

Freeze drying of manila clam: drying kinetics and mathematical modelling

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This study investigates the freeze-drying kinetics and mathematical modeling of Manila clam (*Ruditapes philippinarum*), a highly valued seafood species. While freeze-drying is a preservation method that maintains the physical and chemical integrity of food by removing water through sublimation, its high operational costs due to long processing times necessitate the use of pretreatments. The objective of this research was to examine the impact of various pretreatment parameters, including ultrasonication, blanching, and saltwater immersion, on drying rates and moisture diffusivity to optimize the dehydration process. Samples were subjected to ten different pretreatment conditions: ultrasonication (1 and 5 min), blanching (1 and 5 min), blanching in 10% and 20% saltwater (1 min), and immersion in saltwater (5 and 10 min at 10% and 20% concentrations). The drying data were fitted against 13 mathematical models to determine the most accurate predictive framework for each method. The experimental results revealed that the drying processes were completed within 240 to 480 min, all samples exhibiting a dominant falling rate period. Effective moisture diffusivity (D_{eff}) values were found to range between 3.47 and $6.89 \times 10^{-10} \text{ m}^2/\text{s}$. Notably, blanching emerged as the most effective pretreatment for minimizing drying time and achieving the highest D_{eff} values. In the saltwater immersion groups, D_{eff} increased proportionally with both salt concentration and immersion time. Statistical analysis indicated that Verma and Aghbashlo *et al.* models were the most fitting to all samples.

Keywords: Blanching; lyophilization; modelling; seafood; shell

INTRODUCTION

Drying is simply based on the removal of water from the product and is the most common method used to preserve food products for a long time. While traditional drying methods often struggle to achieve high quality end products, modern methods include techniques developed to improve the quality of the end product and reduce the energy consumed [1]. In the freeze-drying process, water in the product is first frozen and subsequently removed by sublimation under reduced pressure. This low-temperature dehydration method enables long-term storage while preserving the original physical, chemical, and biological properties of the product. Freeze-drying is widely used as an effective preservation technique for high-moisture foods such as fruits, vegetables, and meat products, minimizing quality deterioration and nutrient losses [2]. Although it is an effective method for product preservation, freeze drying is a costly method due to long drying times. Pre-treatments are frequently used to reduce drying times and consequently the energy consumed. Drying pre-treatments are processes carried out before the product enters the dryer to reduce the dryer load, drying time or temperature and to maintain quality parameters [3]. Among these, the most commonly used are ultrasonic and blanching pretreatments. Ultrasonic

pretreatment can be used to change the tissue microstructure of foods to reduce water content or accelerate the drying process. Blanching pretreatment can be called thermal pre-drying. Blanching reduces microorganism load by inactivating common enzymes that negatively affect product quality [4-6].

Seafood, with its high-quality protein content, constitutes a large food category with increasing consumption rates alongside traditional meat products. According to Food and Agriculture Organization (FAO) statistics, approximately 20 million tons of mollusks were consumed as food in 2020 [7]. The Manila clam (*Tapes philippinarum*), a commercially important bivalve species belonging to the family *Veneridae*, is widely distributed in the Adriatic Sea, Mediterranean Sea, Black Sea, North Sea, and Caspian Sea. Due to its high nutritional value and sensory quality, it is widely consumed and considered a valuable seafood product [8].

Freeze-drying is a method widely used for many meat and agricultural products. As the popularity and consumption of seafood increase, the variety of freeze-dried seafood products is also growing. Whilst there are studies on commonly consumed products such as sea cucumber [9], squid [10], shrimps [11, 12], mussels [10, 13], and salmon [14], research into the freeze-drying of Manila clams remains limited. This study aims to examine the

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drying kinetics of freeze-dried Manila clams using various pretreatments. To enhance drying performance, clams underwent ten different pretreatment parameters, including ultrasonication for 1 and 5 min, blanching for 1 and 5 min, blanching in 10% and 20% saltwater for 1 min, 10% and 20% saltwater immersions for 5 min, and 10 min. The collected data were analyzed by comparing them with control samples. Furthermore, the compatibility of the data with 13 mathematical models was tested, and the models exhibiting the highest compatibility were determined.

MATERIALS AND METHODS:

The Manila clams used in this study originated from the Aegean Sea and were purchased frozen from a local market in Istanbul, Türkiye, in May 2022. They were stored in a Flavel FLV 1090 (Flavel, Türkiye) deep freezer at -20 °C until the start of the experiments. Clams were cleaned by removing the shells and weighed at an average weight of 7.0 ± 0.5 g using a Radwag AS 220.R2 digital balance with an accuracy of 0.0001 g (Radwag, Radom, Poland) at each drying step. Then, the moisture content determination before drying treatments was carried out at 105 °C for 3 h using a KH-45 hot air-drying oven (Kenton, Guangzhou, China). Ultrasonic pretreatment was completed with the Isolab water bath with 1 °C sensitivity and 120 W ultrasonic power (Isolab, Germany). Freeze drying was carried out in a Labart LFD-10N model freeze dryer (ART Labortechnik, Istanbul, Türkiye).

Drying experiments

Frozen clams were weighed at 7.0 ± 0.5 g for 11 sets of experiments. Experimental sets included control, ultrasonication for 1 min (1 min US) and 5 min (5 min US); blanching for 1 min (1 min BWB) and 5 min (5 min BWB); blanching for 1 min with 10% saltwater (1 min BWB w/10%) and 1 min with 20% saltwater (1 min BWB w/20%); 5 min immersion in 10% saltwater (5 min SI w/10%) and 20% saltwater (5 min SI w/20%); 10 min immersion in 10% saltwater (10 min SI w/10%) and 20% saltwater (10 min SI w/20%). During freeze drying, the vacuum of the dryer was turned off every 60 minutes and the samples were weighed in less than 2 minutes and the results were recorded. Then, the products were placed back in the dryer and the vacuum was turned on. Samples were dried to 5% moisture content and packed under vacuum.

Mathematical modelling

Moisture content (M) and moisture ratio (MR) were calculated using equations (1, 2) [15]:

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

where M, m_w , and m_d are the moisture content (kg water / kg dry matter), water content (g) and dry matter content (g), respectively.

$$MR = \frac{M_t - M_e}{M_i - M_e} \quad (2)$$

where M_t is the moisture content at a specific time, M_e is the moisture content at equilibrium, and M_i is the moisture content of the material initially. Mathematical models were tested for compatibility with clam MR and time data. The 13 published mathematical models examined for compatibility are listed in Table 1. In these models, a, b, and c are drying exponent coefficients defined for each equation separately; k, k_1 , and k_2 are drying coefficients defined for each equation separately, and t stands for time in min.

Table 1. Drying models and equations

Model	Equation	Ref.
Aghbashlo <i>et al.</i>	$MR = \exp(-k_1 t / (1 + k_2 t))$	[16]
Alibas	$MR = a \cdot \exp((-kt^n) + bt) + g$	[17]
Henderson & Pabis	$MR = a \cdot \exp(-kt)$	[18]
Jena & Das	$MR = a \cdot \exp(-kt + b\sqrt{t}) + c$	[19]
Lewis	$MR = \exp(-kt)$	[20]
Logarithmic	$MR = a \cdot \exp(-kt) + c$	[21]
Midilli <i>et al.</i>	$MR = a \cdot \exp(-kt^n) + bt$	[22]
Page	$MR = \exp(-kt^n)$	[23]
Parabolic	$MR = a + bt + c t^2$	[24]
Verma <i>et al.</i>	$MR = a \cdot \exp(-kt) + (1 - a) \cdot \exp(-gt)$	[25]
Wang & Singh	$MR = \exp(-(t/b)^a)$	[26]
Modified page	$MR = a \cdot \exp(-(kt^n))$	[27]
Two-term	$MR = a \cdot \exp(k_0 t) + b \cdot \exp(k_1 t)$	[28]

To find the best suitable model coefficient of determination (R^2), root-mean-square error (RMSE), and reduced chi-squared (χ^2) statistic are used. Required parameters for given models are estimated using the non-linear regression method with Statistica (Statistica, 2016). Equations for these methods are given below in equations (3-5) [29]:

$$R^2 = 1 - \frac{\sum_{i=1}^n (MR_{exp,i} - MR_{pre,i})^2}{\sum_{i=1}^n (MR_{exp,i} - (\frac{1}{n} \sum_{i=1}^n MR_{exp,i}))^2} \quad (3)$$

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

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

where MR_{exp} is the experimental moisture ratio values, MR_{pre} is the predicted moisture ratio values, n is the total number of the experiments, and z is the number of the constants in the given model. In the

selection of best fitted model, R^2 values are expected to be closer to 1. On the other hand, RMSE and χ^2 values are expected to be closer to 0 [29].

Effective moisture diffusivity determination

Moisture removal during food drying is generally governed by diffusion mechanisms, particularly during the falling-rate period. Several mathematical approaches have been applied to express Fick's second law for this stage of drying, as presented in equation (6) [29, 30];

$$\frac{\partial M}{\partial t} = \nabla[D_{eff}(\nabla M)] \quad (6)$$

Fick's second law for unsteady-state diffusion is commonly used to determine the moisture ratio. By assuming that moisture migration occurs only through diffusion, the effective diffusion coefficient remains constant, and volume shrinkage is negligible, the analytical solution for unsteady diffusion can be expressed as equation (7):

$$MR = \frac{8}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{(2n+1)^2} e^{-\frac{(2n+1)^2 \pi^2 D_{eff} t}{4L^2}} \quad (7)$$

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 (8):

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

When equation (8) is rearranged by taking the logarithm, then it can be expressed as a linear function. This linear function is given as shown in equation (9):

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

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

RESULTS AND DISCUSSION

The initial moisture content of the clam samples varied noticeably with the applied pre-treatment, indicating that these processes already influence water distribution before freeze-drying. As shown in Table 2, which lists the moisture content and drying times for all samples, the control sample exhibited an initial moisture content of 84.42% (5.4180 kg water/kg dry matter). Among the treatments, US application slightly increased the initial moisture content, reaching 85.53% for 1 min US and 86.27% for 5 min US. This increase may be attributed to

structural disruption caused by cavitation, which enhances water retention within the tissue matrix.

In contrast, BWB significantly reduced the initial moisture content. The values decreased to 75.49% (3.0794 kg/kg dry matter) for 1 min BWB and further to 69.92% (2.3242 kg/kg dry matter) for 5 min BWB. This reduction is likely due to protein denaturation and cell structure collapse, facilitating water loss during the pretreatment stage. When salt was incorporated into blanching (10% and 20%), the initial moisture content remained lower than the control but slightly higher than plain BWB, suggesting that osmotic effects partially limited water loss. SI treatments showed intermediate behavior. Initial moisture contents ranged from 76.69% to 82.94%, depending on salt concentration and immersion time. Higher salt concentrations and longer immersion times generally resulted in lower moisture contents, indicating osmotic dehydration effects.

Table 2. Moisture content and drying time of the clam samples

Sample	Initial moisture		Drying time min	Final moisture kg water/ kg dry matter
	% (wet basis)	kg water/ kg dry matter		
Control	84.42	5.4180	420	0.2926
1 min US	85.53	5.9114	480	0.2816
5 min US	86.27	6.2821	480	0.4155
1 min BWB	75.49	3.0794	300	0.1291
5 min BWB	69.92	2.3242	240	0.1526
1 min BWB w/10%	74.53	2.9264	240	0.2134
1 min BWB w/20%	72.93	2.6943	240	0.1936
5 min SI w/10%	82.94	4.8625	480	0.2253
5 min SI w/20%	80.91	4.2375	300	0.2108
10 min SI w/10%	82.30	4.6492	360	0.1604
10 min SI w/20%	76.69	3.2895	300	0.1603

Regarding drying time, pre-treatments had a pronounced impact on the efficiency of the freeze-drying process. The control sample required 420 minutes to reach the target moisture level. Ultrasonic treatments resulted in the longest drying times (480 min for both 1 min and 5 min US), despite their relatively high initial moisture contents. This suggests that although ultrasonication disrupts tissue

structure, it may also lead to water being more strongly retained within the matrix, thereby slowing down sublimation during freeze-drying. In contrast, blanching treatments significantly reduced drying time. The 1 min BWB sample required 300 minutes, while 5 min BWB further decreased the drying time to 240 minutes, representing the shortest duration among all samples. This improvement can be attributed to thermal damage to cellular structures, which enhances moisture diffusivity and facilitates ice sublimation. Blanching combined with salt (BWB with 10% and 20%) maintained similarly short drying times (240 min), indicating that the addition of salt does not hinder the drying rate and may even support moisture removal through osmotic effects prior to freeze-drying.

SI treatments showed variable effects on drying time depending on process conditions. While 5 min SI with 10% salt resulted in a prolonged drying time (480 min), increasing salt concentration or immersion time reduced the drying duration (300–360 minutes). In particular, 10 min SI treatments demonstrated improved drying efficiency compared to shorter immersion, likely due to enhanced osmotic dehydration and partial removal of free water before drying. All these changes in moisture content observed during drying are also displayed in the graphs showing the change in moisture content over time (Figure 1) and the change in drying rate as a function of moisture content (Figure 2).

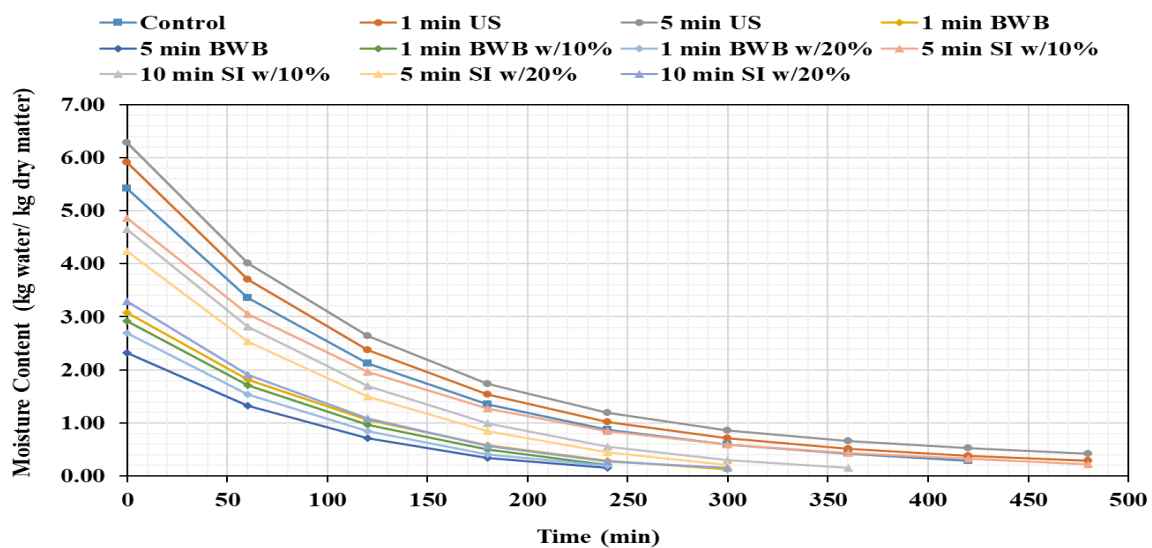


Figure 1. Change in moisture content with time

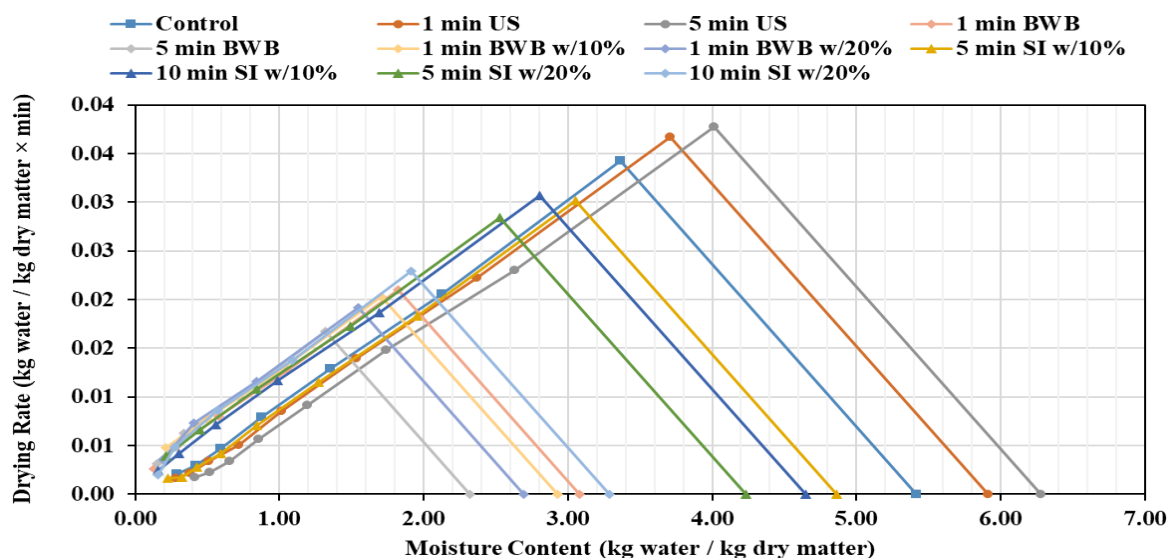


Figure 2. Change in drying rate with moisture content

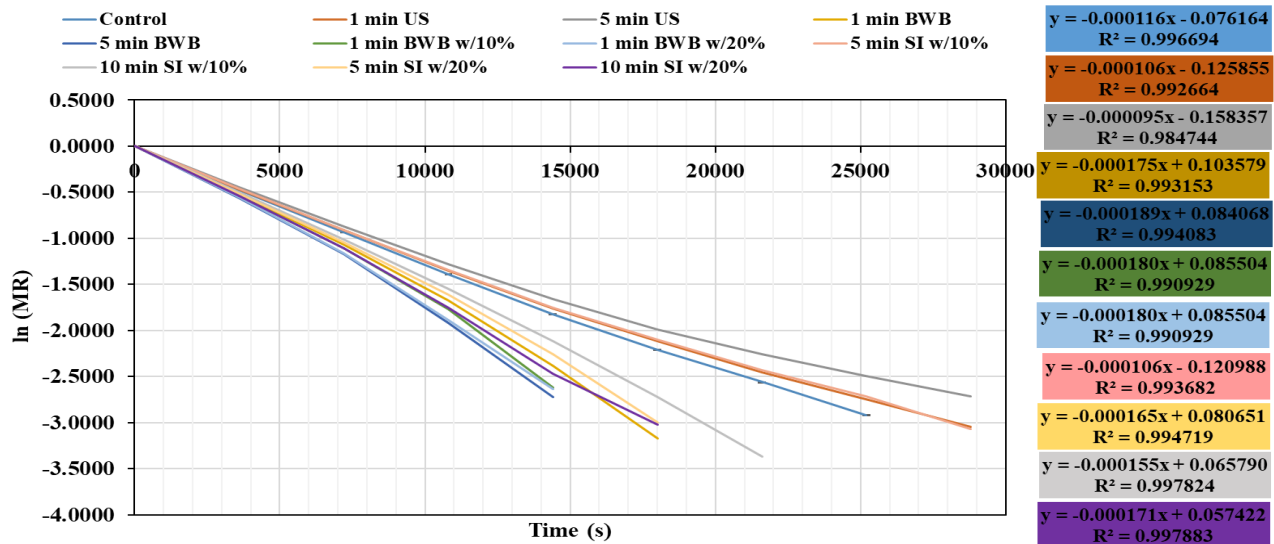


Figure 3. $\ln(MR)$ change over drying time

Regarding the final moisture content after freeze-drying, all pretreated samples exhibited lower or comparable values relative to the control (0.2926 kg water/kg dry matter), except for the 5 min US sample, which showed a higher final moisture (0.4155 kg/kg dry matter). This suggests that prolonged ultrasonication may hinder effective moisture removal, possibly due to structural compaction or reabsorption effects. Blanching treatments resulted in the lowest final moisture contents, particularly 1 min BWB (0.1291 kg/kg dry matter), indicating enhanced drying efficiency. Similarly, salt-assisted treatments, especially longer immersion times (e.g., 10 min SI), also achieved low final moisture levels (~ 0.1603 – 0.1604 kg/kg dry matter), demonstrating the effectiveness of osmotic dehydration in facilitating freeze-drying.

Effective moisture diffusivities

The graph of $\ln(MR)$ versus drying time, plotted using MR data calculated based on Fick's second law, is shown in Figure 3. The D_{eff} values of the samples were calculated from the slope of this graph, in accordance with equation 9. D_{eff} values clearly demonstrate the influence of pre-treatments on mass transfer during freeze-drying. The control sample exhibited the lowest D_{eff} value ($3.47 \times 10^{-10} \text{ m}^2/\text{s}$), indicating limited internal moisture movement in untreated clam tissues. All pre-treatment methods increased D_{eff} to varying extents, confirming their role in enhancing moisture transport. US treatment resulted in a moderate increase in D_{eff} , reaching $3.87 \times 10^{-10} \text{ m}^2/\text{s}$ for 1 min and $4.23 \times 10^{-10} \text{ m}^2/\text{s}$ for 5 min. This improvement can be attributed to cavitation-induced microchannel formation, which facilitates moisture diffusion. However, the relatively small increase compared to other treatments suggests that ultrasound alone provides limited structural

modification. BWB showed the most significant enhancement in effective diffusivity. D_{eff} values increased to $6.38 \times 10^{-10} \text{ m}^2/\text{s}$ (1 min) and $6.89 \times 10^{-10} \text{ m}^2/\text{s}$ (5 min), representing a nearly twofold increase compared to the control. This substantial improvement is likely due to protein denaturation and cell membrane disruption, which reduce internal resistance to moisture movement. The addition of salt during blanching (10% and 20%) maintained similarly high D_{eff} values (6.57×10^{-10} and $6.68 \times 10^{-10} \text{ m}^2/\text{s}$), indicating that osmotic effects complement thermal damage and further promote water mobility. SI treatments exhibited time and concentration-dependent behavior. While 5 min SI with 10% salt showed only a slight increase ($3.87 \times 10^{-10} \text{ m}^2/\text{s}$), extending the immersion time to 10 min significantly improved D_{eff} ($5.65 \times 10^{-10} \text{ m}^2/\text{s}$). Similarly, higher salt concentration (20%) resulted in greater diffusivity values (6.02×10^{-10} and $6.24 \times 10^{-10} \text{ m}^2/\text{s}$ for 5 and 10 min, respectively). These results indicate that osmotic dehydration plays a key role in enhancing moisture diffusion, particularly at higher concentrations and longer treatment times.

Mathematical modeling results

Drying values obtained during freeze drying for all samples were modeled according to commonly used drying mathematical models. Table 3 presents the coefficients and constants for the four models—Aghbashlo *et al.*, Page, Verma, and Two-term—which demonstrated the highest fit for all samples. Upon examination of the table, it is observed that the Verma and Aghbashlo *et al.* model exhibits the highest fit among all models, respectively.

In previous studies involving shrimp [11] and squid [30] freeze-dried following similar pre-treatments, the Verma and Aghbashlo *et al.* models were also found to be among the models showing

high fit. This suggests that the Verma and Aghbashlo *et al.* models can be used to provide consistent predictions for the freeze-drying of seafood following ultrasonication, blanching, or saltwater immersion pre-treatments.

CONCLUSION

This study focused on investigating the effects of different pre-treatments on the freeze-drying kinetics of Manila clams and evaluated the suitability of several mathematical models for describing the drying behavior. The results showed that pre-treatment application significantly influenced the initial moisture content, drying time, final moisture content, and effective moisture diffusivity of the clam samples. Compared with the untreated control, blanching-based treatments were the most effective in improving drying performance. In particular, 5 min blanching resulted in the shortest drying time, while blanching with saltwater also maintained high drying efficiency. These improvements were associated with structural changes caused by thermal treatment, which reduced internal resistance to moisture movement and facilitated sublimation

during freeze-drying. Ultrasonic pre-treatment increased the effective moisture diffusivity to some extent; however, it also resulted in longer drying times compared with the control. This indicated that although ultrasonication may modify tissue structure through cavitation effects. Saltwater immersion showed concentration- and time-dependent effects. Longer immersion times and higher salt concentrations generally improved drying efficiency, suggesting that osmotic dehydration contributed to water removal before freeze-drying and enhanced moisture diffusion. The control sample exhibited the lowest diffusivity value, whereas blanching and salt-assisted treatments produced considerably higher values. Among the mathematical models tested, Verma and Aghbashlo *et al.* models provided the best fit for the experimental drying data. Overall, the findings demonstrated that appropriate pre-treatment selection can substantially improve the freeze-drying efficiency of Manila clams by reducing drying time, lowering final moisture content, and enhancing effective moisture diffusivity.

Table 2. Mathematical model parameters and coefficients

Model	Parameter	Control	1 min US	5 min US	1 min BWB	5 min BWB	1 min BWB w/10%	1 min BWB w/20%	5 min SI w/10%	5 min SI w/20%	10 min SI w/10%	10 min SI w/20%
Aghbashlo <i>et al.</i>	k_1	0.008181	0.008119	0.007940	0.008283	0.008808	0.008402	0.008746	0.008099	0.008158	0.008207	0.008663
	k_2	0.000379	0.000520	0.000724	-0.00066	-0.000926	-0.000862	-0.000866	0.000530	-0.000316	-0.000520	-0.000574
	R^2	0.999955	0.999907	0.999732	0.999907	0.999933	0.999834	0.999941	0.999913	0.999967	0.999911	0.999887
	χ^2	0.000006	0.000011	0.000030	0.000015	0.000013	0.000031	0.000011	0.000010	0.000005	0.000014	0.000019
	RMSE	0.002061	0.002909	0.004857	0.003206	0.002772	0.004291	0.002578	0.002809	0.001854	0.003097	0.003538
Page	k	0.010122	0.010984	0.012051								
	n	0.944712	0.922128	0.892688								
	R^2	0.999820	0.999573	0.999005								
	χ^2	0.000023	0.000050	0.000113								
	RMSE	0.004135	0.006245	0.009364								
Verma	a	0.061517	0.081900	0.062816	-0.35365	-1.141420	-0.040304	-1.230980	0.112937	-0.113017	-0.048372	-1.870660
	k	0.001935	0.001922	0.000591	0.003871	0.004990	-0.002064	0.005100	0.002484	0.003345	0.000340	0.005980
	g	0.008409	0.008404	0.008008	0.007306	0.006910	0.008392	0.006860	0.008569	0.007781	0.008080	0.006990
	R^2	0.999989	0.999989	0.999981	0.999975	0.999927	0.999997	0.999898	0.999975	0.999990	0.999992	0.999815
	χ^2	0.000002	0.000002	0.000003	0.000006	0.000021	0.000001	0.000029	0.000003	0.000002	0.000002	0.000041
	RMSE	0.001006	0.001003	0.001311	0.001668	0.002897	0.000614	0.003386	0.001506	0.001034	0.000919	0.004528
Two-term	a				-136.369	-43.91150	-11.88150	-21.37190	0.113942	225.02400	-202.5990	-31.70450
	k_0				0.013000	0.015100	0.005500	0.006000	0.002499	0.011000	0.012000	0.006500
	b				137.3680	44.91080	12.88070	22.37240	0.886244	-224.0240	203.5980	32.70540
	k_1				0.013000	0.015000	0.005800	0.006100	0.008577	0.011000	0.012000	0.006600
	R^2				0.999713	0.999746	0.999965	0.999902	0.999975	0.999925	0.999788	0.999818
	χ^2				0.000095	0.000145	0.000020	0.000056	0.000004	0.000018	0.000069	0.000061
	RMSE				0.005634	0.005391	0.001977	0.003335	0.001505	0.002804	0.004795	0.004494

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