

Effects of chemical pretreatments on air-drying process of black mulberry (*Morus nigra* L.)

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For drying as the preservation method, black mulberries (*Morus nigra* L.) placed into sample bags were dipped into one of six different chemical solutions for one minute at room temperature to accelerate the moisture loss. Pretreated black mulberry samples were dehydrated by artificial air-drying, solar drying and open sun drying. The samples treated by 2% ethyl oleate were the fastest ones reaching to the final weight loss percentage (75%) in 22 h by the laboratory tray dryer providing 45.2 °C drying air (artificial air drying) and in 90 h by solar drying. The pretreatment combination caused only slight changes in measured color values of mulberries during all drying trials.

Keywords: Air-drying, Black mulberry, Ethyl oleate, Open sun drying, Solar drying

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Introduction

Any drying process to preserve agricultural materials is meant for the retention of product quality, reduction of operational/investment cost and protection of environment¹. The retention of product quality typically increases the cost of drying process such as in freeze drying². This difficulty has been tried to alleviate by developing new drying strategies such as heat pump drying, microwave drying and radio frequency drying¹⁻³ or various chemical pretreatment processes⁴⁻⁷ in both industrial and scientific arenas. On the other hand, solar drying and open sun drying of agricultural materials are extensively practiced all over the world because of their ease of operation and low cost⁸⁻¹⁰.

Black mulberry (*Morus nigra* L.) is used in treatment of type II diabetes mellitus, inflammations of throat, tongue and mouth, and strengthening the solid tissues of teeth¹¹. It is good source of phenolic compounds and anthocyanin (2000-2600 mg⁻¹ as juice), which is four times higher than in grapes¹¹. Anthocyanin from mulberry may reduce the incidence of heart diseases with its anti-atherosclerotic activity by inhibiting the oxidation of LDL in the arterial wall¹².

Mulberry fruits are normally harvested by handpick or by shaking tree branches and collecting the fruits over a sheet on the ground. Shelf life of fresh mulberries is very short; therefore, they should be processed for preserving as juice, jam, leather, frozen fruit, dried fruit, etc. immediately after harvesting¹¹.

This research presents appropriate chemical pretreatments for black mulberries dehydrated by three different drying methods (artificial air drying, solar drying and open sun drying).

Materials and Methods

Black mulberry fruits were handpicked from a local fruit farm in Tokat city located in the Central Black Sea Region of Turkey. Moisture content of fresh fruits was determined according to the vacuum oven method (T=70°C). Fruits (75-80g) were placed into sample bags made of plastic net. Six different pretreatment solutions (T₁-T₆) were prepared as follows (% w/v): T₁ 2 % ethyl oleate; T₂, 1% NaOH; T₃, 1 % citric acid; T₄, 1 % ethyl oleate; T₅, 1 % ethyl oleate + 1 % citric acid; and T₆, water (control). Dipping temperature corresponded to room temperature (23°C) without heating chemical solution. Sample bags were dipped into each solution for 1 min. at room temperature. Right after rinsing with

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Table 1 — ANOVA for weight loss percentage (WLP) values of black mulberries dried in laboratory tray dryer (artificial drying)

Source	Df ^a	F-values			
		6 h	12 h	24 h	36 h
Chemical solution (CS)	5	6.07*	7.81*	15.19*	25.90*

^aError 12 and total 17; *Statistically-significance at $\alpha = 0.01$

water, bags were placed over paper towels to remove free water flowing over them.

Pretreated fruits were dried by artificial air drying using a laboratory tray dryer, solar drying using a greenhouse dryer and open sun drying (under open atmosphere). There were three replications of each pretreatment for each drying method. The laboratory tray dryer had a radial fan, an electrical resistance heater and a drying chamber. The drying air temperature was adjusted by controlling the heater with a thermostat. The total heater power was 9 kW. The drying chamber was cubical metal box (55 cm x 55 cm x 55 cm). There was an air inlet (diam, 9 cm) on one of the chamber sidewalls near its bottom. The top of the drying chamber was covered by three layers of perforated metal sheet having 5 mm circular holes to have uniform air distribution. The metal sheets were 5 cm away from each other. Sidewalls and the bottom of the drying chamber were insulated by 2.5 cm thick Styrofoam. Black mulberry samples were evenly placed on the top of the drying chamber. The fan sucked fresh air during drying instead of recirculating the air.

The samples were placed on drying trays in both, greenhouse dryer (tunnel plastic dryer: 6 m x 18 m x 3 m) and outdoor for open sun drying. Thickness of mulberries in the bags was kept thin to accelerate the free airflow. Drying trays were equipped with four legs (20 cm high) and covered by a layer of metal screen to let drying air pass through them from bottom to top by natural convection.

The weights of fruits were determined at various time points. The empty weights of sample bags were determined to find the net weight of fruits samples. The weight measurements were made with a digital balance with 0.01 g sensitivity (Sartorius, Type: BA3100P, Germany). Relative humidity and dry bulb temperature of drying air were measured and stored every 15 min by an electronic measurement device (HOBO RH/Temp,

Type: HO8-003-02, USA) for all drying methods. Airflow velocity through mulberry samples in the laboratory tray dryer was measured with a hot-wire anemometer (Testo, Type: 425, Germany). The drying trials were conducted along 7-27 July 2004.

Weight loss percentage of mulberries during drying processes was calculated as

$$WLP = \frac{W_i - W_s}{W_i} \times 100 \quad \dots(1)$$

where *WLP* is weight loss percentage of fruits (%); W_i is the initial weight of fruits and W_s is the weight of fruits at the s^{th} weighing time point during drying process.

One-way, analysis of variance (ANOVA) was performed over the weight loss percentage values of mulberries as the dependent variable to determine the main effects of pretreatment combinations for artificial black mulberry drying (Table 1). Two-way factorial ANOVA was performed over the weight loss percentage values of fruits as the dependent variable to determine the main effects and two-factor interactions of drying method and chemical solution for solar drying and open sun drying¹³. SPSS 10.0 statistical software was used for statistical analysis.

Colors of fresh and dehydrated fruits were quantified using a Minolta (CR-300) Chromameter (Japan). Ten color measurements were done for each treatment. This method gives numeric values of three chromatic scales (L, a, b). L is the brightness ranging from no reflection for black (L = 0) to perfect diffuse reflection for white (L = 100). The value "a" is redness ranging from negative values for green to positive values for red. The value "b" is yellowness ranging from negative values for blue and positive values for yellow. The color at the grid origin (a = 0 and b = 0) is achromatic (gray)¹⁴. Color scales (L, a and b) do not provide an

Table 2 — ANOVA for weight loss percentage (WLP) values of black mulberries dried in greenhouse dryer (solar drying) and under open atmosphere (open sun drying)

Source	Df ^a	F-values			
		18 h	42 h	90 h	186 h
Drying method (DM)	1	228.28**	5.59*	8.94**	15.34**
Chemical solution (CS)	5	1.46 ^{NS}	3.12*	4.78**	0.66 ^{NS}
DM*CS	5	1.82 ^{NS}	3.89*	5.98**	2.72*

**Statistically significance at $\alpha = 0.01$, *Statistically-significance at $\alpha = 0.05$ a while,

^{NS}Statistically non-significance at $\alpha = 0.05$, ^aError 24 and total 36

indication of hue and chroma aspects of color, which are intuitively understood by those in the marketing chain from producer to consumer. Furthermore, it is recommended to use hue angle (h°) and chroma (C) as more practical measures of color. Hue angle is defined as a color wheel, with red-purple at an angle of 0° and 360° , yellow at 90° , bluish-green at 180° and blue at 270° . On the other hand, Chroma (C) represents color saturation, which varies dull at low chroma values to vivid color at high chroma values¹⁴. The hue angle was calculated as follows:

$$h^\circ = \tan^{-1}\left(\frac{b}{a}\right) \quad \dots(2)$$

Arctangent assumes positive values in the first and third and negative values in the second and fourth quadrants. Therefore, its negative values should be readjusted for useful interpretation¹⁴. If its values are in the second quadrant, they should be summed by 180° . If its values are in the fourth quadrant, they should be summed by 360° . The chroma was calculated as follows:

$$C = (a^2 + b^2)^{1/2} \quad \dots(3)$$

Results and Discussion

Average values of a single fresh black mulberry are: length, 26.12 mm; width, 17.8 mm; weight, 4.69 g; volume, 4.67 ml; and moisture content, 80.16 %.

The dry-bulb temperature and relative humidity of air used to dry mulberries in the laboratory tray dryer were $45.2 \pm 0.03^\circ\text{C}$ and $21.87 \pm 0.003\%$, respectively. The velocity of drying air changed between 0.75 and $1.0\text{ m}\cdot\text{s}^{-1}$ during drying trials. The results of ANOVA (Table 1) performed over the weight lost percentage values obtained at various time points of drying at which the samples were weighed (6h, 12h, 24h and 36h). Main effect of chemical solutions was statistically significant ($P < 0.01$) at all weight measurement points of drying. Increase of F-values with elapsed drying time revealed that the effect of pretreatments became more apparent as the drying time progressed. The reason of this increasing effect of pretreatments can be related to the volume and area shrinkages of fruits, which influence the drying rate of fruits by modifying significantly diffusion coefficients as observed for banana drying¹⁵. Some chemical pretreatments in this study are expected to alleviate the effect of shrinkage on diffusion coefficient since chemical pretreatments normally breaks down the waxy cuticular fruit surface and creates microscopic cracks which increase moisture permeability⁶. As a result, this type of pretreatment action is expected to increase F-values with the progress of drying process (Table 2). Fruits dipped in 2% ethyl oleate solution for one minute dried faster than any other mulberry samples did (Fig. 1). Two different concentrations of ethyl oleate solutions (T_1 and T_4) resulted in relatively different weight loss pattern (Fig. 1). The increase of ethyl oleate concentration from 1% to 2% (w/v) decreased the drying time from 34 h to ~22 h to reach 75% weight loss. The untreated samples

lost only 67.86 % at the drying time of 52 h. The samples treated with 1 % NaOH (T_2) lost 75% of their initial weight at the drying time of 44 h. Citric acid slightly improved the weight loss of mulberry samples (T_3) while it eliminated the effect of ethyl oleate solution (T_5).

During solar drying and open sun drying of mulberries, average values of dry bulb temperature and relative humidity of drying air were 32.81 °C and 38.1 % inside greenhouse dryer, 22.75 °C and 60.17 % under open atmosphere, respectively. Hence, greenhouse dryer provided 10.06 °C hotter air than outdoor atmosphere on the average. However, dry bulb temperature of greenhouse dryer air increased up to 59.9 °C while maximum dry bulb temperature of ambient air was 45.89 °C. On the other hand, minimum temperature values were 10.2 and 9.82 °C for greenhouse dryer and ambient air, respectively. Cyclic natures of the properties of both greenhouse dryer air and ambient air prolonged the completions of black mulberry drying process compared to the laboratory tray dryer with constant drying air properties. The two-factor interactions of drying method and chemical solution (DM*CS) were found to be statistically significant ($P < 0.05$) at 42 h, ($P < 0.01$) at 90 h and ($P < 0.05$) at 186 h (Table 2). Therefore, total effects of chemical pretreatments depend on the drying methods by the reason of higher drying air temperatures associated with solar drying of mulberries. Reduction of drying time of white mulberries by chemical pretreatment varied with drying air temperature and the maximum reduction (44.4 %) occurred at 70 °C, which are in agreement with two-way interactions observed in this study. At earlier and later times of drying (18 h and 186 h), two-way interactions were found not to be statistically significant ($P > 0.05$). The main effect of drying method was found to be statistically significant at all weight measurement points (Table 2) but had different statistical significance levels ($P < 0.05$ only at 42 h and $P < 0.01$ at 18 h, 90 h and 186 h) because of daily climatic variations. Solar drying (Fig. 2) of mulberries was superior to the open sun drying (Fig. 3). Activation energy was determined as 51.85 kJ·mol⁻¹ and 50.87 kJ·mol⁻¹ for untreated white mulberries and treated white mulberries with the solution of 2% ethyl oleate and 5% potassium carbonate, respectively⁷. The main effect of chemical solutions was found to be significant ($P < 0.05$) at 42 h and ($P < 0.01$) at 90 h. However, the significant effects of two-way interaction at those points

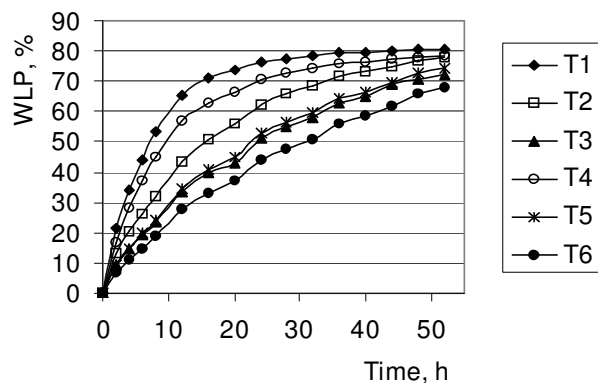


Fig. 1 — Weight loss of black mulberries dried in laboratory tray dryer (artificial drying)

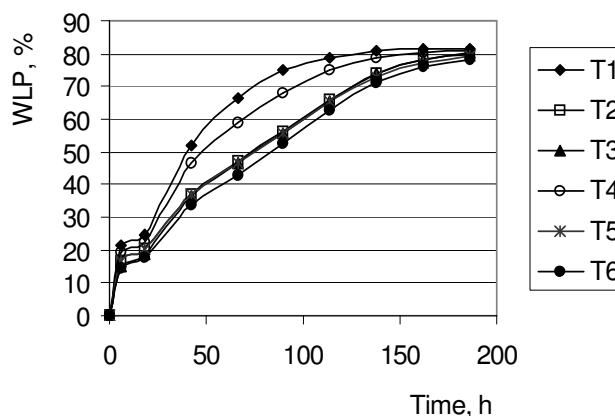


Fig. 2 — Weight loss of black mulberries dried in greenhouse dryer (solar drying)

Fig. 3 — Weight loss of black mulberries dried under open atmosphere (open sun drying)

Table 3 — Measured color values of dried black mulberries

Treatments	Artificial air drying			Solar drying			Open sun drying		
	L	a	b	L	a	b	L	a	b
T ₁	11.33	5.27	2.44	13.07	4.06	2.72	15.14	2.78	1.99
T ₂	12.37	6.89	2.96	14.21	3.17	1.91	14.81	3.34	2.64
T ₃	13.63	5.68	2.28	14.82	3.25	1.73	16.19	3.11	2.07
T ₄	11.81	4.86	2.09	13.03	4.14	2.25	15.37	3.51	2.11
T ₅	12.32	5.69	2.41	13.10	4.27	2.35	15.87	3.73	2.27
T ₆	12.16	6.71	2.67	14.97	6.52	3.11	15.71	2.72	1.65

Table 4 — Calculated color values of dried black mulberries

Treatments	Artificial air drying		Solar drying		Open sun drying	
	Hue	Chroma	Hue	Chroma	Hue	Chroma
T ₁	24.84	5.81	33.82	4.89	35.60	3.42
T ₂	23.25	7.50	31.07	3.70	38.32	4.26
T ₃	21.87	6.12	28.03	3.68	33.65	3.74
T ₄	23.27	5.29	28.52	4.71	31.01	4.10
T ₅	22.95	6.18	28.83	4.87	31.32	4.37
T ₆	21.70	7.22	25.50	7.22	31.24	3.18

unnecessitate the separate consideration of the main effect of chemical solutions.

Overlapped curves (Fig. 3) show that chemical pretreatments did not accelerate weight loss of fruits placed under sun while the solutions of ethyl oleate (T₁ and T₄) showed considerable effect on the weight loss of fruits dried in greenhouse (Fig. 2). Pretreatment solution of 1% NaOH was ineffective for solar drying of fruits contrary to its partial effect observed with the artificial air-drying of fruits (Fig. 1). Fruits treated with 2% ethyl oleate solution and untreated fruits fell to 25 % of their initial weight in 90 h and 157 h in greenhouse dryer, respectively. On the other hand, all mulberry samples under sun fell to 25% of their initial weight in 238 h. As a result, dipping mulberries in 2% ethyl oleate solution is recommended before artificial air drying or solar drying to accelerate drying process. The positive effect of ethyl oleate on the drying of black mulberries can be a result of its solvent effect on cuticular wax¹⁶ or its getting solubilized within plant cuticle¹⁷.

The color values of fresh and ripe fruits are: L, 14.57; a, 9.60; and b, 2.84. These values show the darkish color of ripe fruits. Artificial air-drying of fruits had slightly lower L values than other two drying methods (Table 3). This result indicates that occurrence of non-enzymatic browning reaction (Maillard reaction) depends on drying temperature and oxygen concentration^{18,19}. Ethyl oleate pretreatment did not prevent severe browning of red peppers during solar and open sun drying²⁰. A similar but less intensive outcome was obtained in this study since fruits treated with ethyl oleate (T₁ and T₄) had lowest L values.

Chroma, indicator of color saturation and intensity, with higher values are more desirable. Chroma value and hue angle of fresh black mulberries are 10.01 and 16.48°, respectively. Pretreatments T₂ with artificial drying gave more vivid colored dried black mulberries (Table 4). On the other hand, ethyl oleate pretreatments did not have a positive effect on chroma. However, the chroma values of artificially dried black mulberries did not show very big differences. The increase of drying

time decreased the chroma values as seen in open sun drying and solar drying. In terms of hue angles, pretreatments T₁ and T₂ with open sun drying gave the highest hue angles (35.60° and 38.32°), which normally means more yellowish color. The effect of pretreatments on the hue angle of dried black mulberries is not very noteworthy. On the other hand, sunlight and lengthy drying period brightened the color of black mulberries from darkish color to pale color. Above all, chemical solutions did not create big differences on the color of dried black mulberries.

Conclusions

Effects of pretreatments on drying process of black mulberry at relatively low and moderate drying air temperatures indicated that recommended chemical solution for dipping black mulberries before artificial air drying and solar drying is 2% (w/v) ethyl oleate solution. However, open sun drying of mulberries did shade the positive effect of ethyl oleate solution and prolonged the total drying period (the time to fall to 25 % of initial sample weight) up to 238 h. Total drying periods for artificial air drying and solar drying of black mulberries dipped in 2% ethyl oleate solution for one minute at room temperature (23°C) are 22 h and 90 h, respectively. Pretreatment combination did not cause drastic changes in measured color values of mulberries during all drying trials. Artificial drying decreased brightness values of dried mulberries slightly more than solar and open sun drying.

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