Effects of pre-treatments on drying kinetics and energy consumption, heat-mass transfer coefficients, micro-structure of jujube (Zizyphus jujuba L.) fruit

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Abstract
In this study, jujube fruits were investigated effects on drying kinetics and energy consumption (total-specific), heat-mass transfer coefficients, micro-structure of pre-treatments 2% ethyl oleate, 540 W microwave and freeze-thaw + 720 W microwave. Initial moisture of fruit from 0.75 ± 0.2 g (water).g-1 (dry matter) to 0.15 ± 0.1 g (water).g-1 (dry matter). The shortest drying durations were observed in 2% ethyl oleate pre-treated samples. The greatest total power consumption (1.502 kW) was observed in control samples. The highest energy consumption (158.98 kW.kg-1) was observed in freeze-thaw pre-treated samples. The effective diffusion values varied between 9.53 x 10-4 - 5.26 x 10-8 m2.s-1. Lewis and Jena-Das models the best estimated time-dependent moisture ratios in freeze-thaw pre-treated samples. The average speed values was determined between 0.0025 - 0.0005 (gr db.minute-1) at the beginning of the drying and decreased to about 0.0005 (gr db.minute-1) at the lasting of the drying. The largest mass transfer coefficient value was found depending on the time varied between 1.035 x 10-7 - 8.256 x 10-12 m.s-1. In the samples dried by dipping into a 2% ethyl oleate solution. The average heat transfer coefficient value is calculated 0.204 W.m-2 °C-1. With regard to micro-structure of the dried samples, 2% ethyl oleate pre-treatments yielded the least deformations and had the closest structure to fresh samples.

Keywords: drying process; drying pre-treatments; effective diffusion; energy values.

Practical Application: The aim of this study is to dry the jujube fruit under the most suitable conditions. While doing this, the effect of some pre-treatments (2% ethyl oleate, 540 W microwave and freeze-thaw) on energy analysis and quality characteristics was investigated originally from the literature. The highest energy consumption was determined in the drying process of the samples applied freeze-thaw pretreatment. The quality feature was determined in the drying of the samples dipped in 2% ethyl oleate solution.

1 Introduction
Freshly harvested agricultural products generally have quite high moisture content (75-95%). Therefore, physical, chemical and nutritional attributes can easily be influenced by surrounding environments. Such products are generally preserved by drying (Doymaz, 2011; Ghanbarian et al., 2020). With the drying of the products, the losses in nutritional and visual quality properties are reduced to a minimum and food safety is ensured (Moloto et al., 2021).

Throughout the process of drying, various physical and chemical changes occur at different levels based on drying temperature, duration and moisture content to be achieved. Therefore, drying methods and pre-treatments should be so selected as to control undesired conditions and to keep the final quality values at desired levels (Wang & Brennan, 1995; Rubinskiene et al., 2015; Majdi & Esfahani, 2019). Producers generally lay out the agricultural products over trays or concrete surface to dry them through moisture diffusion by the heat generated with the photons coming from the sun (Wojdylo et al., 2014; Panagopoulou et al., 2019). In such natural drying processes at open spaces, drying takes quite a long time, products are exposed to solar heat for longer durations, thus significant losses in quality parameters are experienced. With these methods, it is impossible to get dry and healthy products in a short time (Doymaz & Pala, 2003; Özgen, 2015; Polatci & Taşova, 2018). On the other hand, to eliminate such problems experienced in open-space drying processes and to control drying conditions, more sensitive scientific drying approaches were developed for the best preservations of quality and nutritional attributes of agricultural products. Such approaches include oven, vacuum, microwave, vacuum-microwave and freeze-drying methods (Wojdylo et al., 2014; Panagopoulou et al., 2019).

It is quite significant to preserve final quality of dried products. However, drying methods are expected to be reliable and economic in energy consumption. Therefore, some physical and chemical pre-treatments are applied to products to enlarge pores and accelerate the moisture removal rates and ultimately to shorten drying durations. Shortened drying durations will also reduce energy consumptions, reduce the impacts of non-enzymatic reactions, thus improve final quality parameters. Rojas & Augusto (2018a); used chemical-dipping pre-treatments and reported reduced drying durations in pumpkins (Rojas & Augusto, 2018b); bananas (Corrêa et al., 2012); rice and pea powder mixtures (Tatemoto et al., 2015). It was reported that...
electromagnetic pre-treatments shortened drying durations in apples (Brncic et al., 2010) and pears (Yao, 2012).

Jujube is a fruit of Chinese origin and has been cultivated for 4000 years. It is thought to be one of the 5 most valuable fruits in China such as peach, apricot, plum and pear. It is widely grown in Russia, India, Middle East, Anatolia, Southern Europe and North Africa after China (Yao, 2012; Gulcuoglu & Baspinar, 2020). Approximately 90% of jujube production in the world is made in China (Li et al., 2005; Wang et al., 2016). In Turkey, production is made around 1772 acres of the 960 tons. The highest production is in Amasya with 281 tons and Antalya with 204 tons (Kaplan & Okcu, 2020).

It was also reported that jujube fruits had healing effects on lung diseases. Due to its texture and water content, it is a delicate fruit and can remain at room temperature for up to a week without shrinking or darkening (Moradinezhad et al., 2018). Therefore, jujube fruits are mostly consumed as dried. There are studies investigating the effect of pre-treatments applied before drying for jujube fruit (Wojdylo et al., 2019); they investigated the most suitable method in terms of final quality values by drying the jujube fruit in vacuum microwave (480, 120 W) and hot air dryer (50, 60 and 70 °C). They found that the process performed at a temperature of 50 °C in a hot air dryer was better in terms of color, polyphenol and antioxidant properties. However, they found that the energy consumption was several times higher than the vacuum microwave dryer (Tepe & Ekinci, 2021); they investigated the effect of temperature values on water-soluble vitamin, total phenol and atopal antioxidant properties by drying jujube fruit with a hot air dryer (50, 60 and 70 °C). Water-soluble vitamins, total phenolic content, and antioxidant capacity were significantly reduced by the drying process. Degradation of water-soluble vitamins increased with the drying temperature, although total phenolic content and antioxidant capacity were not significantly affected by temperature (Niu et al., 2021); they dried the jujube fruit at three different air velocities (3.6 and 9 m.s⁻¹) in a hot air dryer (55, 60, 65 and 70 °C). They investigated the effects of drying conditions on vitamin C, drying pattern and activation energy values. They reported that vitamin C is broken down by drying processes and the activation energy varies between 36.48-153.51 kJ.mol⁻¹.

The primary purposes of this study are: (i) comparing the drying durations of three drying pre-treatments with relation to the kinetics, (ii) selecting the most favourable thin-layer drying model and lastly, (iii) determining effects of effective diffusion (Deff) values to drying pre-treatments, (iii) identifying the variations between the dried samples regard to last moisture content and specific energy consumption, total energy consumption features, micro-structure.

2 Material and method

2.1 Sample preparation

Wild jujube fruits to be used in present experiments were supplied from Aksaray province/Turkey. Samples were brought to laboratory under reliable conditions and preserved in a fridge at +4 ± 0.5 °C until the time of analysis. Drying experiments were conducted at drying laboratory of Tokat Gaziosmanpaşa University Agricultural Faculty. To get wet basis moisture content, about 30 ± 0.5 g sample was taken, dried in an oven at 70 °C until a constant mass and reweighed (Yaşcioğlu, 1999).

2.2 Drying pre-treatments

Some pre-treatments were applied to jujube fruits before the drying process to reduce drying durations and energy consumption values and preserve micro-structure. Pre-treated fruits were dried separately and compared with the control fruits. There are studies on the effects of pre-treatments applied to jujube fruit (Table 1).

Present pre-treatments included; 1) Immerse into 2% ethyl oleate solution for 10 min; 2) Intermittent microwave application of 540 W for 2 min; 3) Intermittent microwave application of 720 W to freeze-thawed samples for 1.5 min. Fruit pores were enlarged with these pre-treatments to accelerate mass diffusion and to increase drying rates.

2.3 Drying equipment and process

Şmuşek Laborteknik-brand ST-120 (Turkey) type oven was used in drying experiments. Drying air temperatures were controlled with PID controllers on dryer. About 28 ± 0.5 g fresh fruits were used in drying processes. Pre-treated and control samples were dried constant 65 °C drying air temperature. In many studies, jujube fruit has been dried at temperatures of 50, 60 and 70 °C (Izlı & Polat, 2019); in the study, they determined the optimum drying temperature at 60 °C in terms of rehydration, color, and microstructure analysis.

2.4 Theoretical thin layer drying models

Time-dependent dimensionless moisture ratio (MR) released from the pre-treated and control samples at different drying pre-treatments were calculated with the aid of Equation 1 (Maskan, 2000).

\[
MR = \frac{M_t - M_e}{M_0 - M_e}
\]  (1)

where;

MR: Moisture ratio
M₀: Instant moisture content
Mₑ: Equilibrium moisture content
Mₜ: Initial moisture content

The drying speed values at different drying pre-treatments were calculated with the aid of Equation 2.

\[
DS = \frac{N(t) - N(t + \Delta t)}{\Delta t}
\]  (2)

where;

DS: Drying speed
N₀: Moisture content t time
Δt: after t time
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Jena and Das and Lewis equations were used to model drying curves generated for jujube fruits. Model equations are provided in Table 2.

### 2.5 Diffusion coefficient \((D_{eff}, m^2/s)\)

The area in which the moisture released from pre-treated and control fruits during the oven drying process is diffused was calculated with the aid of Equation 3 (Crank, 1979; Türker & İşleroğlu, 2017).

\[
MR = \frac{M - M_e}{M_0 - M_e} = \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp \left[ \frac{(2n+1)^2}{4L^2} \right]
\]

where; \(D_{eff}\): effective diffusion \((m^2.sn^{-1})\), \(L\): half of slice thickness (m). Then, natural logarithm of the equation was taken and following equation (Equation 4) was obtained (Doymaz, 2007).

\[
\ln\text{MR} = \ln \frac{8}{\pi^2} - \frac{\pi^2 D_{eff} t}{4L^2}
\]

Resultant moisture ratios (MR) were plotted against drying durations in a line-graph and \(D_{eff}\) values were calculated from the slope of the resultant lines (Zakipour & Hamidi, 2011; Motevali et al., 2011).

### 2.6 Energy consumption values

Energy consumptions were determined for each drying process with the aid of Polaxtor-brand PLAX-15366 model power analyzer (Motevali et al., 2011); specific energy consumptions were calculated with the use of the changes in time-dependent mass loss (Equation 5).

\[
SEC = \frac{TEEC}{TWL}
\]

where;

- \(SEC\): Special energy consumption \((kW.kg\text{ water}^{-1})\)
- \(TEEC\): Total electric energy consumption \((kW)\)
- \(TWL\): Total water loss \((kg)\)

### 2.7 Convective mass transfer coefficient \((h_m)\)

The effect of different drying pretreatments on the convective mass transfer coefficient \((h_m)\) values of jujube fruit was calculated by Equation 6 (Lahsasni et al., 2004; Daş et al., 2021).

Table 1. Effect of various pretreatment processes on the drying characteristics and quality of jujube.

| Pre-treatment and application method | The advantage it provides | References |
|-------------------------------------|---------------------------|------------|
| Cold plasma pre-treatment-Power 650 W, the gas flow 0.2 Mpa, working voltage of 5 kV and frequency of 40 kHz. Air is used to generate plasma at atmospheric pressure, and the flow rate of plasma afterflow was 135 L.min⁻¹. Soaking pre-treatment-in a solution of 5% potassium carbonate and 0.5% olive oil Soaking pre-treatment-ethyl oleate solution followed by slow freezing at -18 °C high pressure carbon dioxide (HPCD), and hot water blanching (HWB). Carboxylic acid (CO₂) pre-treatment-containing 5% CO₂ and 95% N₂ by modified atmosphere packaging machine. Soaking pre-treatment-boiling treatment with 3% sodium chloride dipping treatment in glycerol. Carbon dioxide (CO₂) pre-treatment-boiling treatment with 3% sodium chloride dipping treatment in glycerol. Soaking pre-treatment-dipping in 2% ethyl oleate plus 5% K₂CO₃ for 10 min. | The pre-treatment improved the contents of procyanidins, flavonoids, and phenolics by 53.81%, 33.89%, and 13.85% at most, respectively, and thereby enhanced antioxidant capacity by 36.85% at most. The pre-treatment positive effect on effective diffusion, total phenol, total antioxidant and total color change properties. The method of AEEO + freeing pre-treatment damaged epidermis structure of jujube. This pre-treatment led to the moisture in jujube much more easily diffused and evaporated. The AEEO + freeing groups were the minimum and the HWB groups were the maximum. The pre-treatment changes the texture and aroma of jujube that caused an accumulation of acetaldehyde and ethanol. The bulk density was highest with DTG. The soluble solids content was highest with. The titratable acidity was higher with DTG, and BTS showed the best browning-retarding effect,. Time-saving effect. The beneficial effect was considered based on its cuticle destruction. Activation energies decreased. | (Bao et al., 2021) (Doymaz et al., 2016) (Dongsheng et al., 2017) (Chen et al., 2017) (Kim et al., 2013) (Baomeng et al., 2014) (Xu et al., 2019) |

Table 2. Thin layer drying models.

| Models | Equation | References |
|--------|----------|------------|
| Jena ve Das | \(MR = \text{hexp} \left(-J(\chi^2)\right) + (\text{mr})\) | (Jena & Das, 2007) |
| Lewis | \(MR = \exp(-lt)\) | (Lewis, 1921) |
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\[
l_h = \frac{V}{A_m} \cdot \ln(MR)
\]

where;

\(h_m\): Convective mass transfer coefficient \((m \cdot s^{-1})\)

\(V\): Material volume \((m^3)\)

\(A_m\): Material surface area \((m^2)\)

\(t\): Time \((s)\)

2.8 Convective heat transfer coefficient \((h_c)\)

The effect of different drying pretreatments on the convective heat transfer coefficient \((h_c)\) values of jujube fruit was calculated by Equations 7-14 (Lahsasni et al., 2004; Daş et al., 2021).

\[
N_u = 0.664 \sqrt{Re \cdot Pr}
\]

\(N_u = \frac{h_c \cdot L}{K_v}\)

\[
R = \frac{L \cdot v \cdot \mu_v}{\mu}
\]

\[
P = \rho \cdot C_v \cdot T
\]

\[
P_v = \frac{353.44}{T + 273.15}
\]

\[
K_v = 0.0244 + \left( 0.677 \times 10^{-4} \cdot T \right)
\]

\[
C_v = 999.20 \times 0.1434 T \times 1.101 \times 10^{-4} \cdot T^2 - 6.7581 \times 10^{-8} \cdot T^3
\]

\[
\mu_v = \left( 1.718 \times 10^{-5} + 4.620 \times 10^{-8} \cdot T \right)
\]

2.9 Micro-structure analysis

Samples were taken from dried products and a longitudinal section was taken from the peals right from the center of product. The section was placed into distilled water between slide-lamella of Olympus-brand light microscope. The deformations in fruit peel cell walls were imaged under 400x imaging of light microscope (Eim et al., 2013).

3 Results and discussion

3.1 Drying performance values

The wet-basis initial moisture content of jujube fruits was measured as 42.67% and fruits were dried to an average dry-basis moisture content of 0.15 ± 0.1 g \((water) \cdot g^{-1} \) (dry matter). Drying durations of pre-treated and control fruits are provided in Table 3.

As can be inferred from Table 2, pre-treatments influenced drying durations. The longest drying duration (580 min) to bring the fruits to a desired range of moisture (10-15%) was observed in control fruits and the shortest drying duration (510 min) was observed in 2% ethyl oleate-treated fruits. Data showing the effect of pre-treatments on drying speed values is given in Figure 1.

Pre-treatments reduced drying times by 6.90-12.07% compared to control (An et al., 2019); when they dried the black mulberry fruit at 60 °C, the ethyl oleate pretreatment reduced the drying time by 17.17-40.70% (İzlı et al., 2017); in the drying process of mango samples at 60, 70 and 80 °C temperatures, the drying times were determined as 175, 140 and 95 min, respectively (Aydar, 2021). It has been determined that the increase in ultrasound pretreatment and power values in the drying process of olive leaves reduces the drying time by 42.50% on average. Data showing the effect of pre-treatments on % moisture content cumulative values is given in Figure 2.

3.2 Theoretical thin layer drying model values

Drying curve coefficients, \(R^2\) and \(p\) values of thin layer drying models for jujube fruits are provided in Table 4.

As can be inferred from Table 3, among the thin layer drying models used in this study, Lewis and Jana-Das models the best estimated time-dependent moisture ratios during the drying process of jujube fruits. The \(R^2\) value of these models was identified as 0.9988 and the best estimations were achieved in free-thaw pre-treated fruits.

3.3 Effective diffusion values

While calculating effective diffusion values of dried jujube fruits with different pre-treatments, required time-dependent \(\ln MR\) values and the linear graph are presented in Figure 3 and effective diffusion coefficients are provided in Table 5.

As can be inferred from the equations of time-dependent \(\ln MR\) lines, the greatest \(R^2\) \((0.9986)\) was observed in control samples and the lowest \(R^2\) \((0.9463)\) was observed in 2% ethyl oleate pre-treated samples. Effective diffusion coefficients for drying processes of control and pre-treated jujube fruits are provided in Table 5.

As can be inferred from Table 5, the greatest and the lowest effective diffusion coefficients were respectively observed in 2% ethyl oleate and control treatments. Pre-treatments influenced effective diffusion coefficients and increased the effective diffusion as compared to the control samples. It has been reported that pre-treatments performed improve the drying kinetics on fruits okra; (Tüfekçi & Özkal, 2017), kiwifruit; (Nowacka et al., 2014), shiitake mushrooms; (Zhao et al., 2019).
Figure 1. The drying speed values of dried samples.

Figure 2. The % moisture content cumulative change values of dried samples.

Table 4. Values of mathematical models.

| Pre-treatments | Models   | k   | h     | j     | m     | R²    |
|----------------|----------|-----|-------|-------|-------|-------|
| Control        | Lewis    | 0.0028 |      |       |       | 0.9957 |
|                | Jena-Das | 1.0154 | 0.4053 | 0.8049 | 0.0117 | 0.9978 |
| 2% Ethyl oleate| Lewis    | 0.0034 |      |       |       | 0.9979 |
|                | Jena-Das | 1.0107 | 0.4056 | 0.8043 | 0.0070 | 0.9987 |
| 540 W          | Lewis    | 0.0028 |      |       |       | 0.9983 |
|                | Jena-Das | 1.0030 | 0.4053 | 0.8050 | -0.0015 | 0.9983 |
| Freeze-thaw    | Lewis    | 0.0027 |      |       |       | 0.9988 |
|                | Jena-Das | 1.0009 | 0.4052 | 0.8050 | -0.0039 | 0.9988 |

3.4 Energy consumption values

Total and specific energy consumption graphs for pre-treated and control samples are respectively presented in Figures 4-5.

According to Figure 4, the greatest total power consumption (1,502 kW) was observed in control samples and the lowest total power consumption (1.352 kW) was observed in 2% ethyl oleate-treated samples. It was observed that pre-treatments influenced total power consumption values (Figure 5). It has been understood from the findings of the study that there is an inverse relationship between the drying temperature and the energy consumption values (0.31-0.43 kWh) (Alibas & Köksal, 2014).

As can be inferred from Figure 5, specific energy consumption of pre-treated samples continuously increased during the initial 100 min of drying process, then a parabolic decrease was observed.
in specific energy consumptions. Such a case revealed that initially power consumptions were greater than the removed moisture, then removed moisture was greater than power consumption. Convective mass transfer coefficient values depending on time are given in Figure 6.

### 3.5 Convective mass transfer coefficient ($h_m$)

The effect of drying pre-treatments on convective mass transfer coefficient was calculated (Figure 6).

According to Figure 6, it is seen that the effect of pre-treatments performed before drying on convective mass transfer coefficient values is significant. It changed the convective mass transfer coefficient values of the pre-treatment of dipping in 2% ethyl oleate solution more than other pre-treatments.

### 3.6 Convective heat transfer coefficient ($h_c$)

The effect of drying pre-treatments on convective heat transfer coefficient was calculated. It was determined that the average heat transfer coefficient value this study is calculated 0.204 W.m$^{-2}$.°C$^{-1}$ (Jain & Tiwari, 2004); cabbage and peas dried sera tip in a dryer to a certain amount of moisture. They calculated that the convective heat transfer coefficient took values between 0.16-0.36 W.m$^{-2}$.°C$^{-1}$ (Kaya et al., 2006); they calculated that the convective heat transfer coefficient varied between 4.33-96.16 W.m$^{-2}$.°K$^{-1}$ (0.016-0.37 0.16-0.36 W.m$^{-2}$.°C$^{-1}$) in their drying studies. There are similar findings in the literature.
3.7 Micro-structure images

Micro-structure images of pre-treated and untreated control samples taken under light microscope are presented in Figure 7.

Figure 4. Total power consumption values.

Figure 5. Specific power consumption values.

Figure 6. Convective mass transfer coefficient ($h_m$).

Micro-structures presented in Figure 7, revealed direct information about rehydration of dried samples and indirect information about nutritional values. Image of fresh fruits revealed that jujube cells were full and intercellular spaces were distinctive. Images of control samples revealed deformations in upper epidermis cells and the underlying 2-3 rows of cell. The image of 2% ethyl oleate-treated fruits revealed that microstructure of the treated fruits was close to fresh samples, slight
shrinkage was observed in upper cells, but cellular disintegration was not observed at all (Ando et al., 2019); carrot dried in microwave and conventional ovens and at different temperatures. The freeze-thaw pretreatment determined that this pretreatment has a positive effect on the microstructure. Partial deformations were observed in upper epidermis cells of 540 W microwave-treated samples and these samples had the best micro-structure after 2% ethyl oleate-treated samples. For freeze-thaw samples, significant deformations were observed in upper epidermis cells and underlying 4-5 rows of cells. As compared to fresh and the other pre-treated samples, freeze-thaw pre-treatments the worst preserved micro-structure of the fruits.

4 Conclusions

In this study, effects of different pre-treatments (2% ethyl oleate, 540 W microwave and freeze-thaw + 720 W microwave) on drying durations, power consumption and micro-structures of oven-dried (60 °C) jujube fruits were investigated. Better outcomes for investigated parameters were achieved with 2% ethyl oleate pre-treatments. The shortest and the longest drying durations were respectively observed in 2% ethyl oleate pre-treated and the control samples. Lewis and Jena-Das models the best estimated time-dependent moisture ratios in freeze-thaw pre-treated samples. The greatest power consumption (1.502 kW) was observed in control samples and the lowest power consumption (1.352 kW) was observed in 2% ethyl oleate pre-treated samples. It was determined that the average speed values started between 0.0025-0.002 (gr db/min) at the beginning of the drying and decreased to about 0.0005 with the decrease of the moisture content. The greatest effective diffusion value (5.26 x 10^{-8} m^2.s^{-1}) was observed in 2% ethyl oleate pre-treated samples and the lowest effective diffusion (9.53 x 10^{-8} m^2.s^{-1}) was observed in control samples without any pre-treatments. In the study, it was found that the largest mass transfer coefficient value depending on the time varied between 1.035 x 10^{-7}-8.256 x 10^{-12} m.s^{-1} in the samples dried by dipping into a 2% ethyl oleate solution. It was determined that the average heat transfer coefficient value this study is calculated 0.204 W.m^2 °C^{-1}. With regard to micro-structure of the dried samples, 2% ethyl oleate pre-treatments yielded the least deformations and had the closest structure to fresh samples. It was concluded based on present findings that jujube fruits should be immersed into 2% ethyl oleate solution before drying and then dried accordingly to get shorter drying durations, lower power consumptions and better micro-structures.

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