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INVESTIGATION OF THE EFFECT OF DIFFERENT PRETREATMENTS ON DRYING CHARACTERISTICS IN FREEZE DRYING OF SALMON

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Abstract

Atlantic salmon is regarded as a high-quality protein source for human consumption and is high in essential amino acids and Omega-3 fatty acids. In this study, the effect of different pretreatment applications on the drying kinetics in freeze drying of salmon was examined. The samples were treated with ten pretreatments such as blanching, blanching in saltwater, saltwater immersion, and ultrasonication at different application times and salinity rates. Drying rates and effective moisture diffusion (D_{eff}) values of the samples were calculated and the three mathematical models showing the highest agreement among thirteen well-known models were determined. Since the preservation of color values is an important parameter in food processing, color analysis was performed to examine the color change. Drying processes were completed between 240–540 minutes and D_{eff} values were found between 1.71–5.91×10^{-10} m^2/s. The lowest drying time and the highest D_{eff} value were found in the samples with blanching pre-treatment, and it was observed that the D_{eff} value increased as the pre-treatment time increased. The most compatible model for all pretreatments and the control sample was found to be the Midilli & Kucuk model. Additionally, among the pre-treatments applied, saltwater immersion was determined to be the most effective pre-treatment in preserving color values.

Keywords: drying kinetics; freeze drying; seafood; *Salmo salar*; salmon**.**

ДОСЛІДЖЕННЯ ВПЛИВУ РІЗНИХ СПОСОБІВ ПОПЕРЕДНЬОЇ ОБРОБКИ НА ХАРАКТЕРИСТИКИ СУШІННЯ ЛОСОСЯ ЛІОФІЛЬНИМ СУШІННЯМ

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Анотація

Атлантичний лосось вважається високоякісним джерелом білка для споживання людиною і має високий вміст незамінних амінокислот і жирних кислот Омега-3. У цьому дослідженні був вивчений вплив різних способів попередньої обробки на кінетику сушіння лосося під час ліофільного сушіння. Зразки були оброблені десятьма способами попередньої обробки, такими як бланшування, бланшування в солоній воді, занурення в солону воду та ультразвукова обробка з різним часом застосування та рівня солоності. Були розраховані швидкості сушіння і значення ефективної дифузії вологи (D_{eff}) зразків і визначені три математичні моделі, які показали найвищу узгодженість з експериментом серед тринадцяти відомих моделей. Оскільки збереження кольору є важливим параметром переробки харчових продуктів, для дослідження зміни кольору був проведений колориметричний аналіз. Процеси сушіння були завершені в межах 240–540 хвилин, а значення D_{eff} були знайдені в межах 1.71–5.91×10^{-10} м^2/с. Найменший час сушіння й найвище значення D_{eff} було виявлено в зразках з попередньою обробкою бланшуванням, і було помічено, що значення D_{eff} зростало зі збільшенням часу попередньої обробки. Найбільш сумісною моделлю для всіх попередніх обробок і контрольного зразка виявилася модель Midilli & Kucuk. Крім того, серед застосованих попередніх обробок було визначено, що занурення в солону воду є найефективнішою попередньою обробкою для збереження колірних показників.

Ключові слова: кінетика сушіння; сублімаційне сушіння; морепродукти; *Salmo salar*; лосось.

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Introduction

Atlantic salmon (*Salmo salar*) is an anadromous fish species with silver color and distinctive spots, found mainly in the northern waters of North America and Europe. These fish migrate to freshwater rivers to spawn and make breeding migrations to lay their eggs. After the fry grows in freshwater, they begin to feed in the oceans [1-3]. Atlantic salmon play an important role in commercial and sport fishing and are valued as a food source in many cultures. Salmon is especially rich in the omega-3 fatty acids EPA (eicosapentaenoic acid) and DHA (docosahexaenoic acid) [4].

The high demand for seafood and their perishability makes it necessary to apply the techniques required to preserve these foods. Drying salmon is a crucial method for preserving its flesh due to the various benefits it offers. The process of drying salmon helps to extend its shelf life by reducing water activity, which inhibits the growth of spoilage microorganisms and pathogens [5]. Additionally, drying reduces the weight and volume of the salmon, making it easier to store and transport, thus contributing to its preservation. Furthermore, the reduction in water activity through drying also helps to concentrate the flavor of the salmon, enhancing its taste and culinary appeal [6; 7]. In this context, freeze-drying has emerged as one of the most useful methods for preserving food among the various drying procedures. When compared to alternative preservation methods, this method delivers a superior nutritional quality. It is a common dehydration technique based on sublimation that extends the shelf life of food items. Freeze-dried foods have excellent rehydration qualities, high porosity, low color, taste, scent, and nutritional deterioration [8, 9]. Conversely, because freeze drying takes a very long processing time, it requires a lot of energy. As a result of its higher running costs than other drying procedures, it has been regarded as the priciest drying method. Therefore, before freeze-drying, the food needs to go through certain pre-treatment to generate high-quality products at less cost [4; 10].

Some of the most common applications before freeze drying in the literature are blanching, osmotic dehydration (immersion), and ultrasonication [11]. Blanching is a frequently applied method to inactivate enzymes and remove toxic substances. For example, they have shown successful results in freeze drying of fruits and vegetables such as tomatoes [12], red beets [13], onions [14], apples [15]. Osmotic dehydration, or water immersion as it is called in this study, is applied by removing water from the food and increasing a solute content such as salt or sugar. The solute can be selected based on the type of food. For example, in osmotic dehydration studies conducted with sucrose in apples [16, 17] and raspberries [18] and with sucrose in kiwis [19], it was observed that the drying time was shortened and there was an improvement in color and texture properties. Ultrasonication, another pretreatment, is used in many stages of food processing processes [20]. It basically serves to accelerate drying by increasing or widening the pores of the product [21]. It has been applied in the pretreatment of many fruits and vegetables before freeze drying, such as okra [22], apples, carrots, and eggplants [23], sweet potatoes [24]. However, these pretreatments are generally applied to freeze-drying fruits and vegetables. No studies have been conducted on the aforementioned pre-treatments on freeze-drying of seafood.

The main objective of this study was to determine the most effective pretreatment method for freeze-drying Atlantic salmon by evaluating established pretreatment techniques to optimize drying efficiency and maintain product quality. As an innovative aspect of this research, the shortest and most effective drying pretreatment for Atlantic salmon was investigated by applying blanching [25], saltwater blanching, saltwater immersion, and ultrasonic bath treatments. This research aimed to compare and understand how each pretreatment affects drying kinetics, efficiency, and quality results. For this purpose, drying curve data were analyzed using statistical parameters, and mathematical modeling was used to gain insight into the drying behavior of each pretreatment method. Effective moisture diffusion coefficients were calculated for each process, providing baseline data on moisture removal efficiency. Furthermore, to ensure product quality, color changes, an important indicator of appearance and quality in food products, were investigated between treatments. The findings from this study are expected to provide valuable information for optimizing the freeze-drying process of Atlantic salmon and ultimately benefit food processing practices that prioritize quality retention, efficiency, and economic feasibility.

Materials and Methods

Samples and Equipment

Fresh salmon was bought from a fish market in Istanbul, Turkiye, in 2023. The samples were cut at 32×32 mm in length and 8 mm in thickness. Each sample was weighed 10 ± 0.05 g with Radwag AS 220.R2 digital balance (Radwag, Radom, Poland). In order to determine the effect of the pre-treatments, one set of samples was set aside as a control sample without pre-treatment. The other samples were subjected to pre-treatments which are 2 sets of ultrasonication (1 minute and 5 minutes) (US1, US5); 2 sets of blanching (1 minute and 5 minutes) (BW1, BW5) [25], 2 sets of blanching with saltwater (1 minute blanching with 10 % salt and 5 minute blanching with 10 % salt) (BSW10-1, BSW10-5), 4 sets of saltwater immersion (5 minutes with 10 % salt and 20% salt, 10 minutes with 10 % and 20 % salt) (ISW5-10, ISW5-20, ISW10-10, ISW10-20). The moisture contents of each sample were determined using a KH-45 hot air-drying oven (Kenton, Guangzhou, China) at 105 °C. Experiments were repeated three times for each treatment, and the average moisture contents were calculated for each sample. Ultrasonication pretreatment was carried out using an Isolab water bath (Isolab, Germany) with a sensitivity of 1°C and an ultrasound power of 120 W.

Experiments were carried out with a Labart LFD-10N model freeze dryer (ART Laborteknik, Istanbul, Turkiye). The weight of each sample was recorded every 60 min during the experiments. The drying process continued until the moisture content dropped 5 %. After the drying process, color measurements were noted with a color measuring device (PCE-CSM 1; PCE Instruments UK Ltd., Southampton Hampshire, United Kingdom) to determine the effect of drying conditions on the color of the dried material. After the drying process was completed, the samples were packed under vacuum.

Mathematical modeling of drying curves

As the drying process progresses, the moisture content of the sample decreases. During the falling rate period, moisture diffuses from the body to the surface, resulting in the mass transfer of the product's moisture to the surrounding environment through evaporation.

The moisture content, drying rate, and moisture ratio were calculated according to equations that are given in (1), (2), and (3) [26; 27]:

$$M = \frac{m_w}{m_d} \quad (1)$$

$$DR = \frac{M_{t+dt} - M_t}{dt} \quad (2)$$

where M represents moisture content (kg water/kg dry matter), m_w represents water content (kg) and m_d represents dry matter content (kg). DR represents the drying rate (kg water/kg dry matter × min), M_{t+dt} represents moisture content at t+dt (kg water/kg dry matter), t represents the time (min).

$$MR = \frac{M_t - M_e}{M_i - M_e} \quad (3)$$

where MR represents the moisture ratio which is a dimensionless number. M_t represents moisture content at any time, M_e represents equilibrium moisture content and M_i represents initial moisture content, respectively. As M_e is very small, it is neglected in the calculations [28].

Mathematical modeling was performed with 13 different models of Aghbashlo et al., Alibas, Henderson et al., Jena and Das, Lewis, Logarithmic, Midilli and Kucuk, Page, Parabolic, Verma et al., Wang and Singh, Weibull, Two-Term Exponential models with Statistica 8.0 (StatSoft Tulsa, USA) program using the Levenberg-Marquardt algorithm for parameter estimation in nonlinear regression. The best outcomes for the estimate are those with the highest coefficient of determination (R^2), and the lowest reduced chi-square (χ^2) and root mean square error (RMSE) values. R^2, χ^2, and RMSE equations are given in Eqs. (4), (5), and (6), respectively [29]:

$$R^2 \equiv 1 - \frac{\sum_{i=1}^{N}\left(MR_{exp,i} - MR_{pre,i}\right)^2}{\sum_{i=1}^{N}\left(MR_{exp,i} - \left(\frac{1}{n}\right)\sum_{i=1}^{N} MR_{exp,i}\right)} \quad (4)$$

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

$$RMSE = \left(\frac{1}{N}\sum_{i=1}^{N}\left(MR_{pre,i} - MR_{\exp,i}\right)^2\right)^{1/2} \quad (6)$$

where $MR_{exp,i}$ and $MR_{pre,i}$ represent the experimental and predicted MR values respectively. The number of constants in the model is z, and N is the total number of experiments [29].

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Determination of the Effective Moisture Diffusivity

The drying of foods occurs as a function of diffusion and in most cases, diffusion occurs in the falling rate period [30]. There are a few mathematical models that have been used to describe Fick's second law during the falling rate period and it is shown in the following Equation (7) [31]:

$$\frac{\partial M}{\partial t} = \nabla\left[D_{eff}(\nabla M)\right] \qquad (7)$$

Fick's second law of unsteady-state diffusion equation can be used as a way to find the moisture ratio. With an assumption that mass transfer is done by the diffusion only, the diffusion coefficient is constant, and shrinkage is negligible, therefore a drying process in unsteady thin layer diffusion can be given as shown in the following Equation (8) [32]:

$$MR = \frac{8}{\pi^2}\sum_{n=1}^{\infty}\frac{1}{(2n+1)^2}\exp\left(-\frac{(2n+1)^2\pi^2 D_{eff}\times t}{4L^2}\right) \qquad (8)$$

In those equations D_{eff} represents effective moisture diffusivity (m^2/s), t represents time (s), L represents the half-thickness of samples (m) and n represents a positive integer. This complex equation can be simplified for longer drying periods without much affecting the results, thus accuracy and this equation can be given as shown in Equation (9) [33]:

$$MR = \frac{8}{\pi^2}e^{\left(-\frac{\pi^2 D_{eff} t}{4L^2}\right)} \qquad (9)$$

When Equation (9) is rearranged by taking the logarithm, then it can be expressed as a linear function. This linear function is given as shown in Equation (10) [33]:

$$\ln(MR) = \ln\left(\frac{8}{\pi^2}\right) - \left(\pi^2\frac{D_{eff}\times t}{4L^2}\right). \qquad (10)$$

From the slope of the *ln(MR)* versus *t*, D_{eff} can easily be calculated.

Color analysis

Salmon was performed using a colorimeter with the average of values taken from samples from 5 different areas. As a result of the experiment, the L*, a*, and b* values of four randomly taken samples from each salmon were measured and recorded, and average results were obtained. Hunter color analysis is a method that displays the product's lightness value with +L*, redness value with +a*, greenness value with -a*, yellowness value with +b*, and blueness value with -b*. According to these results, how the pretreatment and drying parameters affect the color properties has been interpreted. ΔE (color difference) values were calculated with Eqs. (11) [33; 35].

$$\Delta E = \sqrt{(L_0 - L)^* + (a_0 - a)^* + (b_0 - b)^*}, \qquad (11)$$

where L*, a*, and b* values are the color parameters of the dried samples while L_0*, a_0*, and b_0* values are the color parameters of the fresh (raw) samples.

Results and Discussion

Drying curves

The pre-treatments caused variations in the samples' moisture content and porosity prior to drying, which led to differences in the samples' final moisture values and freeze-drying times. The freeze-drying process was completed in 540 minutes for the control sample, US1 and US5, and the final moisture contents were 2.2646, 2.3317, and 2.3944 kg water/kg dry matter, respectively. Freeze drying times were 300, 300, 240, and 420 minutes for BS1, BS5, BSW10-1, and BSW10-5, respectively, and final moisture contents were 1.6048, 1.2160, 1.3681 and 1.1710 kg water/kg dry matter [25]. Freeze drying times were 540 minutes for all saltwater immersion pretreatments. Final moisture contents were 1.8537, 1.6826, 1.7620 and 1.5904 kg water/kg dry matter for ISW5-10, ISW10-10, ISW5-20, and ISW10-20 pretreatments, respectively. Before and after drying images for all samples are given in Fig. 1.

As shown in the moisture content vs time (Fig. 2) and drying rate vs moisture content (Fig. 3), all blanched samples showed a rapid loss of moisture and dried before all other samples. It is observed that in immersion samples, the drying time does not reduce even while the initial moisture values do. In addition, while the initial moisture values increased in the ultrasonicated samples, no increase in drying time was observed.

Fig. 1. Fresh, pre-treated and dried salmon samples

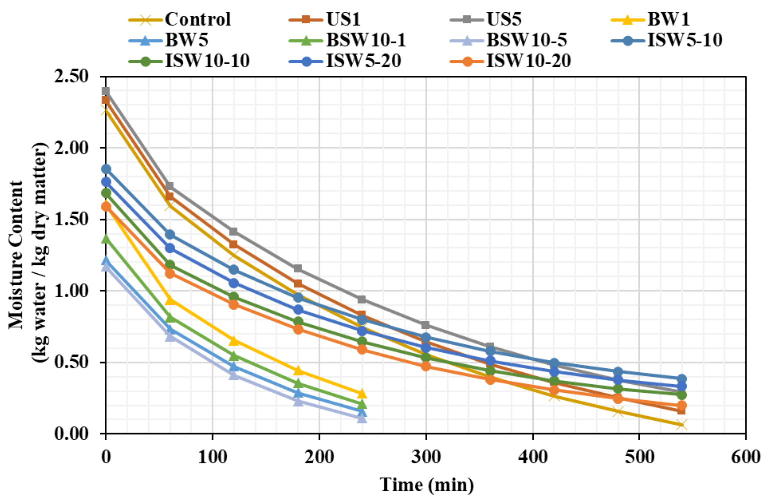

Fig. 2. The graph of moisture content change over time

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The lowest final moisture rates were seen in blanched samples, while the highest values were seen in saltwater immersion samples. Likewise, the final drying rates were obtained just the opposite. As the samples lose water during the immersion process, the salt in the dehydration solution may have transferred into the sample. Since this mutual transport situation cannot be monitored from the weight of the sample, the drying rate may have been much lower than the other samples, while the final moisture content was the highest. In blanching samples, the prediction that moisture loss will be greater than salt uptake in heat-affected samples supports these results.

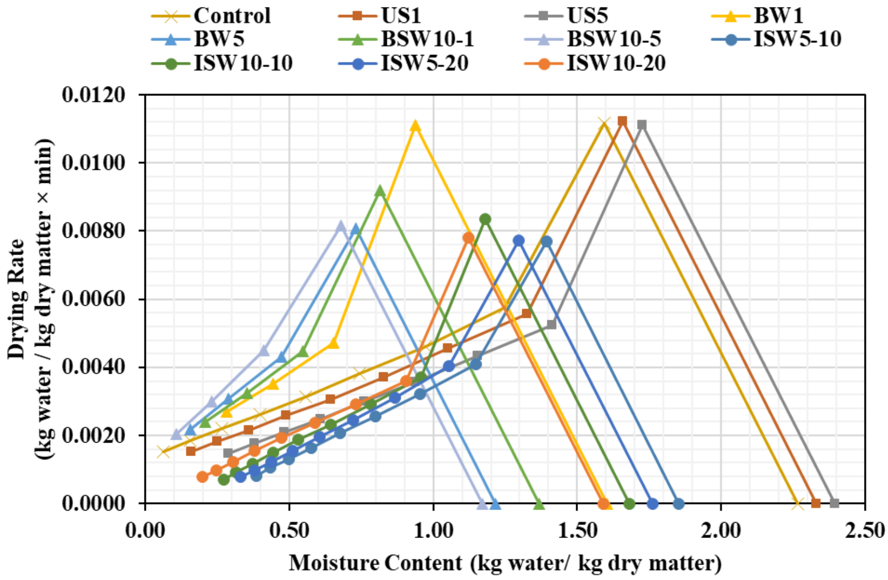

Fig. 3. The graph drying rate variation with moisture contents

Table 1

Moisture content, drying rate, and D_{eff} values of control and pretreated samples

Sample	M (wet basis, %)	M (kg w/kg dm)	DR (kg w/kg dm × min)	$D_{eff} \times 10^{10}$ (m^2/s)
Control	69.37	2.2646	0.0112	3.61
US1	69.98	2.3317	0.0112	2.85
US5	70.54	2.3944	0.0111	2.30
BW1	61.61	1.6048	0.0111	4.27
BW5	54.87	1.2160	0.0081	5.11
BSW10-1	57.77	1.3681	0.0082	4.63
BSW10-5	53.94	1.1710	0.0092	5.91
ISW5-10	64.96	1.8537	0.0077	1.71
ISW10-10	62.72	1.6826	0.0083	1.97
ISW5-20	63.79	1.7620	0.0077	1.82
ISW10-20	61.40	1.5904	0.0078	2.26

Mathematical modeling of drying curves

Model constants and parameters were obtained by fitting the drying data into mathematical model equations. The thirteen models analyzed, the model outputs of the three best-fit models with the highest R^2 and the lowest χ^2 and RMSE values for each method are given in Table 2. According to the evaluated parameters, the highest compatibility with the drying data was found in Midilli & Kucuk with R^2 values greater than 0.999731, followed by Jena & Das with R^2 values greater than 0.994354, and Verma et al. models with R^2 values greater than 0.994080.

Table 2

Mathematical model coefficients and statistical data

Model	Sample	Parameters								
		a	b	c	g	k	n	R^2	χ^2	RMSE
Midilli & Kucuk	Control	0.999137	−0.00028			0.013577	0.077032	0.999853	0.000021	0.003575
	US1	0.999159	−0.00025			0.014360	0.751036	0.999836	0.000022	0.003611
	US5	0.999129	−0.00021			0.015428	0.723941	0.999775	0.000026	0.003982
	BW1	0.999986	−0.00061			0.036423	0.627330	0.999991	0.000004	0.000872

Continuation of the table 2

	BW5	0.999961	–0.00044			0.016024	0.822621	0.999986	0.000007	0.001170
	BSW10-1	0.999975	–0.00051			0.022916	0.735181	0.999989	0.000005	0.000997
	BSW10-5	0.999974	–0.00036			0.013435	0.886202	0.999995	0.000002	0.000701
	ISW5-10	0.999263	0.00002			0.010606	0.800135	0.999882	0.000012	0.002630
	ISW10-10	0.999145	–0.00006			0.017269	0.726614	0.999731	0.000029	0.004138
	ISW5-20	0.999367	0.00001			0.011729	0.791609	0.999907	0.000009	0.002385
	ISW10-20	0.998930	–0.00012			0.015332	0.746941	0.999675	0.000038	0.004774
	Control	0.211538	–0.00256	1.547037		0.004742		0.994354	0.000819	0.022168
	US1	0.205927	–0.00884	1.575103		0.003916		0.996940	0.000406	0.015605
	US5	0.253774	–0.01506	1.367451		0.003026		0.998627	0.000161	0.009834
	BW1	0.174093	–0.02714	1.747486		0.005222		0.999178	0.000345	0.008309
	BW5	0.288649	–0.00285	1.241854		0.007893		0.999053	0.000450	0.009487
Jena & Das	BSW10-1	0.322276	–0.01413	1.131671		0.006563		0.999157	0.000376	0.008672
	BSW10-5	0.108960	0.00598	2.216134		0.009602		0.998950	0.000547	0.010464
	ISW5-10	0.233728	–0.02199	1.455003		0.002047		0.999656	0.000034	0.004491
	ISW10-10	0.185716	–0.02760	1.683067		0.00222		0.999880	0.000013	0.002761
	ISW5-20	0.285652	–0.02349	1.254229		0.002168		0.999736	0.000027	0.004021
	ISW10-20	0.171998	–0.02011	1.758014		0.002896		0.999532	0.000055	0.005726
	Control	0.236920			0.004945	0.004945		0.994080	0.000736	0.022700
	US1	0.932705			0.269449	0.004191		0.997674	0.000264	0.013606
	US5	0.900747			0.308707	0.003537		0.999483	0.000052	0.006033
	BW1	0.865704			0.357618	0.006425		0.999569	0.000091	0.006017
	BW5	0.187052			0.008154	0.008155		0.998990	0.000240	0.009800
Verma et al.	BSW10-1	0.925585			0.29022	0.007175		0.999367	0.000141	0.007515
	BSW10-5	0.252871			0.009057	0.009057		0.998772	0.000320	0.011316
	ISW5-10	0.784141			0.015829	0.002524		0.999603	0.000033	0.004821
	ISW10-10	0.804923			0.028783	0.003061		0.999721	0.000025	0.004212
	ISW5-20	0.779733			0.017013	0.002705		0.999672	0.000029	0.004482
	ISW10-20	0.877243			0.333728	0.003602		0.999992	0.000001	0.000729

Color Analysis Results

Since the main purpose of color analysis is to compare the change in the final product colors of the pre-treatments, fresh sample color values were used instead of the initial values after the pre-treatments in the ΔE calculation. The fresh sample mentioned here is the unpretreated and fresh state of the sample, all other samples were pre-treated, and their color changes were measured after freeze-drying. Measurements were obtained by averaging the values obtained from 5 different regions of the samples with a 5% error value in color measurements. The obtained color values and calculated ΔE values are given in Figure 4.

However, it should be taken into consideration that the initial color distribution and biological composition of each sample processed may not be the same. For this reason, it would not be a consistent conclusion to expect the applied processes to cause the same color changes in all samples.

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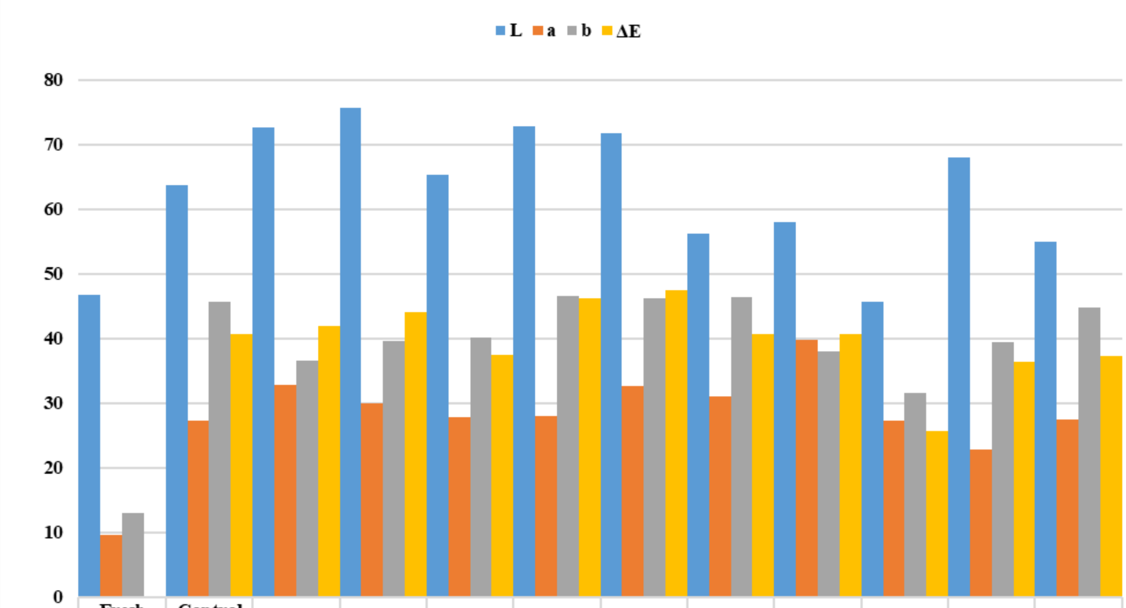

	Fresh salmon	Control sample	US1	US5	BW1	BW5	BSW10-1	BSW10-5	ISW5-10	ISW10-10	ISW5-20	ISW10-20
L	46.79	63.71	72.63	75.68	65.31	72.84	71.72	56.22	57.99	45.62	67.95	54.92
a	9.60	27.28	32.78	29.95	27.87	28.07	32.73	31.13	39.77	27.34	22.81	27.54
b	13.10	45.63	36.55	39.58	40.17	46.54	46.18	46.39	38.07	31.53	39.52	44.76
ΔE	0	40.71	41.90	44.16	37.55	46.24	47.45	40.76	40.74	25.62	36.34	37.29

Fig. 4. Color parameters of fresh salmon and freeze-dried salmon samples

However, when the changes observed in the majority of the samples are evaluated, it was found that all pre-treatments caused an increase in the L*, a*, and b* values, and the most dramatic increase was observed in the positive direction in the L* values. Upon comparing the ΔE values, it can be concluded that the samples immersed in saltwater performed significantly better in terms of color preservation; the ISW10-20 sample displayed the least amount of color change, the blanched samples displayed a significant amount of color change, and the BSW10-1 sample displayed the most amount of color change.

Conclusion

The present study discusses the technique of freeze-drying salmon while maintaining its nutritional content through the use of several pre-treatments. In light of experimental data, the drying kinetics of salmon samples were investigated. Although samples blanched with different pretreatments showed significantly shorter drying times [25], samples immersed in saltwater were found to have a higher success rate in maintaining their visual quality. Furthermore, although the ultrasonication pre-treatment drying periods and visual quality produced outcomes comparable to the control samples, no optimal indicator was obtained. In the study where pretreatment time and salinity ratio were also examined, it was found that increasing both parameters resulted in a decrease in the final moisture content. Besides, the calculated D_{eff} values showed an increase with increasing salinity ratio and pre-treatment time. In addition, the most compatible mathematical models were determined to be Midilli & Kucuk for all samples.

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