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Research Paper

Study the Characteristics of Kinetic Model of Drying Freeze-Dried Rosehip (*Rosa canina*)

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Abstract: This study has been carried out with the "Freeze Drying" topic and freeze-drying technology (FD), which proved to be a healthy drying model that increases the shelf life of the products and preserves their aroma values. The product that we have dried is Rosehip, which is utilized for the cooking jam in winter, brew tea, which contains high nutritional values and countless merits for human health. We started the study with the experiment of drying the rose hips, which were cut into 5 mm slices and removed from the seeds, by keeping them in the refrigerator. By recording the experiments of the rose hips, which were placed in our drying device in 7 samples of 100 grams, one by one. The weights of the 2, 4, 6, 8-, 10-, 12- and 14-hour experiments were measured, and a kinetic model was created also calculate the moisture ratio (MR). A total of 8 different kinetic drying models were studied by using the MATLAB program in the presence of experimental data, and according to the results of the experiment, X^2: 2,156×10^{-4}, RMSE: 0.011874 value was regulated. It has been observed that the closest value of R^2 to1 is R^2 with the amount of R^2 =0.9985. According to the results of the experiment, it was determined as the Diffusion Approach model that gave the best results among eight models. Also, we have found the effective diffusivity value, which was 1, 99828×10^{-10} m^2/s for the mentioned Rosehip slices. It was confirmed that the calculated effective diffusivity value was within the reference range mentioned in the literature (10-12 – 10-8 m^2/s) for food products.

Keywords: *Freeze Drying, Diffusion Approach Model, and Rosehip.*

Introduction

The drying of heat-sensitive materials such as vegetables and fruits will lead to deterioration due to thermal decomposition and enzymaticization (Moraga *et al*. 2011). Methods such as freeze-drying are much needed to prevent deformation and deterioration and are also used to increase the shelf life. The main quality characteristics welcomed by the consumer are colour and texture (Nowak & Jakubczyk. 2020; Barbosa-Cánovas *et al*. 2005; Karam et al. 2016). During freeze-drying, the structural properties of freeze-dried products may change or completely disappear and depend on the water content of the food. One of the methods used to improve the quality and shelf life of foods is freeze-drying under a vacuum. As a definition, freeze-drying is a low-temperature dehydration process that is based on the removal of water from food by depressurization, freezing it, and even sublimation process. During the process, low pressure and low temperature conducted quality products. By utilizing the mentioned technique, the originality of the product would be preserved additional the quality of that would remain very high (Martínez-Navarrete *et al*. 2019).

Sublimation definition is the transition of the body from a solid form to the gas form; The change of state without becoming a liquid, that is, without melting and changing from solid to a liquid phase. The logic of freeze-drying is shown in figure 1. In the process of direct transition from solid-state to gas state with the vacuum at low pressure and temperature, the liquids in the foods are removed and their solid-state remains to preserve them, to keep remain and fix their biological state (Strommen *et al*. 2004; Xiang *et al*. 2004)

The freeze-drying method is the process of reducing the liquid inside of foods to extend their shelf life and make them easier to package (Shukla 2011; Wolff et al. 1990). Freeze design

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technology is actively used in areas such as figs, coffee, vegetables, serums, pharmaceutical industry, fruit and fruit juices, as well as meat and milk drying (Wang *et al*. 2007). Freeze drying is a costly and strenuous process. Some of the abundant advantages of the freeze-drying technique are such as providing a higher quality product, extending the shelf life of products, reducing their weight, and reducing their volume (Jiang *et al*. 2010). In the specific research, Dr.Kirmaci and his friends calculated the moisture rates of strawberries cut in 5 mm and 7 mm thickness by freeze-drying and taking the weight losses during the process and finally found the RMSE error rate at the end of the experiment (Menlik *et al*. 2009).

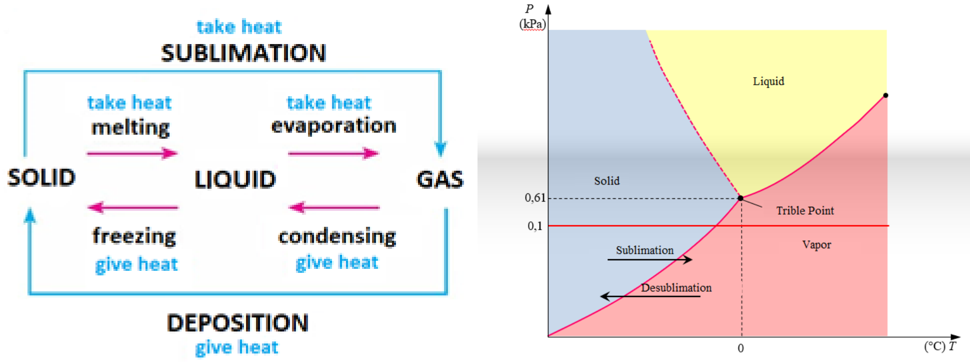

Figure 1. The logic of freeze-drying

Dr.Acar in specific studies and works concluded that freeze-drying is more effective in preserving the nutritional values of the saffron plant in the experimental results by drying the saffron plant with normal and freeze-drying (milling drying) methods (Acar *et al*. 2015). As stated in the introduction, we have decided to freeze-dry the rosehip fruit in our study. It is known that rosehip fruit, which is estimated to have spread all over the world originally belongs to Turkey, or in other statement Turkey is its homeland, generally grown in the Black Sea and Marmara regions of our country. Rosehip which is collected in September and October is consumed as dried or fresh (Koyuncu et al. 2003; Erenturk *et al*. 2005; Erenturk *et al*. 2004)

Material and Method

First of all, we created 7 experimental groups from 100 grams. We chopped the rose hips into slices, put them in our containers with the seeds removed, and waited for them to freeze for 48 hours by placing them in the freezer 2 days before the experiment. In this way, it was ensured that the products were ready for experimentation.

Figure 2. Rosehip fruit

The drying device we used in our experiment is the COOLSAFE type device of the Labogene brand, as seen in Figure 2. The evaporator temperature can drop down to -55 °C and the freezing process of the products took place inside the device. The experiment started with the linked equipment to the vacuum pump having technical details of 4×10^{-4} mbar as power and the pressure of the gadget was decreased to 1×10^{-2} kPa and the internal temperature was minimized to -40 °C. The test device is as in the below picture. Necessary adjustments were made by entering the duration of the operation, appropriate pressure values, temperature, and operating values on the screen of the device. The logic of freeze-drying is based on sublimation. When the product is frozen, it freezes in the moisture inside. If the test head is kept below the critical pressure value and the temperature increase is created, the moisture passes directly to the gas phase and is separated from the product moisture.

The operation of the device in Figure 3 is based on the principle of increasing the temperature of a frozen product in a low-pressure environment and performing sublimation. In our study, the vacuum pump function brings the pressure of the drying chamber to the desired pressure to obtain the desired physical properties (temperature, pressure), while the compressor of the device adjusts the temperature suitable for the in-cabin drying processes. To keep the temperature and pressure under the conditions of our study, after the sample was placed in the drying enclosure of the device, the temperature and pressure control panel was adjusted, and the device was operated, and our experiment was carried out. While the compressor in Figure 3 adjusts the temperature in the cabin, the vacuum pump reduces the ambient pressure. Thus, the necessary environment for sublimation is provided. In this study, the product was placed in the drying chamber and then the temperature and pressure settings were made from the control panel of the device, and the device is ready to operate. The freeze-drying time of the samples was set to 14 hours. The time and temperature chart are arranged as shown in Figure 4. According to the planned system, the Rosehip, which was taken out of the deep freezer at -15 °C and is placed in the device for the first 60 minutes. It was adjusted at -40 °C and 0.01 kPa pressure after by keeping the pressure constant for 180 minutes at -30 °C, subsequently 180 minutes at -20 °C, later120 minutes at -10 °C, 120 minutes at 0 °C, respectively, 120 min at 5 °C and finally at 10 °C for 60 min. After processing, the freeze-drying process is carried out at the end of a total of 14 hours.

Figure 3. Schematic view of the freeze-drying device (Dagdeviren et al. 2021)

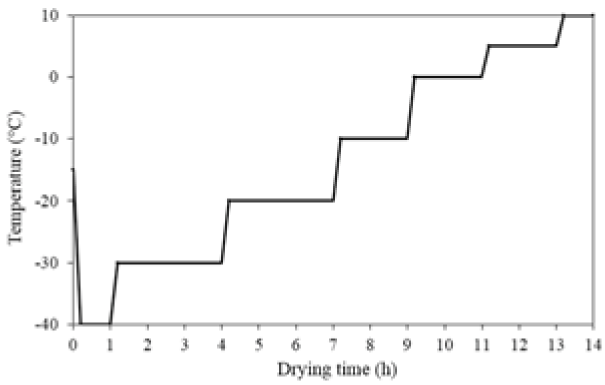

Figure 4. Graph of temperature and time

In the study, 7 different samples were prepared to measure weight losses every two hours. To carry out this measurement, the first sample was placed in the device and the device started to work, after 2 hours the sample was removed. Consequently, the weight loss of the sample was measured by a precision scale and weighing machine. The mentioned scale was sensitive up to 0.001 g. Then the second sample had taken into the device and the device operated due to the same drying settings.

Then, the sample is taken from the device at the end of 4 hours, and the weight loss was calculated. This process was applied as 6, 8, 10, 12, and last 14 hours for each sample. Then the samples were placed in the oven and waited for about 60 minutes. The sample had taken from the oven placed in a desiccator made of curved glass with plenty of silica gel and kept for about 15 minutes. At the end of 15min, the sample taken from the desiccator and weighed on a precision balance and weighing machine finally the result was recorded. By performing the previous process for other date samples, the sample is taken to the device at the end of the 6th, 8th, 10th, 12th, and last 14 hours, and the loss of the mass is determined. In Figure 5, the mass loss of the Kanlıca mushroom sample according to the drying time is shown. Then it is placed in the oven and approximately 60 minutes and kept waiting. The purpose of this process is to remove as much moisture as possible from the product to calculate the moisture content of the product more accurately. As a result of the moisture content determination made with an oven and desiccator, it was determined that 100 g product contains 65,979 g moisture, and 34,021 g as dry part of the product. After calculations, the desired moisture of the dry part of the product is 34.021 g. In Figure 5, the weight loss curve of Rosehip fruit samples had taken every two hours because freeze-drying was demonstrated.

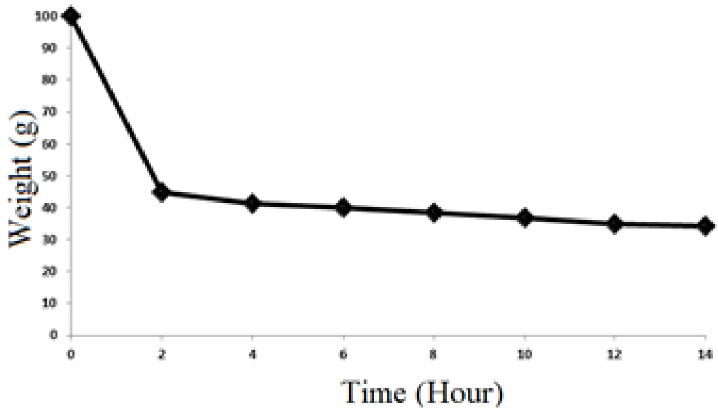

Figure 5. Graph of weight loss over time

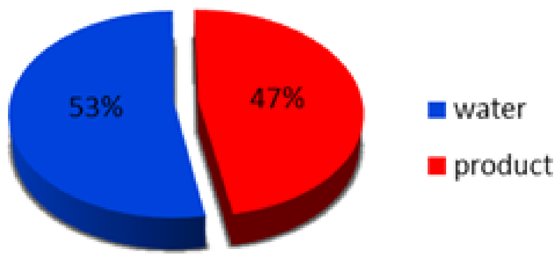

Figure 6. Weight loss of rosehip sample over time

Theoretical models can be applied to all many of substances and conditions. However, the fact is that the equations required for its solution consists of many parameters and complex structures, which will reduce the purpose of such models. Although semi-theoretical models are less complex, their usage was limited by the fact that the parameters they contain are relevant to the products under consideration. There are no complex mathematical equations in determining the drying rate based on experimental data. However, the obtained equations are valid for the sample and test conditions. The equation, which has the most common use among semi-theoretical models, is known as the "logarithmic drying" equation (Sacilik *et al*. 2006; Rayaguru et al. 2011). The variation of moisture ratio (MR) with time (t), which is a dimensionless term, can be determined by the equation given in Equation 1.

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

In the equation (M_0) is the initial moisture content, (M_t) is the moisture content at a time t, and (M_d) is the final equilibrium moisture content. The proportion on the left side of the equation gives the moisture ratio (MR) values at different t moments of drying (Acar et al. 2021).

Results and Discussion

Figure 7 exhibits the experimental moisture content graph obtained after 14 hours of freeze-drying of the rosehip sample.

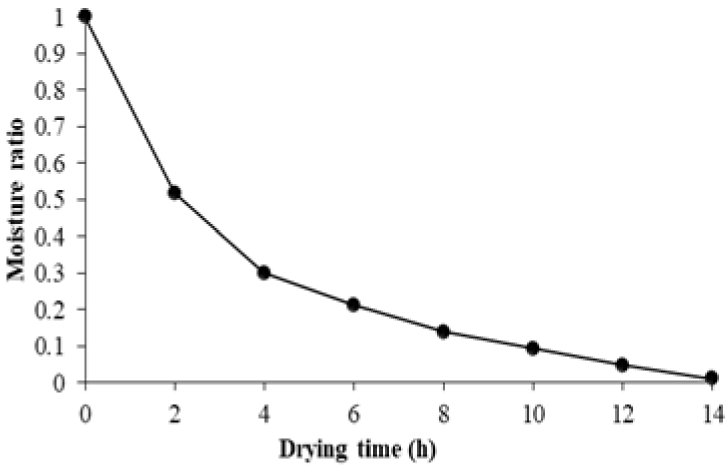

Figure 7. Moisture rate of rosehip sample over time

After determining the moisture content of the products and recording the time-related weight losses, the graph based on the mathematical models was created and the most suitable one among the 8 different drying kinetic models was determined then applied. MATLAB program and software was utilized to perform the introduced operations. In table1 a total of 8 different drying kinetic models with determined estimated moisture content (MR) that employed in the MATLAB software had indicated (Zarein *et al*. 2013; Akpınar *et al*. 2003; Ayrıksa *et al*. 2021)

Table 1. Practical and semiempirical equations for drying kinetics

Model no	Model name	Model
1	Newton	$MR = \exp\ (-kt)$
2	Page	$MR = \exp\ (-kt^n)$
3	Modified Page I	$MR = \exp\ [-(kt)^n]$
4	Henderson and Pabis	$MR = a.\exp\ (-kt)$
5	Logarithmic	$MR = a.\exp(-kt) + c$
6	Two-term exponential	$MR = aexp(-kt) + (1-a)\exp\ (-kat)$
7	Wang and Singh	$MR = 1 + at + bt^2$
8	Diffusion approach	MR = aexp(-kt)+(1-a)exp(-kbt)

With the support of equations given as Equation 2, 3, and Equation 4, the amounts of the root-mean-square-error (RMSE), chi-square (X^2) values and modelling adequacy of the model (R^2) to explain the agreement between the experimental and statistical relations of models and even the estimated moisture values can be determined (Vega-Gálvez et al. 2008; Zogzas et al. 1996; Acar et al. 2020; Dağdeviren et al. 2021).

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{n}\left(MR_{\text{predicted}} - MR_{\text{experimental}}\right)^2\right]^{1/2} \quad (2)$$

$$X^2 = \frac{\sum_{i=1}^{n}(MR_{experimental} - MR_{predicted})^2}{N - z} \quad (3)$$

$$R^2 = 1 - \left[\frac{\sum(MR_{experimental} - MR_{predicted})^2}{\sum(MR_{predicted})^2}\right] \quad (4)$$

The root means square error (RMSE) that is expressed in equation 2 exhibits the deviation between the estimated values obtained by the model and the experimental values. In addition, equation 3, states while the chi-square (X^2) value decreases, the harmony between the experimental and empirical values increases. In addition, the model that is described as experimental data in Equation 4, moreover, the modelling adequacy (R^2) value as an amount whenever would be close to one is an indicator of the availability of the depicted model. At the early freeze-drying duration, the drying ratio exhibits incline behavior because of the high concentration of moisture at the face of the product.

Figure 8. Dried rosehip fruit.

According to the results of the statistical evaluation, the coefficients in the most suitable model are determined by the multiple regression method. In the light of the data obtained, a total of 8 models were applied and the most suitable drying model was determined from these 8 different models. These determination criteria depend on the R^2, X^2, and RMSE obtained from the introduced models.

Table 2. The results calculated by 8 kinetic drying models

Model No	Model Name	Model parameters	R^2	X^2	RMSE
1	Newton	k = 0.2834	0.9916	9.186×10^{-4}	0.028351
2	Page	k = 0.3709 n = 0.8247	0.9982	2.339×10^{-4}	0.013247
3	Modified Page I	k = 0.2966 n = 0.7982	0.9977	2.943×10^{-4}	0.014859
4	Henderson and Papis	a = 0.9779 k = 0.2772	0.9922	9.83×10^{-4}	0.027158
5	Logarithmic	a = 0.9551 c = 0.03282 k = 0.3093	0.9944	8.449×10^{-4}	0.022979
6	Two-term Exponential	a = 0.2389 k = 0.9408	0.9984	2.243×10^{-4}	0.012378
7	Wang and Sing	a = -0.1835 b = 0.008454	0.9336	8.424×10^{-3}	0.079488
8	**Diffusion Approach**	**a = 0.2597** **b = 0.2205** **k = 0.9888**	**0.9985**	$\mathbf{2.156\times10^{-4}}$	**0.011874**

Table 2 depicts the R^2, X^2, and RMSE values of 8 models. Here, the Page model has been seen as the most suitable drying model with an R^2 value of 0.9985, which is almost close to 1. and 2.156×10^{-4} as X^2, which is the closest quantity to 0. Another factor that supports the suitability of the Page model is that the root means square error (RMSE) value, which is close to 0, with the amount of 0.011874.

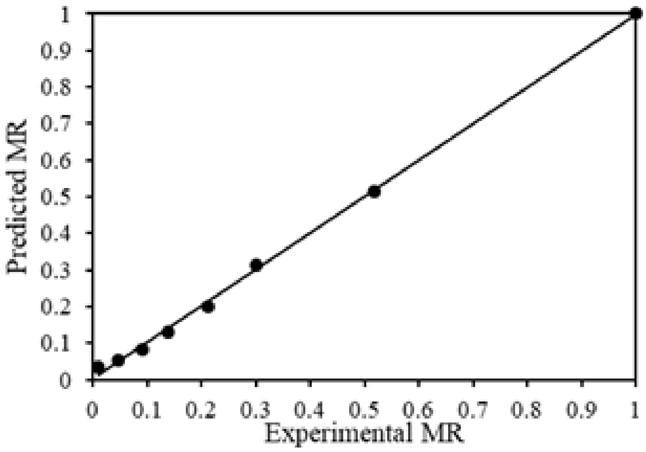

Figure 9. Comparison between experimental and predicted moisture ratio values applying the Diffusion Approach.

For food and material drying efficient diffusivity is an important transport characteristic that depends on the moisture content and temperature of a material. Fick's diffusion equation has a second law, which makes it a mass-diffusion equation for drying agricultural products in a fall-rate phase. The drying processes' theoretical model can be determined by its solution, which is shown in the equation given below:

$$\frac{\partial M}{\partial t} = D_{eff}\, \nabla^2\, M \quad (5)$$

Diffusion equation solution (Eq. 5) for slab geometry was first used by Crank (1975). He assumed that there is a negligible exterior resistance, uniform initial moisture distribution, negligible shrinkage, and constant diffusivity:

$$\boldsymbol{MR} = \frac{8}{\pi^2}\left[exp\left(-\frac{\pi^2 D_{eff}\, t}{4L^2}\right) + \frac{1}{9} exp\left(-9\frac{\pi^2 D_{eff}\, t}{4L^2}\right) + \frac{1}{25} exp\left(-25\frac{\pi^2 D_{eff}\, t}{4L^2}\right) + \frac{1}{49} exp\left(-49\frac{\pi^2 D_{eff}\, t}{4L^2}\right) \ldots \right] \quad (6)$$

Here t defines drying time (s), Deff shows effective diffusivity (m^2/s), n presents a positive integer, and L shows half-thickness of the samples (m). Keeping in view long drying duration with steady diffusion coefficient in a Cartesian coordinate system, we simplified Eq. 6 to a limiting form of the diffusion equation, as Eq. 7 reveals (Dagdeviren *et al*. 2021).

$$\boldsymbol{MR} = \frac{8}{\pi^2} exp\left(\frac{\pi^2 D_{eff}\, t}{4\, L^2}\right) \quad (7)$$

After plotting the experimental drying data for ln (MR) versus time, we determined effective diffusivity (Deff) values, as Figure 10 shows.

In Figure 10, we found slope (K) from the graph. For 5mm thick rosehip slices, effective diffusion value (Deff) was determined using Eq. 7, and its value was 1.99828×10^{-10} m^2/s. From this research, the effective diffusion value was found within the reference range 10-12 – 10-8 m^2/s for drying food materials. According to the literature, no research has been performed so far to establish a rosehip kinetic model, and no attempt has been made to quantify its effective diffusivity or moisture content in the freeze-drying process. We concluded that hawthorn's effective diffusivity has better agreement with the general effective diffusivity range for drying food materials.

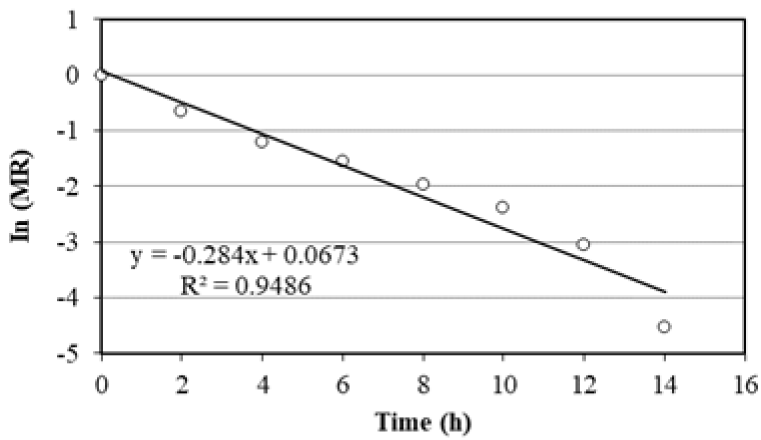

Figure 10. ln (MR) versus freeze-drying time for rosehip samples

Results

In this study, a total of 7 rosehip samples with 5 mm thickness and 100 g, as weight, were freeze-dried for 14 hours. MR (moisture ratio) was calculated with the weight loss data taken every two hours in 7 different samples and the most suitable model was determined on 8 different drying models by utilizing the MATLAB software. According to final calculations, it was seen that the most suitable model was the diffusion model, with the 0.9985 values for R^2, 2.156×10^{-4} as an estimated amount for the X^2, and also for RMSE (the root-mean-square-error) 0.011874. In addition, we found the effective diffusivity value, which was 1.99828×10^{-10} m^2/s for the mentioned rosehip slices. It was confirmed that the calculated effective diffusivity value was within the reference range mentioned in the literature ($10^{-12} – 10^{-8}$ m^2/s) for food products.

Nomenclature

a, b, c, n	The constants of the models
z	Number of parameters in the model
k, k0, k1	Drying rate constants (min^{-1})
t	Time (min)
M0	The initial moisture content (g water/g dry matter)
Mt	The moisture content at a time t (g water/g dry matter)
Md	The final equilibrium moisture content (g water/g dry matter)
MR	The moisture ratio (dimensionless)
N	Number of observations
MC	Moisture content (g water/g dry matter)
DR	Drying rate (g water/g dry matter)
Deff	The effective diffusivity (m2s-1)
L	Half-thickness of samples (m)
R^2	Coefficient of determination
χ^2	Reduced chi-square
RMSE	Root mean square error

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