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Application of a response surface Model for solar and indirect drying of muscat of alexandria grapes (*Vitis vinífera*

Aplicación de un Modelo de superficie de respuesta en el secado al sol y solar indirecto de uvas variedad moscatel de alejandria (*Vitis vinífera*)

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Abstract

By applying the response surface methodology, we optimized the sun and indirect solar drying of *Muscat of Alexandria* grapes. Initial moisture was measured and then the grapes were pretreated by thermochemical treatment with sodium hydroxide. Employing a centralized planning of rotating compounds, the pretreatment conditions were established, considering: temperatures, concentrations and times between 55–95 °C, 0.5–3.0 % and 2–30 seconds, respectively. For each drying process, drying times were determined, considering the final moisture content of 20 % on a wet basis, as well as the overall acceptance of the product. Using quadratic order polynomial equations, response surfaces were generated to obtain a shorter drying time and a maximum value of product acceptability, obtaining high values of coefficient of determination ($R^2 > 0.97$) and significant interactions of the independent variables ($p < 0.001$). Solar and indirect drying were optimized at 80 °C, 1.75 % and 16 s, for the temperature and concentration of the sodium hydroxide solution and immersion time, to obtain drying times of 75 h and 50 h, respectively, with a high degree of overall product acceptability and a score above 8 points on the established hedonic scale. Effective water diffusivity values of $8.57 \cdot 10^{-11}$ and $1.90 \cdot 10^{-10}$ m^2/s were obtained for the sun-drying and indirect solar drying process, respectively. Under optimal conditions, the mathematical model of Midilli et al. (2002) best simulates the variation of moisture versus drying time of *Muscat of Alexandria* grapes.

Keywords: Optimization, Grape drying; Mathematical modeling, Diffusivity, Response surface methodology.

Resumen

Al aplicar la metodología de superficie de respuesta, se optimizó el secado al sol y solar indirecto de uvas *Moscatel de Alejandría*. Se midió la humedad inicial y a continuación se pretrataron las uvas mediante tratamiento termoquímico con bajas concentraciones de hidróxido sódico, temperatura y tiempo de inmersión. Las condiciones se establecieron mediante un diseño experimental de compuesto central rotacional, considerando: temperaturas, concentraciones y tiempos entre 55–95 °C, 0.5–3.0 % y 2–30 segundos, respectivamente. Para cada proceso se determinaron los tiempos de secado, considerando el contenido de humedad final del 20 % en base húmeda, así como la aceptación global del producto. Empleando ecuaciones polinómicas de orden cuadrático, se generaron superficies de respuesta para obtener un menor tiempo de secado y un valor máximo de aceptabilidad del producto, obteniéndose altos valores de coeficiente de determinación ($R^2 > 0.97$) e interacciones significantes de las variables independientes ($p < 0.001$). El secado al sol y solar indirecto optimizó el pretratamiento a 80 °C, 1.75 % y 16 s, para la temperatura y concentración de la solución de hidróxido de sodio y tiempo de inmersión, para obtener tiempos de secado de 75 h y 50 h, respectivamente, con un alto grado de aceptabilidad global del producto y una puntuación superior a 8 puntos en la escala hedónica establecida. Se obtuvieron valores de difusividad efectiva del agua de $8.57 \cdot 10^{-11}$ y $1.90 \cdot 10^{-10}$ m^2/s para el proceso de secado al sol y secado solar indirecto, respectivamente. En condiciones óptimas, el modelo matemático de Midilli et al. (2002) es el que mejor simula la variación de la humedad frente al tiempo de secado de la uva *Moscatel de Alejandría*.

Palabras clave: Optimización, Secado de uva, Modelación matemática, Difusividad, Metodología de superficie de respuesta.

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1 Introduction

Grapes in their fresh state and immediately after harvest have a high moisture content (Gopinath et al., 2022), so their water activity and sugar content make them highly susceptible to microbial spoilage during storage, even under low-temperature refrigeration conditions. Therefore, after harvest, they must be consumed or processed into different products through methods such as drying (Adiletta et al., 2016).

Raisins are an edible product with high nutritional and energy value; in Peru, they are produced using traditional methods through direct sun-drying. This activity takes place in areas with high solar radiation and generally occurs between January and April. In Latin America, Argentina is the country that produces raisins on a large scale, and globally, the leading

The industry has developed artificial drying methods, and there are currently a variety of industrial dryers available for agribusiness. Solar energy is the predominant renewable energy source, compared to wind energy and biomass, due to its constant availability (Gunasekaran et al., 2021; Kumar et al., 2021). Sun drying is one of the oldest forms of preservation and perhaps the cheapest and most common method of preserving agricultural products practiced in most tropical countries. However, this technique has disadvantages: it is weather-dependent, requires a long drying time, leads to dust formation, microbial growth, contamination, insect infestation, spoilage, high labor costs, and variable quality, among others (Djebli et al., 2020; Naigam et al., 2021).

On the other hand, the use of indirect solar drying equipment is a good alternative from both an economic and environmental standpoint. Solar drying involves converting solar energy into thermal energy, which is then used to remove moisture from the product (Shalaby and Bek, 2014); through natural convection, air flows inside the dryer (Gopinath et al., 2022). Therefore, the use of solar energy and hot air in convective dryers is a potential alternative for application in tropical and subtropical countries (Vigneshkumar et al., 2021) and also because it is faster, economically viable, hygienic, and more efficient than sun drying, providing dried products with better color and surface texture (Castillo-Téllez et al., 2017; Singh et al., 2012).

Currently, there are numerous research studies on agro-industrial products that were processed using conventional drying methods (sun and air) and modern drying techniques, such as direct sun drying, solar dryers, hot-air convection dryers, hot-air convection dryers assisted by different types of energy, microwaves, infrared, and a combination of these, the use of the ice sublimation process or freeze-drying, heating and air or vacuum drying, and other combined hybrid drying techniques (Atak et al., 2022; Javed et al., 2023), where attempts have been made to apply pretreatments in various forms prior to drying and to observe several parameters, including, primarily, product quality and overall acceptance, drying times, effects on rehydration characteristics, and nutritional quality in the final product (Pragati and Preeti, 2012).

However, there is interest in optimizing drying processes using response surface methodologies, a technique in which a set of variables is examined simultaneously through experimental design, allowing for the identification of variable interactions and the optimal conditions for a maximum or minimum value or a specific area of the generated surface (Bitaraf et al., 2012; Chopra et al., 2011; Myers et al., 2016; Ramos et al., 2015).

A review of the prior literature revealed no research on the optimization of the drying process for Muscat of Alexandria grapes. Therefore, the objectives of this study are to investigate the effect of sodium hydroxide concentration pretreatment, temperature, and immersion time on drying time under direct sunlight and solar drying using an extended-surface collector; to evaluate the quality of the raisins in terms of overall product acceptance; to estimate the constants of the drying curve equations; and to evaluate the water diffusivity under optimal drying conditions.

2 Materials and methods

2.1 Raw Material

The freshly harvested fresh grapes come from the Ica Valley in Peru and were purchased at the fruit market in the city of Chincha Alta. The grapes were washed, then dried and measured with a Vernier caliper. For the physicochemical characterization of the raw material, preliminary determinations were made of pH, Brix degrees (°Brix), refractive index (–), water content (%), and water activity (a_w).

2.2 Grape Pretreatment

Before drying, the selected samples underwent a chemical-thermal treatment, considering temperature, concentration, and immersion time; they were then washed with plenty of cold water at 20 °C and drained, with surface moisture removed using paper towels.

Table 1 shows the coding of the operational variables (temperature, concentration, and immersion time), investigated at five levels each, and the effects on the response variable, drying time, and overall acceptance.

Table 1. Coding of the operational variables for drying the Muscat of Alexandria grape variety (*Vitis vinifera*).

Variables	**Code**	-1.68	-1	0	$+1$	$+1.68$
Temperature (°C)	x_1	55	63	75	87	95
Concentration (%)	x_2	0.5	1	1.75	2.5	3.0
Immersion time (s)	x_3	2	7.7	16	24.3	30

A rotational central composite design (RCCD) was used, consisting of a full factorial design 2^3, 06 axial points (levels ± 1.68), and 06 central points (level 0), totaling 20 trials, conducted in duplicate and randomly, to determine the optimal process conditions. The design with coded and actual levels and the results are shown in Table 4.

2.3 Drying System

A stainless-steel solar dryer (Figure 1) built specifically for this research project was used. The dryer is a commercial-scale prototype. The total dimensions of the dryer are: $2276 \times 2558 \times 806$ mm (height $\times$ length $\times$ width), constructed from 1.5 mm thick 304 stainless steel sheets and structural supports made of 2-inch square profiles. The dimensions of the solar collector with an expanded surface area were: $173 \times 806 \times 2000$ mm, with a 3-mm-thick transparent glass cover, at a $15°$ angle of inclination relative to the horizontal plane and oriented toward the north. The dimensions of a single fin were $1.5 \times 25 \times 2000$ mm, and there were 27 fins on the collector surface. The approximate weight of the equipment was 350 kg. The drying equipment was installed in the open-air courtyard of the Navarro winery in Chincha Alta, at $13°25.3'$ S latitude and $76°7'57''$ W longitude. The experimental module was equipped with thermometers to measure the air temperatures at the solar collector's inlet and outlet, the drying air temperatures along the length of the dryer, and the drying air velocity.

Following the chemical-thermal treatments, the grape drying process was carried out by placing samples with an average weight of 101.27 ± 4.58 g on their respective sample holders in direct sunlight and in the indirect solar dryer. In the latter, the chimney effect combined with the heating of the air in the solar collector contributed to the movement of the drying air and its flow through the drying chamber until it exited the dryer. During the test period, the average ambient air temperature and the temperature of the air entering the drying chamber were 27 and 40 °C, respectively, with an average solar radiation value of 300 W/m^2.

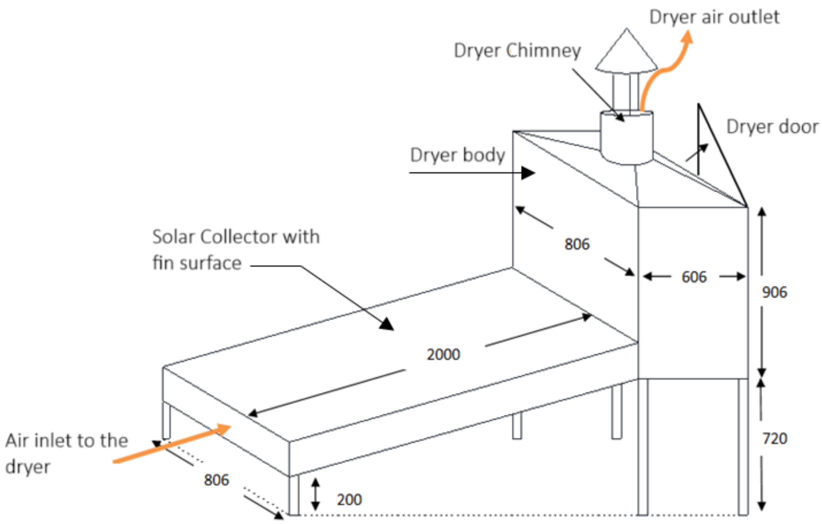

Figure 1. Schematic of the commercial-scale solar dryer.

2.4 Moisture Ratio

The dimensionless moisture module during sun-drying and indirect solar drying (Kusuma et al., 2023) is presented in Equation 1:

$$MR = \frac{X(t) - X_e}{X_o - X_e} \tag{1}$$

Where: $X(t)$ is the average moisture content at time t, X_0 is the initial average moisture content, and X_e is the average moisture content at equilibrium.

2.5 Effective water diffusivity (D_{eff})

In the general form of the diffusion equation in spherical coordinates without considering mass generation, the expression is represented as Equation 2:

$$\frac{1}{r^2}\frac{\partial}{\partial r}\left(r^2\frac{\partial X}{\partial r}\right) + \frac{1}{r^2 sin\theta}\frac{\partial}{\partial \theta}\left(sin\theta\frac{\partial X}{\partial \theta}\right) + \frac{1}{r^2 sin^2\theta}\frac{\partial^2 X}{\partial \phi^2} = \frac{1}{D}\frac{\partial X}{\partial t} \tag{2}$$

Likewise, considering that diffusion occurs only along the radial axis, the simplified equation for water mass diffusion through the grape cross-section in spherical coordinates is given by Equation 3:

$$\frac{\partial X(r,t)}{\partial t} = \frac{1}{r^2}\frac{\partial}{\partial r}\left(r^2 D\frac{\partial X}{\partial r}\right) \tag{3}$$

For the initial and boundary conditions at the surface ($r = R$) and at the center of the grape berry ($r = 0$), they are (Equations 4 and 5):

$$X(t = 0.0 \leq r < R) = X_0 \tag{4}$$

$$X(t = 0.0 \leq r < R) = X_0 \tag{5}$$

It is assumed that the initial moisture distribution is uniform within the sphere, that the surface moisture is equal to the equilibrium moisture content, and that radial diffusion is symmetric.

Considering that the entire drying process occurs during a period of decreasing drying rate and that the liquid diffusivity is the dominant factor in the entire process—*i.e.*, the drying process is entirely controlled by the internal resistance to mass transfer—, Fick's second law of diffusion can be used to describe the drying process (Doymaz, 2007; Saravacos and Maroulis, 2001; Srikiatden and Roberts, 2006).

Equation 3 can be solved analytically by assuming a constant radius (shrinkage is not considered) and a constant diffusivity coefficient during the drying process, which yields Fick's law for diffusion in spherical geometries (Equation 4):

$$MR = \frac{X(t) - X_e}{X_o - X_e} = \frac{6}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{n^2}\exp\left[\frac{-n^2\pi^2 \cdot D \cdot t}{R^2}\right] \tag{6}$$

And for long drying times, Equation 4 can be simplified and expressed (Crank, 1975; Di Matteo et al., 2000; Doymaz, 2004; Rizvi, 1986) as shown in Equation 7:

$$MR = \frac{X(t) - X_e}{X_o - X_e} = \frac{6}{\pi^2} \exp\left(\frac{-\pi^2 \cdot D_{\text{eff}} \cdot t}{R^2}\right) \quad (7)$$

Taking the logarithm of both sides (Ecuation 8):

$$\text{Ln}(MR) = \text{Ln}\left(\frac{6}{\pi^2}\right) - \left(\frac{\pi^2 D_{\text{eff}} \, .t}{R^2}\right) \quad (8)$$

Where R (m) is the average radius of the grape and D_{eff} is the effective moisture diffusivity (m^2/s), which is determined from the slope of the graph of $\ln(\text{MR})$ versus drying time (t).

2.6 Response Surface Methodology

This methodology comprises a set of mathematical techniques used to optimize the drying process, where the response variables were drying time and overall product acceptance, which were influenced by the quantitative factors: temperature (°C), concentration (%), and immersion time (s).

According to the proposed experimental design, the response surface methodology technique approximates a second-order polynomial mathematical relationship, as shown in Equation 9:

$$y = \beta_o + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 \sum_{i<1} \sum \beta_{ij} x_i x_j + \varepsilon \quad (9)$$

2.7 Mathematical Simulation of the Drying Curve

Six mathematical models (Table 2) were used to simulate the drying curve under optimal conditions, namely: Henderson-Pabis, modified Henderson-Pabis, Page, modified Page, Newton, and Midilli et al. (2002) (Bombana et al., 2023; Kusuma et al., 2023; Subbian et al., 2021).

Table 2. Mathematical models for simulating the drying curve.

Model	Ecuation	
Henderson-Pabis	$MR = a \, . \exp(-k.t)$	(10)
Henderson-Pabis Modificado	$MR = a \, . \exp(-k.t) + b \, . \exp(-g.t) + c \, . \exp(-h.t)$	(11)
Page	$MR = \exp(-k.t^n)$	(12)
Page Modificado	$MR = [\exp(-k.t)]^n$	(13)
Newton	$MR = a \, \exp(-k.t)$	(14)
Midilli *et al.*	$MR = a \, . \exp(-k.t^n) + b.t$	(15)

2.8 Statistical Analysis

The Statistica v. 7.0 software was used to analyze the effects of the independent variables on drying time and overall product acceptance. The coefficient of determination R^2 was also determined, indicating the degree of fit of the quadratic polynomial to the response surface. The fit of the constants of the proposed nonlinear mathematical models to simulate the drying curve was determined using the SOLVER software package (Microsoft Corporation MS-Excel®), which employs the generalized reduced gradient method with a convergence level of 10^{-5}.

For the proposed models, the quality of fit of the equation to the experimental data was evaluated by calculating the coefficient of determination (R^2), the sum of squared errors (SSE), the root mean square error (RMSE), the mean relative percentage error (EMR), and the reduced chi-square (χ^2) (Equations 7, 8, 9, 10, and 11, respectively).

$$R^2 = 1 - \frac{\sum_{i=1}^{N} (MR_{\text{exp}_i} - MR_{\text{mod}_i})^2}{\sum_{i=1}^{N} \left(\overline{MR}_{\text{exp}_i} - MR_{\text{mod}_i}\right)^2} \quad (16)$$

$$SSE = \frac{1}{N} \sum_{i=1}^{N} (MR_{\text{exp}_i} - MR_{\text{mod}_i})^2 \quad (17)$$

$$RMSE = \left[\frac{1}{N} \sum_{i=1}^{N} (MR_{\text{exp}_i} - MR_{\text{mod}_i})^2\right]^{1/2} \quad (18)$$

$$EMR(\%) = \frac{100}{n} \sum_{i=1}^{n} \frac{\left| MR_{\exp_i} - MR_{\mathrm{mod}_i} \right|}{MR_{\exp_i}} \quad (19)$$

$$\chi^2 = \frac{1}{N-n} \sum_{i=1}^{N} (MR_{\exp_i} - MR_{\mathrm{mod}_i})^2 \quad (20)$$

3 Results and Discussion

3.1 Physicochemical Characterization

The results of the grapes' physicochemical properties are shown in Table 3, presenting the mean values and corresponding standard deviation. The pH value ranges from 4.00 to 3.26, classifying it as a sweet-and-sour product. Meanwhile, the water content ranges from 79.24 % to 80.76 %, falling within the typical range found in all grape varieties, which ranges from 70 % to 80 % (Srivastava et al., 2021), and the water activity value fluctuated between 0.9627 and 0.9671.

Meanwhile, the concentration of soluble solids in the form of sucrose found in the berry ranged from 17.21 to 23.35 Brix degrees, a characteristic value of this type of berry product in its ripe state. Similar values of 22.4, 22, and 21.4 °Brix and 3.63, 3.20, and 3.44 for pH have been reported for Muscat of Alexandria grape varieties of fruits considered ripe and suitable for making grapes or wine (Corona et al., 2020; Harutyunyan et al., 2022; Pedrosa-López et al., 2022).

Table 3. Physicochemical variables of the Muscat of Alexandria grape variety (*Vitis vinifera*)

Physicochemical variable	Mean ± standard desviation
Hydrogen ion concentration (pH)	3.63 ± 0.37
Brix degrees (°Bx)	20.28 ± 0.36
Refractive index (–)	1.364 ± 0.005
Water content (%)	80.00 ± 0.76
Water activity (a_w)	0.9649 ± 0.0022

3.2 Influence of independent variables on drying time under direct and indirect sunlight

Figure 2 (a and b) shows the Pareto chart for analyzing the effect of the independent variables: T (°C), C (%), and t (s), combined on the response variable of drying time, using sun drying and the indirect solar dryer to produce Muscat of Alexandria grapes (Table 4). A strong influence of solution temperature (°C), immersion time (s), and concentration (%) is observed. The linear interaction of concentration with immersion time is not influential in either process; concentration is not influential for sun drying. However, in indirect solar drying, the interactions between temperature-concentration and temperature-immersion time were included in the regression model. Although they did not show a significant influence on the response variable, their inclusion allows for greater precision in surface area modeling.

3.3 Influence of independent variables on the overall acceptance of fruit berries: sun-drying and indirect solar drying

Table 4 presents the global acceptance values. Furthermore, the estimated effects of the independent variables on the global acceptance of the Muscat of Alexandria grape variety are presented in Figures 2 (a, b) and 3 (a, b).

It is observed that the three variables—temperature, concentration, and immersion time—in their linear and quadratic forms have effects on the dependent variables of drying time and overall acceptance, except concentration in its quadratic function (Q) in sun-drying; likewise, the linear interaction of temperature and concentration (1L × 2L) and temperature and immersion time (1L × 3L) in the evaluation of drying time and overall acceptance is considered less important in indirect solar drying. However, to ensure that the mathematical model did not lose accuracy in the coefficient of determination (R^2), all interactions were considered to obtain the regression coefficients of the response surface.

Table 4. Central rotational composite design with coded and actual values and response variables.

Treatment	Coded variables			Current variables			Sun-drying		Indirect solar drying	
	x_1	x_2	x_3	T (°C)	C (%)	t (s)	Drying de time (h)	Overall acceptance	Drying de time (h)	Overall acceptance
1	-1	-1	-1	63	1	7.7	112	6	110	3
2	1	-1	-1	87	1	7.7	90	7	64	6
3	-1	1	-1	63	2.5	7.7	122,5	6.5	72	8
4	1	1	-1	87	2.5	7.7	63	7	60	9
5	-1	-1	1	63	1	24.3	66.5	7	64	3
6	1	-1	1	87	1	24.3	72	8	51	6
7	-1	1	1	63	2.5	24.3	149	6	92	5
8	1	1	1	87	2.5	24.3	82.5	7	60	8
9	-1.68	0	0	55	1.75	16	142.5	6	112	6
10	1.68	0	0	95	1.75	16	79.5	7	45	7
11	0	-1.68	0	75	0.5	16	84.5	8	80	4
12	0	1.68	0	75	3	16	81.5	6	75	7
13	0	0	-1.68	75	1.75	2	171	6.5	107	4
14	0	0	1.68	75	1.75	30	82.5	9	60	8
15	0	0	0	75	1.75	16	77.5	8.5	68	9
16	0	0	0	75	1.75	16	73	8	65	8
17	0	0	0	75	1.75	16	75	8.5	58	9
18	0	0	0	75	1.75	16	79.5	8	64	9
19	0	0	0	75	1.75	16	74	8.5	68	8
20	0	0	0	75	1.75	16	76	8	63	8

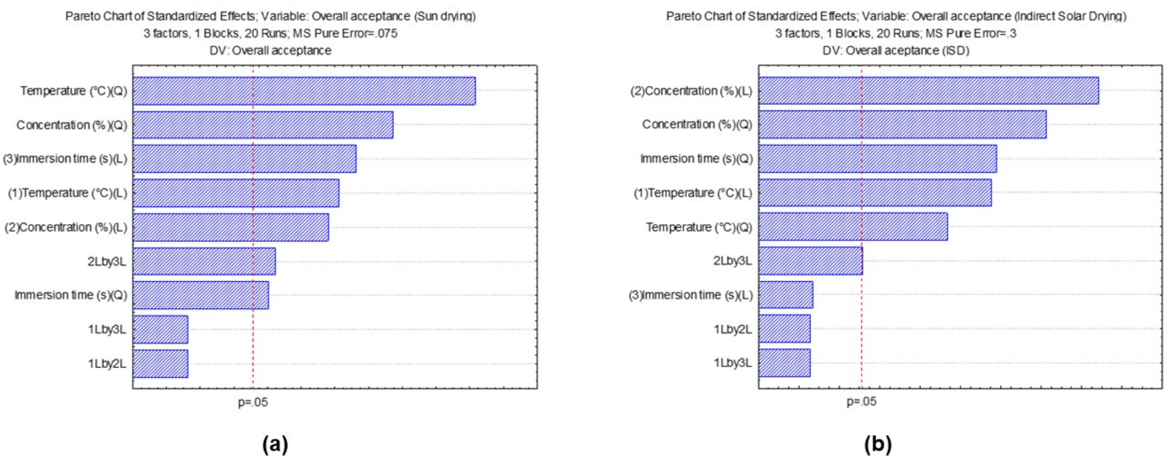

Figure 2. Effect of independent variables on the overall acceptance of the Muscat of Alexandria fruit berry (a) sun-dried (b) indirectly sun-dried.

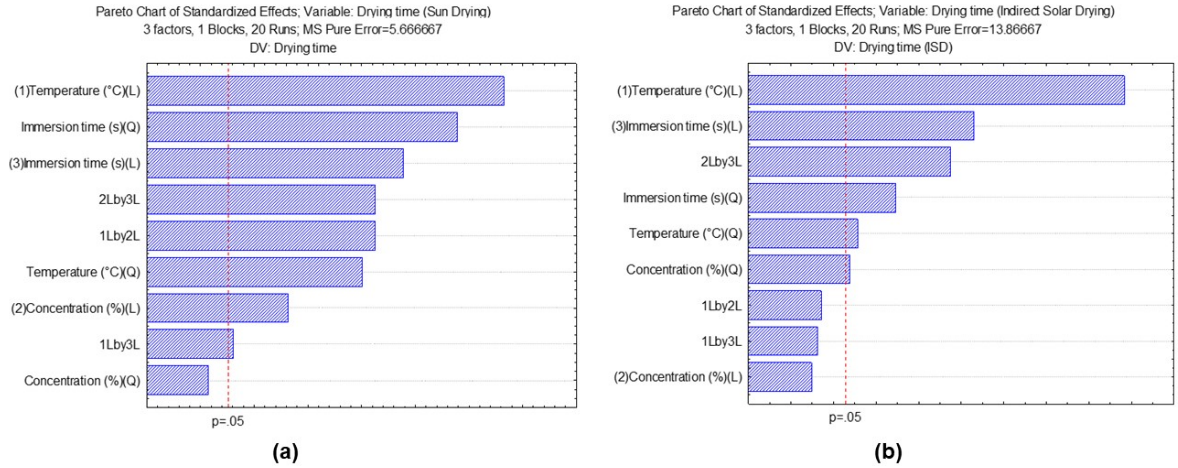

Figure 3. Effect of independent variables on the drying time of the Muscat of Alexandria grape variety (a) sun drying (b) indirect sun drying.

3.4 Analysis of variance in the estimation of drying time and overall product acceptance for sun-drying and indirect solar drying

Upon analyzing the significance of the constants in the regression equations (Table 5), it was observed that the calculated F-value (F_c) exceeded the critical value from the F-table $F_{0.95;9;10} = 3.02$ for a 95 % confidence level. Since in all cases $F_c > F_t$, both for the prediction of drying time and for the overall acceptance of the berry, it is concluded that the regression models are statistically significant. The regression constants for estimating drying time and overall acceptance of the berries are presented in Tables 6 and 7, respectively, for both sun drying and indirect drying.

3.5 Response surface in the estimation of drying time and overall product acceptance in sun-drying

During the drying process, the waxy layer on the grape skin hinders the removal of water *in situ* and extends the drying time. For this reason, chemical pretreatments have been proposed that cause the dissolution of this layer, along with the formation of microcracks on its surface, thereby controlling the rate of water loss and reducing the time required to achieve the correct level of dehydration (Corona et al., 2020; Patidar et al., 2021).

Figure 4 shows the response surface of drying time (h) (a) and contour (b) versus Temperature (°C) and immersion time. For immersion times in the range of 16 to 20 seconds and solution temperatures in the range of 80 to 90 °C and solution concentrations ranging from 1 % to 1.5 %, drying times of less than 80 hours were achieved, equivalent to less than 11 days of direct sun drying, thereby reducing the processing time compared to the results reported by Corona et al. (2020), who applied a chemical pretreatment to Muscat of Alexandria grapes by immersing them in a sodium hydroxide solution (45 g/L) for 185 seconds at a temperature of 25 °C, resulting in a drying time of 13 days under these conditions. This is due to the phenomenon of dehydration, which is explained by mass transfer, influenced by water and sugar content, as well as by the homogeneity of the berries to be dried in terms of size, weight, and density.

The drying rate of berries is directly proportional to the dehydration temperature: at higher temperatures, the mass transfer rate increases and the processing time decreases. For example, in the 'Zicui' cultivar, the most efficient dehydration occurred at 50 °C; in contrast, the time required to complete the process extended to 11 days at 40 °C and up to 20 days when processed at 30 °C (Chen et al., 2022).

Figure 5 shows the response surface (a) and contour (b) of Overall Acceptance versus Temperature (°C) and solution concentration (%), while keeping the immersion time constant at 16 seconds.

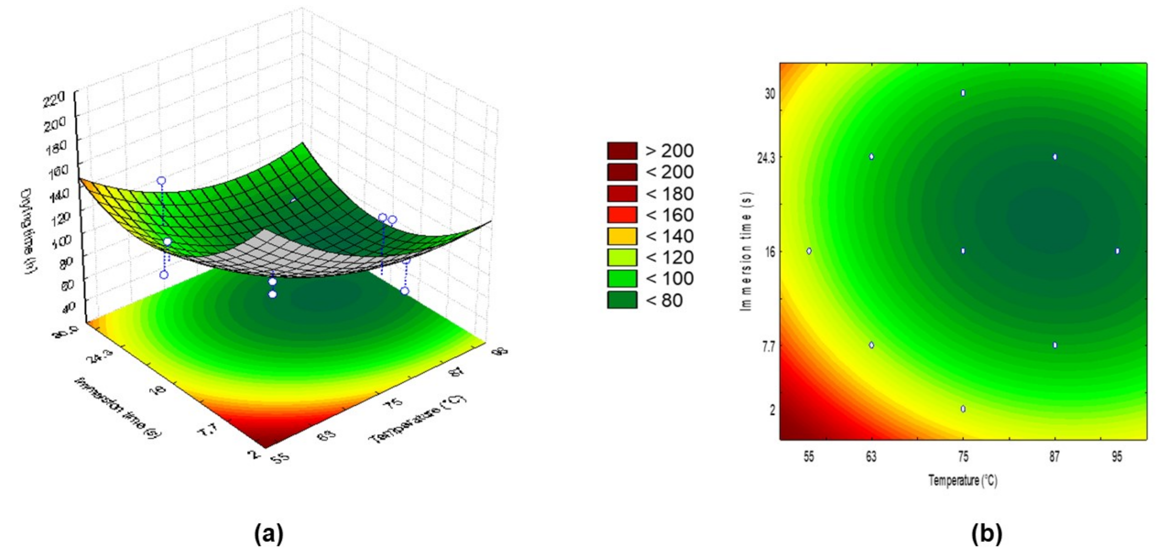

Figure 4. Response surface (a) and contour (b) of the overall acceptance of Alexandria Muscat berries versus Temperature (°C)and solution concentration (%).

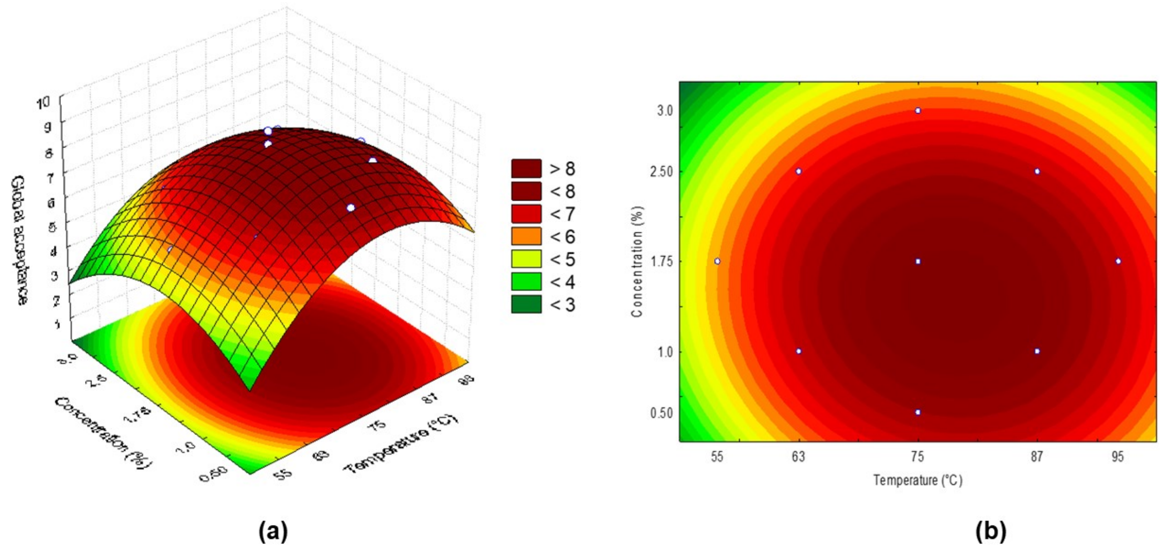

Figure 5. Response surface of drying time (h) (a) and contour (b) for the Muscat of Alexandria grape variety versus temperature (°C)and immersion time (%).

Table 5. Analysis of variance for drying time and overall acceptance of the Muscat of Alexandria grape variety in sun-drying and indirect sun-drying.

		Drying time					Overall acceptance				
Process		SS	GL	MQ	F_c	F_t	SS	GL	MQ	F_c	F_t
Sun-dried	Regression	14 250.11	9	1 583.34	5.10	3.02	15.99	9	1.776	8.27	3.20
	Residual error	3 105.59	10	310.56			2.15	10	0.215		
	Misalignment	3 077.26	5	615.45	108.62		1.77	5	0.335	4.73	
	Pure error	28.33	5	5.67			0.38	5	0.075		
Indirect solar drying	Regression	5 755.14	9	639.46	5.62	3.02	62.06	9	6.896	4.40	3.02
	Residual error	1 138.66	10	113.87			15.69	10	1.569		
	Adjustment error	1 069.32	5	213.87	15.42		14.19	5	2.838	9.46	
	Pure error	69.33	5	13.87			1.50	5	0.30		

Note: SS: sum of squares; DF: degrees of freedom; SM: standard mean; F_c and F_t: calculated and table F-values, respectively.

Table 6. Estimated effects and regression coefficients of the independent variables on the response variable of sun-drying time and overall acceptance of the Muscat of Alexandria raisin

	Drying time (h) ($R^2 = 0.82062$)				**Overall acceptance** ($R^2 = 0.88033$)			
Coded parameter	Estimated effect	P- value	Regression coefficien	P-value	Estimated effect	P-value	Regression coefficient	P-value
k_0	76.3072	0.000000	76.3072	0.000000	8.256595	0.000000	8.256595	0.000000
x_1	-36.4007	0.000001	-18.2003	0.000001	0.75926	0.003705	0.379632	0.003705
x_2	18.9122	0.000023	9.4561	0.000023	-1.32369	0.000260	-0.661845	0.000260
x_3	10.4743	0.000458	5.2372	0.000458	-0.71236	0.004866	-0.356180	0.004866
x_1^2	-0.9291	0.492746	-0.4646	0.492746	-0.96938	0.001115	-0.484690	0.001115
x_2^2	-24.3580	0.000008	-12.1790	0.000008	0.83548	0.002441	0.417742	0.002441
x_3^2	30.0729	0.000002	15.0346	0.000002	-0.43729	0.029070	-0.218959	0.029070
$x_1 \cdot x_2$	-27.3750	0.000016	-13.6875	0.000016	-0.12500	0.547054	-0.0625000	0.547054
$x_1 \cdot x_3$	5.1250	0.028597	2.5625	0.028597	0.12500	0.547054	0.0625000	0.547054
$x_2 \cdot x_3$	27.3750	0.000016	13.6875	0.000016	-0.62500	0.023271	-0.3125000	0.023271

Nota: $x_1 = \frac{T-75}{12}$; $x_2 = \frac{C-1.75}{0.75}$ y $x_3 = \frac{t-16}{8.30}$, where: T: temperature, C: concentration and t: immersion time.

Table 7. Efectos estimados y coeficientes de regresión de las variables independientes sobre la variable de respuesta del tiempo de secado solar indirecto y aceptación global de la uva-pasa variedad Moscatel de Alejandría.

	Tiempo de secado (h) ($R^2 = 0.82062$)				**Aceptación global** ($R^2 = 0.88033$)			
Parámetro codificado	Efecto estimado	Valor P	Coeficiente de regresión	Valor P	Efecto estimado	Valor P	Coeficiente de regresión	Valor P
k_0	64.6401	0.000000	64.6401	0.000000	8.49475	0.000000	8.49475	0.000000
x_1	-31.5959	0.000019	-15.7980	0.000019	1.71201	0.002192	0.85600	0.002192
x_2	6.1494	0.025972	3.0747	0.025972	-1.35061	0.005468	-0.67530	0.005468
x_3	-1.9641	0.374713	-0.9821	0.374713	2.49765	0.000387	1.24883	0.000387
x_1^2	5.4407	0.039418	2.7204	0.039418	-2.05923	0.000845	-1.02961	0.000845
x_2^2	-17.2901	0.000355	-8.6451	0.000355	0.39869	0.236591	0.19934	0.236591
x_3^2	9.6924	0.004350	4.8462	0.004350	-1.70492	0.001992	-0.85246	0.001992
$x_1 \cdot x_2$	3.7500	0.213701	1.8750	0.213701	-0.50000	0.253170	-0.25000	0.253170
$x_1 \cdot x_3$	3.2500	0.271944	1.6250	0.271944	0.50000	0.253170	0.25000	0.253170
$x_2 \cdot x_3$	19.7500	0.000666	9.8750	0.000666	-1.00000	0.049313	-0.50000	0.049313

Note: $x_1 = \frac{T-75}{12}$; $x_2 = \frac{C-1.75}{0.75}$ y $x_3 = \frac{t-16}{8.30}$, where: T: temperature, C: concentration and t: immersion time.

As the values of the independent variables increase, they generate a curve with a maximum inflection point for Overall Acceptance (> 8) of the berries at 20 % moisture content on a wet basis. It is observed that the highest values of Overall Acceptance occur in the temperature range of 80–85 °C for the NaOH solution at concentrations between 1 % and 1.5 %. It is recommended to use low concentrations of the solution so that it can be easily removed with plenty of cold water after the chemical-thermal treatment.

According to Patidar et al. (2021), many desirable changes in the physical, chemical, and biochemical properties occur during dehydration due to the pretreatment and drying conditions. Thus, the carbohydrates and organic compounds from the fresh grapes are retained in a concentrated form in the grapes, improving their acceptability to consumers. Furthermore, NaOH not only improves drying characteristics but also enhances the color attributes of the grapes (Vázquez et al., 2000).

In addition to producing high-quality raisins, unfortunately there are some environmental and health concerns related to the use of chemicals for the pretreatment of grapes. On the environmental aspect, wastewater containing organic, saline, and highly corrosive solids is generated; and in terms of food safety, these chemicals remain in the dried product, which can cause food safety issues (Adiletta et al., 2016; Deng et al., 2019; Wang et al., 2017). The low concentration of NaOH (1–1.5 %) used in this study accelerates the drying process while reducing the likelihood of producing raisins with a negative environmental impact and unsafe food, as these two factors intensify at higher NaOH concentrations.

Based on the analysis, Table 8 presents the independent variables or processing parameters for direct sun drying to minimize and maximize drying time and overall product acceptance, respectively.

Table 8. Direct sun-drying processing parameters for the Muscat of Alexandria grape variety

Independent variable	**Process parameters**	**Drying time (h)**	**Overall acceptance (–)**
Temperature (°C)	80–85	Approximately ≤ 80 h	Approximately > 8
Concentration (%)	1–1.5		
Immersion time (s)	16–20		

3.6 Response surface for estimating drying time and overall product acceptance in indirect solar drying

Figure 6 shows the response surface (a) and contour surface (b) of the drying time of Muscat of Alexandria grapes versus temperature (°C) and immersion time (s). It is stated that, for immersion times of 16 to 20 seconds, a solution temperature of 80 to 90 °C, and a solution concentration of 1 % to 2 %, using the indirect solar dryer, drying times of less than 60 hours are achieved, equivalent to less than 6 to 7 days of drying.

The shortest drying time was obtained with indirect solar drying compared to sun drying; this is because the former uses a solar collector consisting of a transparent glass surface and a black absorber, where solar energy is collected to heat the fresh air entering the drying chamber, where the grapes are heated by indirect heat absorption. Furthermore, due to the pressure difference generated by the chimney, natural air circulation is achieved, which accelerates the evaporation of moisture present in the grapes (EL-Mesery et al., 2022).

Another factor to consider is that indirect solar drying was performed with an average solar radiation of 300 W/m^2. Leon Dharmadurai et al. (2022), using reflectors, achieved a maximum solar radiation of 1079.8 W/m^2 and a drying time of 5 days for seedless green grapes and concluded that the reduction in drying time is positively influenced by the indirect solar drying method and the natural convection of hot air within the drying chamber.

Figure 7 shows the response surface (a) and contour plot (b) of the overall acceptability score of Alexandria Muscat grapes versus temperature (°C) and immersion time (s). Global acceptability values for the product greater than 8 are achieved provided that the solution concentration is maintained at 1 % and temperatures between 75–90 °C and immersion times of 16–25 seconds are used.

The curves show an inflection point, with maximum acceptance levels for the grapes occurring at concentrations of 1 % and 1.5 %, respectively, and the highest acceptance levels corresponding to approximately 16 seconds. The parameters of the indirect solar drying process are presented in Table 9.

Unlike sun drying, the indirect solar drying process reduces drying time by 20 hours, yielding grapes with a high overall acceptance rate. Likewise, the optimal conditions are found near the central points of the experimental design, where the overall acceptance values were the highest. Therefore, for analyzing the drying curve and kinetics, the experimental data obtained were: $T = 80$ °C, $C = 1.5$ %, and $t = 16$ s.

Figures 8 (a and b) present the simulations, using the regression equations for the drying time of grapes in the sun (Table 6) and the overall acceptance of the product in the indirect solar drying process (Table 7), showing the curves of the minimum and maximum values, respectively.

3.7 Drying curve and kinetics

3.7.1 Drying curve

Figure 9 shows the behavior of the evolution of dry-matter moisture content as a function of drying time, both for the sun-drying of the Moscatel of Alejandría grape variety and for the use of the indirect solar dryer. In the direct sun-drying curve, the moisture content of the grapes decreases gradually until reaching a final moisture content of 20 % on a wet basis, which corresponds to approximately 0.25 kg/kg on a dry basis, with an average drying time of 75 hours. Meanwhile, the grapes dried in the indirect solar dryer exhibited two drying phases: a pronounced initial phase after 24 hours of drying, followed by a gradual decline to the indicated final moisture content, with an average drying time of 50 hours.

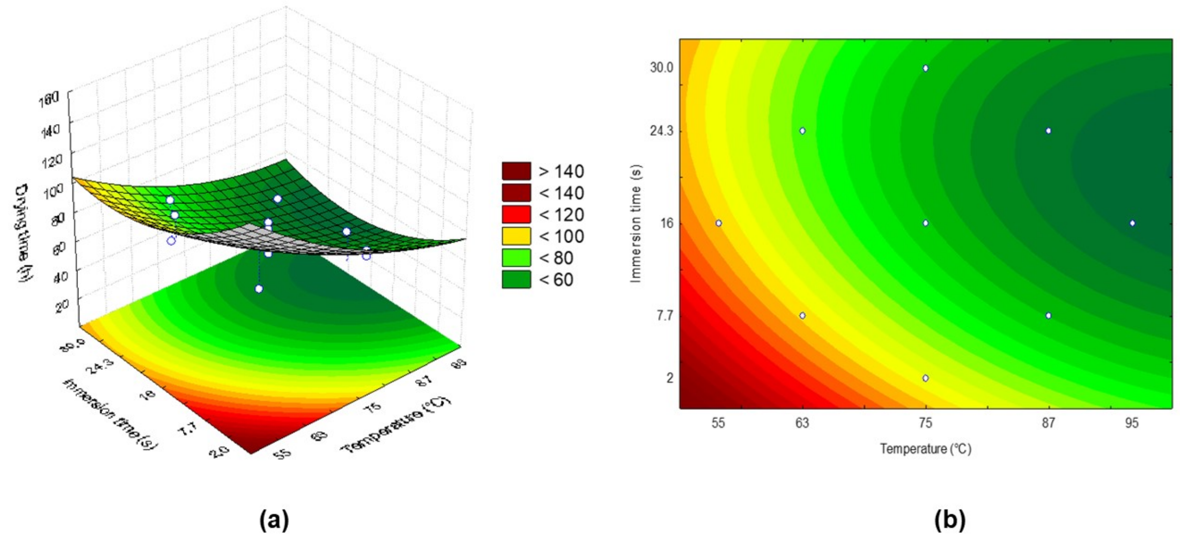

Figure 6. Response surface of drying time (h) (a) and contour (b) versus Temperature (°C)) and immersion time (s) using an indirect solar dryer for drying the Muscat of Alexandria grape variety.

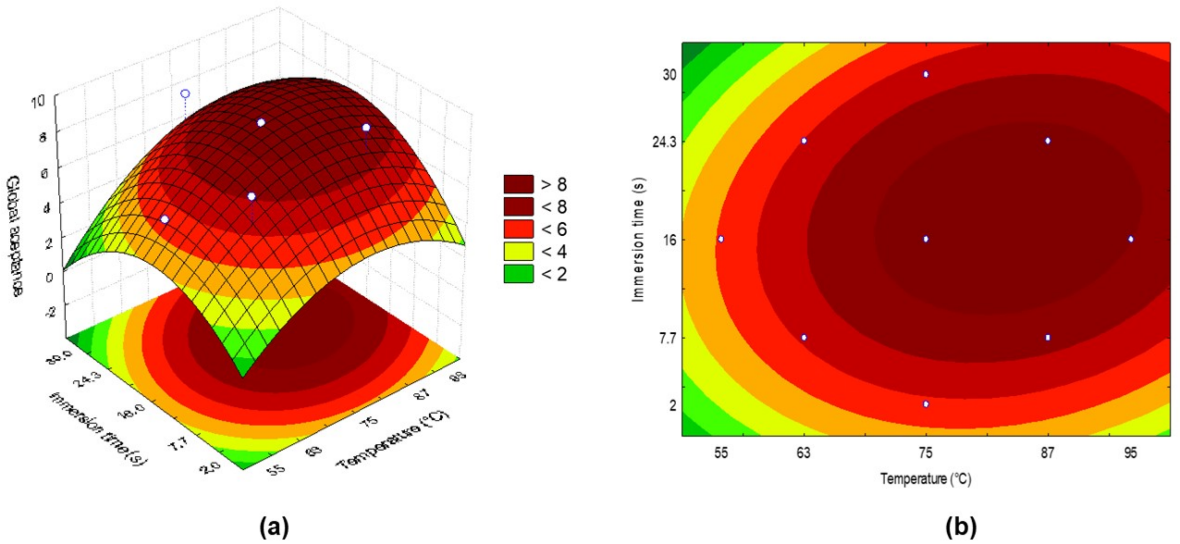

Figure 7. Response surface of global acceptance (a) and contour (b) for the Muscat of Alexandria grape variety versus temperature (°C)and immersion time (s) for drying using an indirect solar dryer.

Table 9. Processing parameters for indirect solar drying of the Muscat of Alexandria grape variety

Independent variable	Process parameters	Drying time (h)	Overall acceptance (–)
Temperature (°C)	80–85	Approximately $\leq$ 60 h	Approximately > 8
Concentration (%)	1–2.5		
Immersion time (s)	16–20		

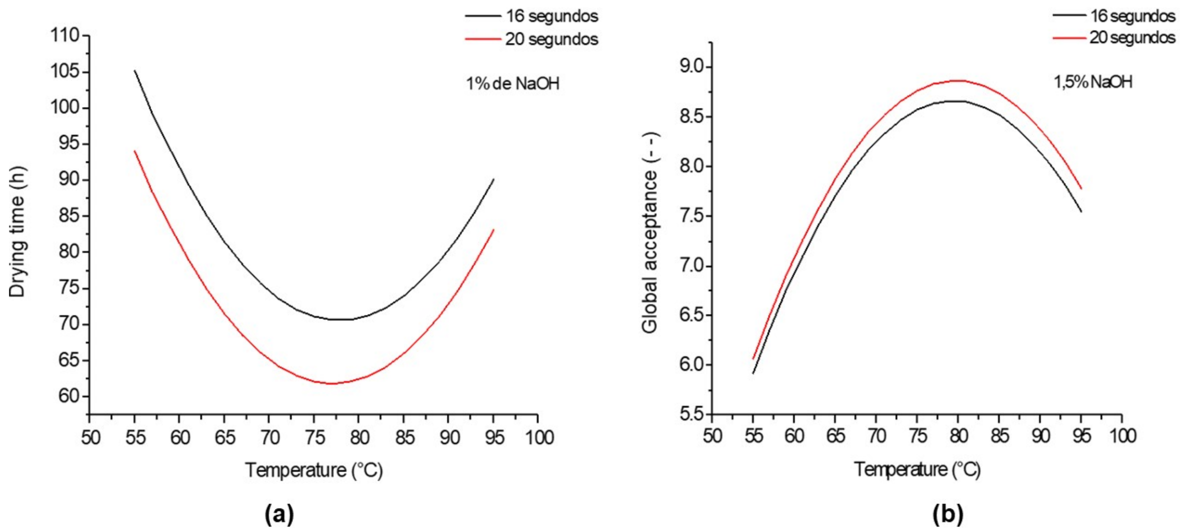

Figure 8. (a) Simulation of grape drying time versus temperature during sun drying (b) Simulation of overall raisin acceptance versus temperature during indirect solar drying

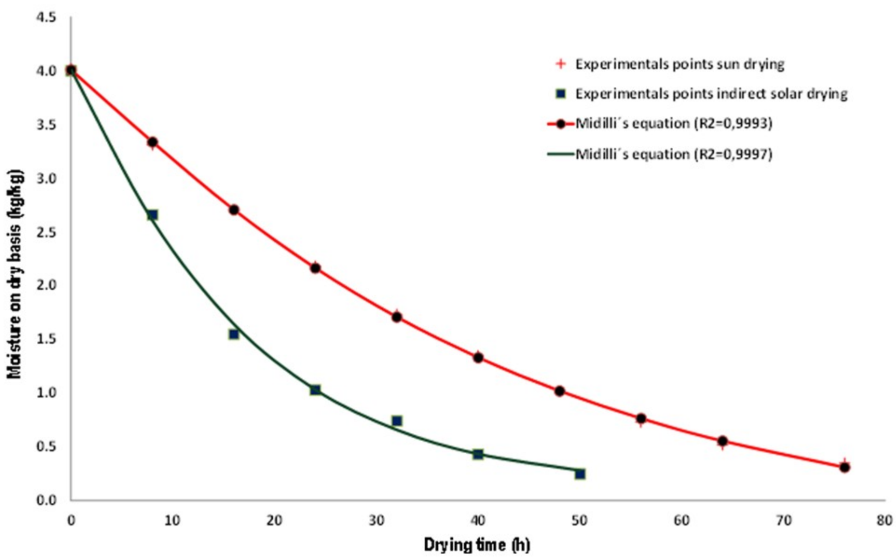

Figure 9. Evolution of moisture content versus drying time and mathematical modeling of the drying curve for the Muscat of Alexandria grape variety

Generally, the drying process consists of three consecutive phases: the initial phase, where the drying rate increases due to the temperature rise of the product and the consequent increase in vapor pressure; a second phase, where the drying rate is constant; and a final phase, characterized by a significantly reduced drying rate (Barbosa de Lima et al., 2016). These phases were observed in the drying of Muscat of Alexandria grapes pretreated with a NaOH solution (80 g/L) at a temperature of 25 °C for an immersion time of 360 seconds, where convective drying was carried out at a temperature of 30 °C for 13 days (Corona et al., 2016).

The first phase of the curve, corresponding to indirect solar drying, is due to microstructural changes in the epicuticular wax layer covering the grape cuticle, caused by pretreatment with NaOH; among these changes, the formation of cracks in the skin and the general softening of the tissue stand out, improving skin permeability and moisture diffusion (Lara et al., 2014). In phase two, the cracks gradually close as the grape shrinks and sugar from the pulp migrates toward the skin, where it acts as a barrier to water diffusion; this retarding effect can be overcome by increasing the drying temperature above 40 °C (Azzouz et al., 2002; Giordano et al., 2009; Toğrul, 2010). Meanwhile, the curve shown for sun drying exhibits almost a linear behavior, influenced only by the type of process. Thus, the solar dryer and the chemical-thermal treatment used in the research project reduced traditional drying time by 33 %, whereas Serratosa et al. (2008) achieved a 25 % reduction during chamber drying at 50 °C of Pedro Ximénez grapes pretreated in an alkaline medium.

The same figure shows the fit of the experimental data for moisture content versus drying time, using the equation by Midilli et al. (2002) to model the drying curve. For model selection, the following values were considered: the coefficient of determination (R^2), and error metrics including the sum of squared errors (SSE)—which served as the cell for the Solver—the root mean square error (RMSE), the mean relative percentage error (ERM), and the reduced chi-square value (χ^2) (Mehta, 2023).

It can be stated that the model by Midilli et al. (2002) best simulates the distribution of the experimental data for the drying curve of the Muscat of Alexandria grape variety, as it presented the highest values of the coefficient of determination and the lowest values of the calculated relative errors. The results are presented in Tables 10 and 11. Martín-Gómez et al. (2019) modeled the drying of whole Tempranillo grapes in a chamber at 50 °C with a relative humidity of 20 %. To do so, they evaluated the Newton, Henderson-Pabis, logarithmic, Wang and Singh, diffusion approximation, and Midilli et al. (2002) models, with the latter being selected based on the values of R^2, SSE, RMSE, EMR (%), and χ^2.

The mathematical model proposed by Midilli et al. (2002) is a suitable model for representing the drying behavior of grapes and tomatoes (Doymaz, 2004; Midilli et al., 2002; Yaldiz et al., 2001).

3.7.2 Drying kinetics

When plotting the drying rate of the Muscat of Alexandria grape variety against the average absolute humidity and drying time, as shown in Figure 10 (a and b), respectively, it is observed that the highest values of the drying kinetics correspond to the first 10 hours of drying at 0.18 and 0.08 kg/(kg·h), which correspond to dR/dt slopes of 0.250 and 0.253 for sun drying and indirect solar drying, respectively. Only the period of decreasing drying rate with a linear fit is observed in both processes.

3.7.3 Value of the effective water diffusivity

Using Equation 5, the slopes of $\ln(\mathrm{MR})$ versus time were determined to calculate the effective water diffusivity ($\mathrm{m^2/s}$). For the diffusivity calculation, the average grape radius of 0.009693 m was used. The values of the effective water diffusivity for the Alexandria Muscat grape were 8.57×10^{-11} and 1.90×10^{-10} $\mathrm{m^2/s}$ for sun-drying and indirect solar drying, respectively. The calculated values of effective water diffusivity fall within the desired range for food material drying, 10^{-12} and 10^{-8} $\mathrm{m^2/s}$ (Zhao et al., 2019). It was observed that the water diffusivity of the grapes was 2.2 times higher in the indirect solar dryer than in direct sun drying.

In general, pretreated grapes exhibit higher water diffusivity; in the case of whole, punctured grapes dried at 50 °C, an effective diffusivity value of 7.31×10^{-11} $\mathrm{m^2/s}$, and in whole, untreated grapes dried at the same temperature, their diffusivity reached 3.22×10^{-11} $\mathrm{m^2/s}$ (Martín-Gómez et al., 2019).

However, the calculated coefficient of determination values were 0.6933 and 0.7647 for the diffusive model of sun-drying and indirect solar drying, as shown in Fig. 10a and b, indicating a poor fit to the experimental data—a common issue in agroindustrial products due to shrinkage and transient moisture gradients that occur within the product during drying (Doymaz, 2007).

Table 10. Fitting parameters of the mathematical models for the drying curve of the Muscat of Alexandria.

Models	Parameters		R^2	SSE	RMSE	EMR (%)	χ^2
Henderson y Pabis	a k	1.0291 0.0290	0.99366	0.0091	0.0952	4.168	0.0113
Henderson-Pabis Modificado	a k b g c h	0.3716 0.0290 0.2859 0.0290 0.3716 0.0290	0.99367	0.0091	0.0952	4.803	0.0226
Page	k n	0.0154 1.1671	0.99890	0.0016	0.0398	1.212	0.00198
Page Modificado	k n	0.0218 1.2897	0.99190	0.0112	0.1060	6.503	0.01404
Newton	k a	0.0281 0.9972	0.99190	0.0112	0.1060	7.219	0.01248
Midilli *et al.*	k n b	0.0182 1.0938 -0.00068	0.99928	0.0006	0.0242	0.096	0.00098

Table 11. Fitting parameters of the mathematical models for the drying curve of the Muscat of Alexandria grape variety using an indirect solar dryer.

Models	Parameters		R^2	SSE	RMSE	EMR (%)	χ^2
Henderson y Pabis	a k	1.0060 0.0564	0.99797	0.0032	0.0567	0.048	0.00451
Henderson-Pabis Modificado	a k b g c h	0.0765 0.0646 0.8537 0.0552 0.0765 0.0646	0.99796	0.0032	0.0568	0.352	0.02259
Page	k n	0.0519 1.0256	0.99806	0.0031	0.0556	1.193	0.00433
Page Modificado	k n	0.2369 0.2369	0.99792	0.0033	0.0576	0.265	0.00464
Newton	k a	0.0561 1.0023	0.99792	0.0033	0.0576	0.455	0.00387
Midilli *et al.*	k n b	0.0841 1.0626 0.00038	0.99973	0.0028	0.0529	0.393	0.00652

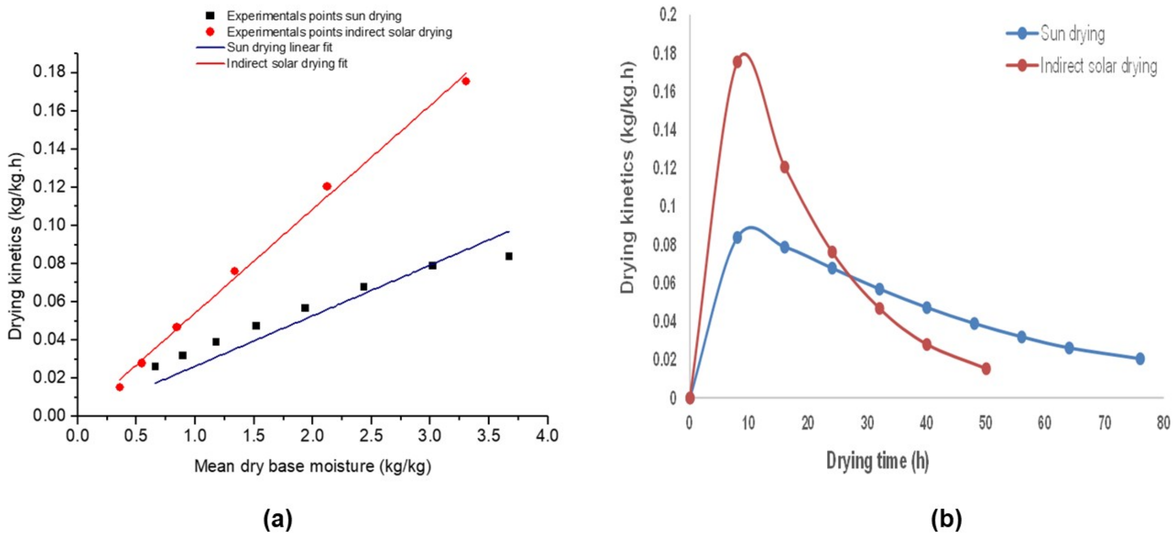

Figure 10. (a) Drying rate versus average moisture content in sun drying and indirect solar drying; (b) Drying rate versus drying time in sun drying and indirect solar drying of the Muscat of Alexandria grape variety

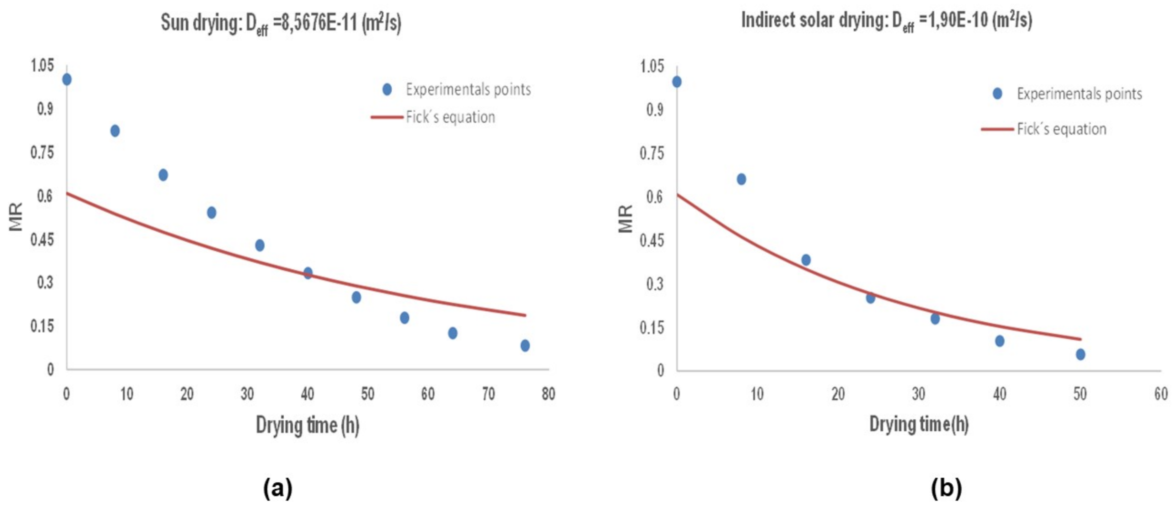

Figure 11. (a) Diffusional mathematical modeling of the sun-drying curve and (b) indirect solar drying curve for the Muscat of Alexandria grape variety

4 Conclusions

The drying of the Muscat of Alexandria grape variety (*Vitis vinifera*) was studied using two processes: sun drying and indirect solar drying using a dryer equipped with a large-surface-area solar collector. Prior to drying, an alkaline pretreatment was performed at temperatures ranging from 55–95 °C, with NaOH concentrations of 0.50–3.0 % and immersion times of 2–30 s. Sun drying and indirect solar drying were optimized, considering pretreatment conditions of 80 °C, 1.75 %, and 16 s to obtain approximate drying times of 75 h and 50 h, respectively, with a high overall product acceptance rate and a score above 8 points on the hedonic scale established for both processes.

Under optimal drying conditions, the mathematical model by Midilli et al. (2002) proved to be the model that best simulated the grape drying curve, as it presented the highest values for the coefficient of determination and the lowest values for the calculated relative errors. Effective water diffusivity values for the grapes were determined to be 8.57×10^{-11} and 1.90×10^{-10} m^2/s for direct sun drying and using an indirect solar dryer, respectively.

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Declaration on the use of Artificial Intelligence

The authors DECLARE that, during the preparation of the manuscript titled "Aplicación de un Modelo de superficie de respuesta en el secado al sol y solar indirecto de uvas variedad moscatel de alejandria (*Vitis vinifera*)", no generative artificial intelligence tools or automated assistance systems were utilized for the drafting, analysis, data interpretation, content generation, translation, or editorial refinement of the text.

The authors assume full responsibility for the content, originality, integrity, and final approved version of the manuscript.

Author Contributions

D.V.P.: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Supervision. **H.R.N.:** Conceptualization, Data curation, Formal analysis, Investigation, Methodology. **R.S.J.:** Conceptualization, Data curation, Formal analysis, Investigation, Methodology. **D.C.R.:** Conceptualization, Data curation, Formal analysis, Investigation, Methodology.

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