fruit.

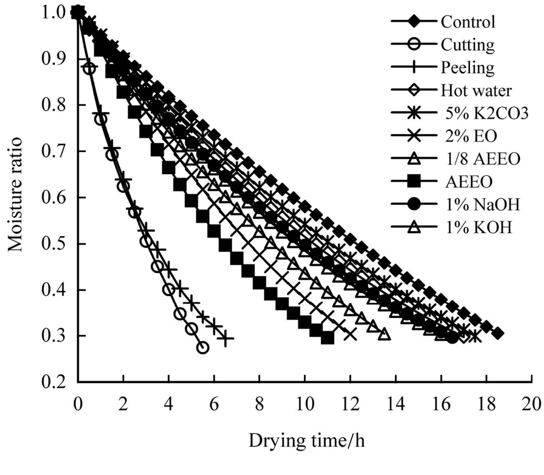
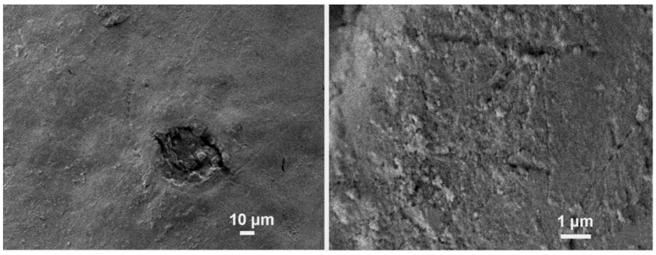


Figure 2 Effects of different pre-treatments on jujube drying at 60°C

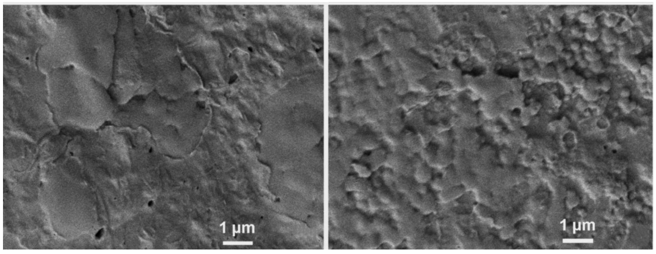
Heat stock, alkaline solutions and surfactants were reported to be effective to shorten drying time by destroying and dissolving the waxy cuticle[22,26-28]. In this study, the results agreed with these reports. The jujube samples pretreated by dipping in hot water, NaOH and KOH solution saved 8.11%, 10.81% and 27.03% of drying time, respectively, compared with control samples. The better effect of KOH than NaOH should be attributed to potassium ion uptake by the guard cells, resulting in the opening of the stomata. Among the chemical pre-treatments, dipping in AEEO spent the shortest drying time (saved by 59.5%), and a concentration-dependent effect was also observed when dipping in its 8-fold dilution (saved by 18.92%). In addition, there was a synergistic effect between K_2CO_3 and EO; because the effect of AEEO was more obvious than that of 5% K_2CO_3 or 2% EO (drying time saved by 5.4% or 35.1%, respectively). The timesaving effect of pre-treatment with AEEO has also been observed in other fruit drying, for example, sour cherry[18], plum[20] and sweet cherry[21].

Under SEM, no individual epidermic cell can be identified, and the stoma is less and sunken (Figure 3a). At a high resolution (×10 000), some irregular etched pattern and micropores can be seen on the surface of jujube pretreated by AEEO (Figures 3c and 3d). In contrast, there were no such structure alterations on the control sample although some tiny scratches could be observed (Figure 3b). The impaired skin of the pretreated jujube may be mainly responsible for its higher drying rate in addition to the benefit of potassium ion.



a. Control jujube, showing epidermis with a stoma at a low magnification (×500)

b. Control jujube showing no impaired surface except tiny scratches (×10 000)



c. AEEO pretreated jujube showing irregular etched pattern and micropores at higher magnification (×10 000)

d. AEEO pretreated jujube showing irregular etched pattern and micropores at higher magnification (×10 000)

Figure 3 Typical surface photographs under SEM of control and AEEO pretreated jujube

3.2 Drying characteristics of AEEO pretreated jujube

The effect of air temperature on the drying of control and AEEO pretreated jujube was examined. As shown in Figure 4, air temperature had a significant influence on the drying behaviour of jujube. In the control samples, drying at 70°C, 80°C and 90°C saved 35.3%, 54.4% and 67.8% of drying time, respectively, compared to drying at 60°C when *MR* reached 0.3. As to the samples pretreated with AEEO, the temperature effect is comparable with the control (saved 34.6%, 52.2% and 62.7% at 70, 80 and 90°C, respectively).

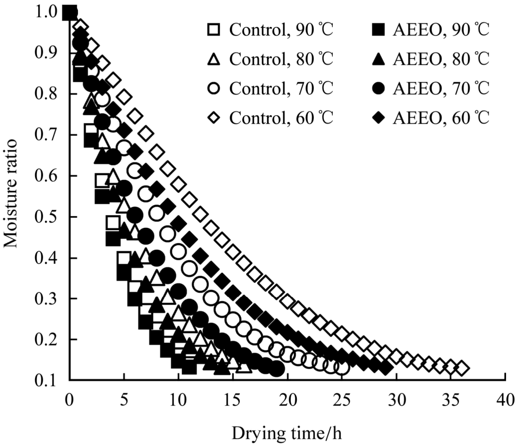


Figure 4 Drying curves of control and AEEO pretreated jujube at different temperatures

What is interesting, though, is that the effect of pre-treatment with AEEO in 2012 (Figure 4) became diluted comparing with that of 2011 (Figure 2), saved 20.6% of drying time at 60°C in 2012 while 59.5% in 2011, when MR reached 0.3. The possible reason for this difference might be that the local weather was dry during the fruit maturing in 2012, thus the thicker cuticle developed and the greater the fresh jujube was resistant to AEEO pre-treating. This assumption was supported by the longer time spent for drying the control samples at 60°C to *MR* = 0.3 (20.6 h in 2012 vs. 18.5 h in 2011). As to the AEEO pretreated samples, the drying time reduced 19.7%, 16.9% and 8.0% at 70°C, 80°C and 90°C, respectively, compared to drying at 60°C, which indicated that the pre-treating effect became weak with the increase of air temperature. According to this result, drying at a higher temperatures (exceed 80°C) did not need pre-treatment with AEEO, which agreed with the suggestion of Price et al.[29] for plum drying.

Drying rate curves can be applied to interpret the variation of moisture diffusion in the sample[22]. The nature of falling rate drying has been proved previously in hot-air and microwave drying of jujube[6-10,14]. In this study, a similar result was obtained as shown in Figure 5. It can be seen that no constant drying rate period was detected, and the drying behaviour at 60 - 90°C belongs to a falling rate drying, except the first two hours, when the inside temperature of the sample was at a low level and then gradually increased, leading to a short increasing rate drying period.

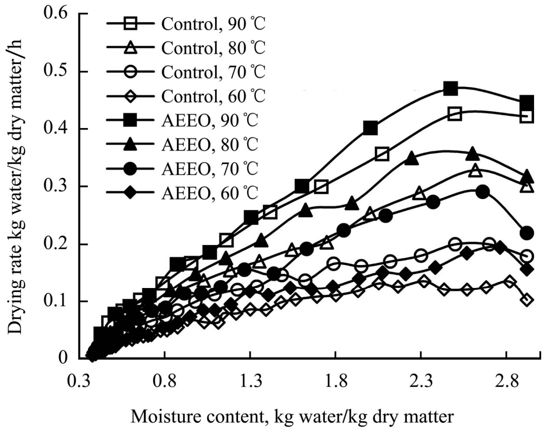


Figure 5 Drying rate of control and AEEO pretreated jujube at different temperatures

As it can be seen in Figure 5, the drying rate generally increased with air temperature increasing at the same moisture content, and reduced with the decrease of moisture content two hours later. The drying rate of AEEO pretreated samples was higher than the corresponding controls, indicating that moisture transport from inside to surface was accelerated by the pre-treatment, but the increased degree reduced gradually with moisture content decreasing. This result indicates that the pre-treatment mainly improves the drying rate in the early drying period, which agrees with the finding of Pahlavanzadeh et al.[23] in grape drying.

Although drying at a higher temperature could substantially boost the productivity, but led to a product with brown appearance[30], which was unacceptable for jujube drying. In view of the significant effect of AEEO pre-treatment at a relatively low temperature, we recommend jujube drying at 60°C after pre-treatment with AEEO.

3.3 Effect of AEEO pre-treatment on drying parameters of jujube

No straight line was obtained when plotting LnMR versus drying time as shown in Figure 6. By regression analysis, three linear segments were identified with $R^2 >$ 0.98. The result indicates that the whole drying period can be divided into three stages, and each of them follows the first order reaction kinetics. This finding may be equivalent to the result of Fang et al.[11], who also divided the drying process of jujube into three segments by examining the shrinkage of the fruit. Motevali et al.[8] has defined two falling rate periods in thin-layer drying of jujube. The dehydration mechanism may be different in these three drying stages. In the first falling rate stage, the drying rate is higher, losing about 30% (28.9% - 35.4%) moisture in a relatively short time, accounting for 16.7% to 21.4% of total drying time (Table 2), and free water removal might dominate this process. In the second stage, the drying rate decreased, about 55% moisture was removed in 53.8% - 62.5% of drying time, which may be held by capillary condensation and multi-molecular adsorption. The third stage lasted for 18.8% - 27.3% of drying time, while only 3.1% - 7.3% of total moisture was removed (Table 2), which means that the water may be held in mono- and/or multi-molecular layers by strong attractive molecular forces[31]. This phenomenon was also found in the study on hot air drying of steam-blanched chrysanthemum[32].

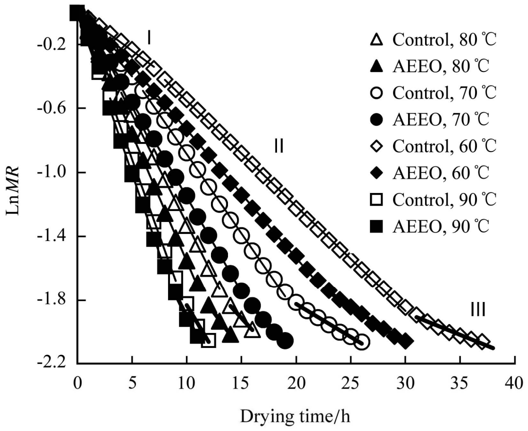


Figure 6 Drying stages of control and AEEO pretreated jujube at different temperatures. I, II and III denote the first, second and third stages, respectively

Table 2 Drying parameters of control and AEEO pretreated jujube at different drying stages

Samples	Temperature /°C	Effective diffusivity ($\times10^{-10}$ m^2/s)			Activation energy (kJ/mol)		
		First stage	Second stage	Third stage	First stage	Second stage	Third stage
Control	60	2.2209	2.8894	1.2226	41.45	35.24	49.52
	70	3.5491	4.3495	1.7460			
	80	5.5721	5.9899	3.3424			
	90	7.5292	8.3428	5.0927			
AEEO	60	3.0565	3.8877	2.0230	32.78	27.45	35.17
	70	4.8685	5.2159	3.2544			
	80	6.3461	6.9662	4.0680			
	90	8.2636	8.7606	6.0163			

The D_{eff} values were calculated based on Equation (5); the slopes of the linear segments were shown in Table 3. The D_{eff} values obtained in this study lie within the general range of 10^{-12} m^2/s to 10^{-8} m^2/s for drying of food materials[33] and were also in the same magnitude of 10^{-10} m^2/s referring to other studies of jujube drying[6,8,13,14]. It can be seen that the D_{eff} value increases with drying temperature regardless of drying stage and pre-treatment. At a given drying condition, the highest D_{eff} value was found in the second stage while the lowest in the third stage. The relatively low D_{eff} value in the first falling rate stage may be in part due to the low internal temperature of the fresh jujube as mentioned above. In the third stage, both binding water inside and surface case hardening may result in its low D_{eff} value. When drying at 60°C, AEEO pre-treatment led to D_{eff} increased by 37.6%, 34.6% and 65.5% in the three stages, respectively; however, the effect reduced at a higher temperature. The satisfactory effect of AEEO pre-treatment on the third stage might be due to the easiness of forming microfissures on the fruit surface upon shrinkage during the drying process. The D_{eff} values may be, to some extent, overestimated for the fruit shrinkage, especially in the second and third stages.

By means of plotting $\ln D_{eff}$ versus $1/T$ according to Equation (6), the E_a values were calculated as shown in Table 3, which fell within the range of 12.7 - 110 kJ/mol for most food materials[33], and were comparable to the Ea values of jujube drying (ranging from 34.97 kJ/mol to 74.20 kJ/mol) reported in literatures[6,8,13]. The differences may be due to the drying equipment and condition, the data enrolled for Ea calculation, or the material used of different cultivars, growing conditions and/or ripeness. In contrast to control, AEEO pre-treatment resulted in an Ea reduction by 20.9%, 22.1% and 29.0% for the first, second and third stage, respectively. The result indicates that lower energy barrier needs to be overcome for water diffusion from the interior of jujube after pretreated with AEEO. Similar effect was also found in the drying of tomato[17] and pomegranate arils[34], while a negative effect was also observed in the pre-treatment of sweet cherry[21].

Table 3 Statistical criteria for models predicting control and AEEO pretreated jujube drying at 60°C

Sample	Model name	χ^2 ($\times 10^{-4}$)	$RMSE$ ($\times 10^{-3}$)	P/%	R^2
Control	Lewis	3.778	19.180	4.0826	0.9951
	Henderson and Pabis	1.698	12.680	3.0515	0.9982
	Logarithmic	1.714	12.560	3.2737	0.9981
	Page	1.328	11.220	3.8222	0.9983
	Wang and Singh	0.819	8.807	2.3011	0.9990
	Midilli et al	0.068	2.474	0.8776	0.9983
	Two term	0.649	7.621	2.7351	0.9993
	Two term exponential	1.668	12.570	3.3450	0.9972
	Modified Henderson and Pabis	1.795	12.290	0.5662	0.9982
	Approximation of diffusion	0.633	7.634	2.3011	0.9990
	Verma	0.632	7.631	2.4754	0.9992
	Weibull distribution	0.986	9.392	2.6160	0.9993
AEEO pretreated	Lewis	1.107	10.350	3.1578	0.9979
	Henderson and Pabis	0.854	8.939	3.0901	0.9988
	Logarithmic	0.734	8.144	2.2865	0.9989
	Page	0.970	9.523	3.3492	0.9991
	Wang and Singh	1.276	10.920	3.8776	0.9983
	Midilli et al.	0.117	3.198	0.7150	0.9999
	Two term	0.515	6.700	1.4798	0.9991
	Two term exponential	0.445	6.453	1.4967	0.9992
	Modified Henderson and Pabis	0.557	6.700	1.4719	0.9988
	Approximation of diffusion	0.768	8.327	3.0252	0.9989
	Verma	0.769	8.334	3.343	0.9991
	Weibull distribution	1.080	9.709	3.519	0.9981

3.4 Modelling of jujube drying

The resulting values of χ^2, $RMSE$, P, and R^2 for different models were calculated and summarized in Table 3. In all cases, the R^2 values were greater than 0.99 (0.9951-0.9999), indicating the fitness is good[35]. The P values, varying from 0.5662% to 4.0826%, were acceptable because a P value lower than 10% was recommended for the selection of models[35,36]. The χ^2 and $RMSE$ values were changed from 6.838×10^{-6} to 3.778×10^{-4}, 2.474×10^{-3} to 1.918×10^{-2}, respectively. With overall consideration of four parameters, the model by Midilli et al. was considered to be the appropriate one to describe the drying behaviour for both control and AEEO pretreated jujube at 60°C. The mathematical expressions for control and AEEO pretreated samples are given as follows, Equation (10) and Equation (11), respectively:

$$MR = 0.9989 \exp(-0.03536t^{1.2106}) + 0.001780t \quad (10)$$

$$MR = 0.9981 \exp(-0.05867t^{1.1182}) + 0.001895t \quad (11)$$

The results agreed with the previous reports[8,9,13,14]. Other models, such as Verma model, Page model and Weibull distribution model, have also been used to predict the hot-air drying of Chinese jujube[6,7,10], but these models are not suitable according to the experimental data.

4 Conclusions

In order to improve the efficiency of hot-air drying of Chinese jujube, nine pre-treatments were applied in this study. According to our data, pre-treatment with AEEO and drying at 60°C was recommended. Its timesaving effect was considered to be due to stomatal opening and cuticle destruction. The falling rate drying was divided into three stages, each of them followed the first order reaction kinetics. The activation energy for the first, second and third stages reduced by 20.9%, 22.1% and 29.0% by AEEO pre-treatment, respectively. In addition, The model by Midilli et al. was preferable for the prediction of jujube drying.

Acknowledgements

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