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Study on Drying of Black Rice (*Oryza sativa* L.) Grains: Physical-Chemical and Bioactive Quality

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Abstract

The present study aimed to assess the drying kinetics of black rice and fit different mathematical models (empirical and diffusive) to the experimental data, and evaluate the effect of drying air temperature on the physical-chemical and bioactive compounds quality of black rice. Drying air temperatures ranged from 40 to 80 ºC and the drying air speed was 1.5 m/s. Physical-chemical characterization of the product was based on the following parameters: moisture, water activity, ashes, total protein content, pH, total acidity, lipids, total carbohydrates, total anthocyanins, flavonoids, total phenolic compounds and antioxidant activity. Among the empirical models, Page showed the lowest mean squared deviations (MSD) and highest coefficients of determination (R^2). For the diffusion model, the values of effective mass diffusivity and convective heat transfer coefficient increased with increasing drying air temperature, and the Biot number indicated that the first-type boundary condition would also describe well the drying process. Physical-chemical parameters and bioactive compounds differed between the temperatures used, and the temperature of 60 ºC led to the best relationship between drying time and preservation of product characteristics.

Keywords: bioactive compounds, diffusivity, grains, quality

1. Introduction

Rice (*Oryza sativa* L.) is considered a staple food by a significant part of the world's population (Papillo et al., 2018). In the last years, grains of pigmented rice, such as black rice, have gained attention because this type of rice has benefits to health, due to bioactive pigments located in its bran layer, which contains higher content of phenolic compounds (Paiva et al., 2014; Vargas et al., 2018). In addition to γ-oryzodiol and vitamin C, there are also some water-soluble pigments which are responsible for its color and antioxidant properties (Hou et al., 2013; Norkaew et al., 2017). Black rice also contains a higher lipid content than non-pigmented and red rice, which makes it more palatable and attractive (Choi et al., 2019).

According to Ding et al. (2018), the enzymatic activity may increase and accelerate lipid degradation during storage, in addition to reducing the sensory quality of rice. Therefore, post-harvest techniques such as the drying process, which involves the reduction of seed moisture content to a safe level, can be applied to guarantee the preservation of physiological and physical-chemical quality of the product to be stored during a long period of time (Sousa et al., 2016). However, many of the properties of agricultural products are affected by the drying conditions (Dehghannya et al., 2016).

According to Silva et al. (2018), through the drying kinetics it is possible to determine the behavior of the dried material, representing it by drying curves and drying rates (Menezes et al., 2013). Several mathematical models have been used to describe the drying process of agricultural products and to determine process information which can be used in future equipment designs (Meneghetti et al., 2012). Under certain conditions (spherical or cylindrical geometries, infinite slabs and constant thermal-physical parameters and volume), the diffusion equation has an analytical solution (Luikov, 1968; Crank, 1992). These solutions are used to describe the thin-layer drying of various agricultural products, besides determining the effective mass flow diffusivity and convective mass transfer coefficient (Silva et al., 2010).

Therefore, the present study aimed to assess the drying kinetics of black rice at different drying air temperatures, fitting different mathematical models to the experimental data as a function of the moisture content, obtain the effective mass diffusivity from the analytical solution of the diffusion equation with third-type boundary condition considering the cylindrical geometry, besides characterizing and evaluating the effect of drying air temperature on the physical-chemical and bioactive compounds quality of black rice.

2. Material and Methods

2.1 Research Site and Acquisition of Grains

The study was conducted in the Laboratory of Storage and Processing of Agricultural Products (LAPPA) of the Federal University of Campina Grande (UFCG), Campus of Campina Grande, Paraíba (PB), Brazil. Black rice grains were purchased in a local store of the city of Campina Grande-PB, and then visually selected for sampling uniformity.

2.2 Drying Kinetics

Black rice grains were dried in triplicate in a forced air circulation oven regulated to operate at temperatures of 40, 50, 60, 70 and 80 ºC, with air speed of 1.5 m/s. Samples were uniformly distributed on steel screen trays, forming a thin layer (Figure 1).

Figure 1. Black rice grains

The experimental data were expressed in terms of moisture content ratio (RX), given by the relationship between the differences in moisture content over time, t, and the equilibrium moisture content (X-Xe) and the initial and equilibrium moisture contents (X_0-Xe), as described in Equation (1),

$$RX = \frac{\left(X - X_e\right)}{\left(X_0 - X_e\right)} \tag{1}$$

where, RX = moisture content ratio (dimensionless); X_e = equilibrium moisture content (dry basis); X = moisture content (dry basis); X_o = initial moisture content (dry basis).

Three empirical models (with up to three parameters) were fitted to the values observed for each drying air temperature (Table 1), by the Quasi-Newton method, using the program Statistica 7.0 (Statsoft Co, 2007).

Table 1. Empirical models used to predict the drying phenomenon of black rice

Models	Equation
Page	RX = exp(-k·t^n)
Henderson & Pabis	RX = a·exp(-k·t)
Parry	RX = a·exp(-k·t) + c

Note. t: drying time (min); k: drying constant; a, c, n: coefficients of the models.

The fitting of each model was evaluated based on the coefficient of determination (R^2) and Mean Squared Deviation (MSD), calculated by Equation (2):

$$MSD = \sqrt{\frac{\left(RX_{\exp} - RX_{pre}\right)^2}{N}} \tag{2}$$

where, RX_{exp} = moisture content ratio (dimensionless moisture content) obtained experimentally; RX_{pre} = moisture content ratio predicted by the mathematical model; N = number of observations along the drying kinetics.

2.3 Diffusion Equation

The diffusion equation which describes the drying of a product in the form of an infinite cylinder can be written for the dimensionless moisture content as:

$$\frac{\partial RX}{\partial t} = \frac{1}{r}\frac{\partial}{\partial r}\left(rD\frac{\partial RX}{\partial r}\right) \tag{3}$$

where, D is the effective mass diffusivity and r defines a position inside the cylinder relative to its axial axis. Since RX is the moisture ratio, it should be noted that its initial value is 1 and the equilibrium value is zero. In the present study, an analytical solution of Equation (3) was used to describe moisture diffusion in cylindrical bodies.

2.4 Analytical Solution for the Convective Boundary Condition

The third-type boundary condition, or the Cauchy boundary condition, is expressed by the imposition of equal internal diffusive flow at the limit of the infinite cylinder and external convective flow close to this limit, given by Equation (4),

$$-D\left.\frac{\partial RX\left(r,t\right)}{\partial r}\right|_{r=R} = h\,RX\left(r,t\right)\Big|_{r=R} \tag{4}$$

where, h is the convective transfer coefficient; $RX(r, t)$ is the moisture ratio at the radial position r and time t; and R is the radius of the infinite cylinder.

The average moisture ratio for a cylindrical solid at time t is given, for the first 16 terms of the series (rather than infinite terms), by (Luikov, 1968; Crank, 1992),

$$RX\left(t\right) = \sum_{n=1}^{16} B_n \exp\left(-\mu_n^2 \frac{D}{R^2} t\right) \tag{5}$$

where, the parameter B_n is given by,

$$B_n = \frac{4Bi^2}{\mu_n^2\left(Bi^2 + \mu_n^2\right)} \tag{6}$$

where, Bi is the Biot number, given by,

$$Bi = \frac{hR}{D} \tag{7}$$

where, h is the convective mass transfer coefficient; R is the cylinder radius; D is the effective mass diffusivity. In Equation 6, μ_n represents the roots of the transcendental equation,

$$\frac{J_0\left(\mu_n\right)}{J_1\left(\mu_n\right)} = \frac{\mu_n}{Bi} \tag{8}$$

where, J_0 is the first-type zero-order Bessel function and J_1 is the first-type first-order Bessel function.

To obtain the analytical solution, the process was optimized using the program "Convective" (F. A. S. Silva & C. A. V. Silva, 2008). The program Convective is used to study water diffusion processes with known experimental data, for the following geometries: infinite slab, infinite cylinder, sphere, finite cylinder and parallelepiped.

2.5 Physical-Chemical and Bioactive Compounds Characterization of Fresh and Dehydrated Grains

Black rice grains, both fresh and after each drying process, were characterized for physical-chemical parameters and bioactive compounds based on: moisture content, determined by drying in oven at 105 ºC until constant weight; water activity (Aw), determined using a Decagon® Aqualab CX-2T device at 25 °C; ashes, determined by incineration in muffle furnace; total protein content, quantified by the Micro-Kjeldahl method, which consisted in determining total nitrogen and converting the result into protein using the 5.95 factor, recommended for cereal proteins, according to the methodology described by Brasil (2008); pH, determined by direct reading in a digital pH meter; total acidity, determined by titrimetry, according to Brasil (2008); and lipids, determined by the method of Bligh and Dyer (1959). The total carbohydrate content was calculated by difference to obtain 100% of the total composition (FAO, 2003).

The contents of total anthocyanins and flavonoids were determined by the single pH method described by Francis (1982). This method consists in a quantitative transfer of an aliquot of the concentrated extract to a container and, subsequently, diluting this aliquot in a quantity of Ethanol—HCl at 1.5 mol L^{-1}, thus creating a diluted volume of extract. Total phenolic compounds were quantified by the Folin-Ciocalteau method described by Waterhouse (2006), using gallic acid as standard. The calculations performed to determine the phenolic compounds were based on a standard curve of gallic acid, and the readings were taken in spectrophotometer at 765 nm, with the results expressed in mg 100 g^{-1} of gallic acid equivalent. Antioxidant activity was determined using the method proposed by Re et al. (1999), with modifications made by Rufino et al. (2007).

2.6 Statistical Analysis

The experimental data were analyzed in triplicate and the results were subjected to single-factor analysis of variance (ANOVA) at 0.05 probability level, and the significant qualitative responses were subjected to Tukey test also at 0.05 probability level. All statistical analyses were carried out using the program Assistat 7.7 (Silva & Azevedo, 2016).

3. Results and Discussion

It is interesting to observe that temperatures above 70 ºC caused cracks in the grains. On the other hand, the drying time decreased proportionally to the increase in drying air temperature, ranging from 450 to 810 minutes, which were respectively found at the temperatures of 80 and 40 °C. Such behavior occurs because the highest rates of water removal from the product occur at the highest temperatures, which consequently reduces the drying time. Table 2 presents the values obtained for the fitting parameters of the drying kinetics and it can be observed that for Page, Henderson and Pabis and Parry models, the drying constant (k) increased with the drying temperature. For these same models, the parameters “a” and “n” were not influenced by the temperature, but for Parry model, like the drying constant (k), the parameter “c” was influenced by the temperature, showing direct relationship with the increase in temperature.

It can be observed that all models showed coefficients of determination (R^2) above 98%, but only the coefficients higher than 99% were considered as satisfactory fits. Therefore, the mathematical model of Page had high values of coefficient of determination (R^2) at all drying temperatures, ranging from 0.99665 (40 °C) to 0.99904 (80 °C). The mean squared deviation (MSD) varied from 0.01291 to 0.04495, but the mathematical model which showed the lowest and best values was the Page model, which obtained MSD from 0.01291(80 °C) to 0.03227 (40 °C). Lang et al. (2018) studied the drying kinetics of black rice using fixed-bed dryer with air speed of 0.5 m/s and also concluded that the Page model was the best one to describe the drying kinetics.

Table 2. Fitting parameters of drying kinetics of black rice, coefficient of determination (R^2), and mean squared deviation (MSD) for the mathematical models at the temperatures of 40, 50, 60, 70 and 80 °C

Models	T (°C)	Fitting parameters				R^2	MSD
		a	k	n	c		
Page	40	-	0.023707	0.73426	-	0.99665	0.03227
	50	-	0.032730	0.70469	-	0.99769	0.02159
	60	-	0.035665	0.70877	-	0.99785	0.02041
	70	-	0.051316	0.68219	-	0.99844	0.01701
	80	-	0.067378	0.65819	-	0.99904	0.01291
Henderson & Pabis	40	0.88710	0.00496	-	-	0.99526	0.04495
	50	0.87478	0.00627	-	-	0.99388	0.03513
	60	0.87921	0.00745	-	-	0.99279	0.03735
	70	0.87795	0.01087	-	-	0.98785	0.04740
	80	0.87156	0.01406	-	-	0.98562	0.04974
Parry	40	0.884314	0.00502	-	0.00386	0.99527	0.04482
	50	0.858236	0.00683	-	0.02446	0.99429	0.03394
	60	0.860448	0.00818	-	0.02789	0.99330	0.03602
	70	0.850637	0.01296	-	0.04813	0.99019	0.04262
	80	0.841938	0.01726	-	0.05582	0.98929	0.04275

Among the models analyzed, Page model fitted best to the experimental data because it showed highest values of coefficient of determination R^2 (> 0.99665) and lowest values of MSD (< 0.0322) for the studied temperatures (Figure 2).

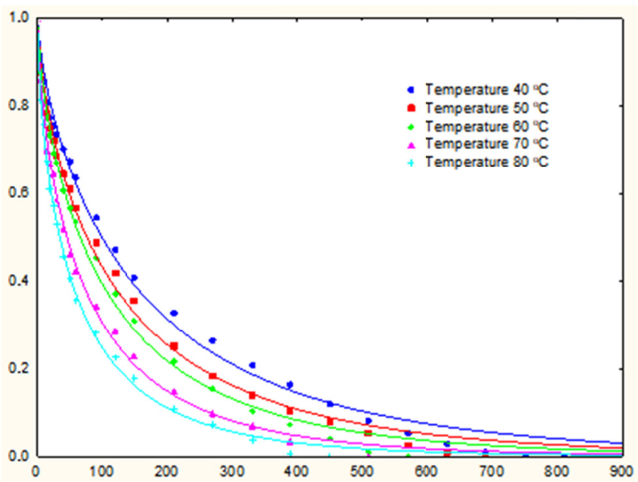

Figure 2. Drying curves (dimensionless moisture content versus time in minutes) of black rice grains determined by the Page model at temperatures of 40, 50, 60, 70 and 80 °C

Since the average value of the radius of the rice grains was 1.73 mm, the values of effective mass diffusivity were calculated (Table 3), and these values increased from 2.24 to 5.21 × 10^{-9} m^2 min^{-1} when the drying air temperature increased from 40 °C to 80 °C. This phenomenon also occurred for the convective heat transfer coefficient, which increased from 3.75 to 60.8 × 10^{-5} m min^{-1}. The Biot number did not show direct relationship with the increase of temperature, since the highest number was obtained at the temperature of 80 °C, indicating that the first-type boundary condition would also describe well the drying process.

Table 3. Results obtained by optimization using the analytical solution

Temperature (°C)	($D_{ef} \times 10^9$ m^2 min^{-1})	(h × 10^5 m min^{-1})	Biot Number
40	2.24	3.75	29
50	2.75	7.65	50
60	3.14	7.82	42
70	4.62	19.9	77.5
80	5.21	60.8	200

According to Lang et al. (2018), diffusivity corresponds to the magnitudes of the drying rates affected by the drying temperature. Thus, the highest drying rates showed the highest values of diffusivity. Silva et al. (2014), evaluating the diffusivity of pigeon pea grains at temperatures from 40 to 70 °C, found values ranging from 2.1 to 6.8 × 10^{-10} m^2 s^{-1}. For red rice grains, Sousa et al. (2016) obtained values which varied from 2.33 to 6.45 × 10^{-11} m^2 s^{-1}.

However, the higher the value of diffusivity, the higher the facility for water molecules to be removed from the product, causing water to be more bound to the molecules constituting the dry mass. High values of heat transfer coefficient tend to lead to shorter time to reach equilibrium moisture. Johann et al. (2015) in studies with the thin-layer drying of grape grains at temperatures from 50 to 80 °C, obtained convective heat transfer coefficients from 4.11 to 21.54 × 10^{-5} m min^{-1}.

The results obtained in the characterization physical-chemical parameters and bioactive compounds of black rice grains before the drying process are described in Table 4.

Table 4. Physical-chemical and bioactive characterization of black rice grains before drying

Parameters	Mean and Standard deviation
Moisture (%, w.b.)[1]	12.75±0.63
Water activity (A_w)	0.648±0.02
pH	6.50±0.07
Total acidity (%)	0.030±0.01
Ashes (%)	1.97±0.15
Proteins (%)	8.66±0.12
Lipids (%)	2.93±0.06
Carbohydrates (%)	73.69±0.30
Total anthocyanins (mg 100 g^{-1})	70.20±0.69
Flavonoids (mg 100 g^{-1})	38.48±0.84
Total phenolic compounds (mg GAE 100 g^{-1})	289.93±1.12
Antioxidant activity[2] (μmol Trolox g^{-1})	209.20±0.98

Note. [1]wet basis. [2]ABTS$^+$.

The black rice showed moisture content of (12.75%), which is close to the value found by Ziegler et al. (2017) for black rice (13.7%) and higher than the value found by Becker-algeri et al. (2017) for whole rice (10.7%). According to Matos (2014), pigmented rice grains tend to show different characteristics from those of non-pigmented rice, such as higher contents of moisture and protein. In addition, water activity in the present study was 0.648, which characterizes the grains as of intermediate moisture. Oliveira (2016) classifies as products of intermediate moisture those which have water activity between 0.6 and 0.85.

Similar values of pH (6.67) and total acidity (0.05%) were verified by Alencar et al. (2017), characterizing black rice grains. For ash content, Marquez (2013) obtained values between 1.5 and 1.8% in black rice grains and between 1.3 and 1.7% in red rice grains, but the ash contents considering both types of pigmentation were similar to those found in the present study.

Based on the values of proteins, lipids and carbohydrates, as observed in Table 4, black rice grains can be considered as of high protein content, with value close to those found also in black rice by Paiva et al. (2014) and Ziegler et al. (2017), respectively 8% and 9.8%. Ziegler et al. (2017), also evaluating lipid and carbohydrate contents in black rice grains, obtained higher lipid content (4.4%) than that of the present study and lower carbohydrate content (70.5%), but also close to the one found in the present study, thus demonstrating that black rice grains are highly rich in total fibers, since the analysis of total carbohydrate includes the contents of fiber.

The content of total anthocyanins was (70.2 mg 100 g^{-1}) and that of flavonoids was (38.38 mg 100 g^{-1}). Abdel-Aal et al. (2018) studied purple wheat grains and obtained a variation of 13.7-57.4 mg 100 g^{-1} for anthocyanin contents, evidencing the technological potential of black rice grains.

Black rice grains had phenolic compounds content of (289.93 mg GAE 100 g^{-1}) and antioxidant activity of (209.20 μmol Trolox g^{-1}). These values of total phenolic compounds were similar to those found by Min et al. (2012) (240 to 540 mg GAE 100 g^{-1}) and lower than those reported by Chen et al. (2012) (400 to 650 mg GAE 100 g^{-1}), both for black rice grains. However, these variations may be related to differences in grain cultivation and extraction methods. The results obtained in the present study demonstrate that black rice grains have significant contents of total phenolic compounds with high antioxidant activity.

According to Braga (2013), quality parameters undergo changes during the drying process. Drying with hot air leads to reduction in nutritional values, besides altering texture, color and causing a slow or incomplete dehydration of the material (Nascimento et al., 2018). The results of physical-chemical and bioactive analyses at drying air temperatures of 40, 50, 60, 70 and 80 ºC are presented in Table 5.

Table 5. Physical-chemical and bioactive characterization of black rice grains after each drying process

Parameters	Drying temperatures				
	40 ºC	50 ºC	60 ºC	70 ºC	80 ºC
Moisture (% w.b.)	11.55 a	8.17 b	6.54 c	4.68 d	3.23 e
Water activity (A_w)	0.321 a	0.183 b	0.165 c	0.156 d	0.121 e
pH	6.69 b	6.70 ab	6.72 a	6.70 ab	6.70 ab
Total acidity (%)	0.029 a	0.027 a	0.028 a	0.028 a	0.029 a
Ashes (%)	3.83 d	4.64 c	5.68 b	6.51 a	6.88 a
Proteins (%)	7.35 a	6.99 a	5.87 b	4.67 c	3.21 d
Lipids (%)	2.95 d	3.05 d	3.22 c	3.61 b	3.89 a
Carbohydrates (%)	74.32 e	77.15 d	78.69 c	80.53 b	82.79 a
Anthocyanins (mg 100 g^{-1})	67.19 a	65.62 b	64.33 c	63.19 d	62.01 e
Flavonoids (mg 100 g^{-1})	29.98 a	29.66 b	29. 42 c	29.29 d	29.14 e
Total phenolic compounds (mg GAE 100 g^{-1})	268.55a	258.87 b	244.23 c	233. 67 d	231.14 d
Antioxidant activity[2] (μmol Trolox g^{-1})	178.80 a	142.16 b	117.12 c	73.32 d	50.92 e

Note. Equal lowercase letters in the same row do not differ significantly between the studied temperatures by Tukey test at 0.05 probability level. [1]wet basis. [2]$ABTS^{+}$.

There was a clear reduction in the moisture content of black rice grains as the temperature increased, which was expected because free water evaporated as the grains were subjected to heat, with variation from 11.55 (40 ºC) to 3.23 (80 ºC). This same parameter shows significant statistical difference when its values are compared between the different drying temperatures. In order to ensure the time of preservation and guarantee quality, it is necessary to know the water activity. In black rice grains, its behavior was similar to that of moisture contents, showing significant difference, with highest value (0.321) for the temperature of 40 ºC and lowest value (0.121) for the temperature of 80 ºC.

The values obtained for pH are close to neutrality (pH = 7.00) and ranged between 6.60 and 6.72. Significant statistical difference in pH values was observed between the temperatures of 40 and 60 ºC, but there was no statistical difference between the temperatures of 50, 70 and 80 ºC at 0.05 probability level. For total acidity, the values obtained for all temperatures did not differ significantly; there was a small variation of up to 0.002% between treatments, with no influence of temperature on this parameter.

The ash contents did not differ significantly between the temperatures of 70 and 80 ºC. Its increase was proportional to the increase of temperature, and the sample subjected to drying at 80 ºC had the largest amount of ashes (6.88%). By comparing the ash contents in fresh grains (Table 4) and in dehydrated grains (Table 5), it was possible to note a 4.91% gain of mineral salts. However, the opposite was observed in the protein content, where the increase of temperature led to protein degradation; grains subjected to temperatures of 40 and 50 ºC did not differ statistically, but those dried at 60, 70 and 80 ºC were statistically different at 0.05 probability level. Consequently, the lowest protein content was obtained in grains subjected to drying at 80 ºC (3.21%), showing a reduction of 5.45% compared to grains not subjected to the drying process.

The same pattern observed for ash content occurred for lipid content but, as the drying temperature increased, there was a slight increase of lipid content in the grains. According to Table 5, there was no difference between the drying temperatures of 40 and 50 ºC but, as the temperature increased, there was an increase of up to 0.96% in the lipid content of the grains, compared to those presented in Table 4. Carbohydrates were determined by difference based on the other constituents and, therefore, the reduction of moisture automatically led to an increase of up to 8.47% in carbohydrate content between the applied temperatures. Nevertheless, the values obtained for each treatment were significantly different from one another.

In relation to the contents of anthocyanins and flavonoids, there was significant difference between the temperatures applied, and these pigments decreased by up to 5.18 mg 100 g^{-1} and 0.84 mg 100 g^{-1}, respectively. According to Modesto Júnior et al. (2016), several factors interfere with anthocyanin stability, including pH, action of oxygen, enzymes, temperature variation and light incidence.

The contents of total phenolic compounds in the grains did not differ significantly between the drying temperatures of 70 and 80 ºC, but differed from those found in grains subjected to temperatures of 40, 50 and 60 ºC. This parameter varied from 231.14 to 268.55 mg GAE 100 g^{-1} and these values were respectively found at temperatures of 80 and 40 ºC.

For antioxidant activity, it can be observed that the different temperatures applied led to degradation of antioxidant compounds, which, for being unstable natural compounds, underwent significant alterations. The antioxidant activity of black rice grains decreased as the drying air temperature increased, and there was a reduction of up to 158.91 µmol Trolox.g^{-1} at the temperature of 60 ºC, compared to the antioxidant activity of fresh grains (Table 4).

4. Conclusion

The study on the drying kinetics of black rice demonstrated that increasing temperature led to reduction in drying time, and temperatures above 70 ºC caused cracks in the grains. In the fitting of mathematical models to the experimental data, as a function of moisture content, the Page model showed the lowest values of MSD and highest coefficients of determination (R^2). Obtaining the results of the diffusion equation by optimization through an analytical solution allowed observing that the highest values of diffusivity and convective heat transfer coefficient occurred at the temperature of 80 ºC, indicating higher facility for water molecules to be removed from the grains. The Biot number indicated that the first-type boundary condition would also describe well the drying process. There was a significant difference between the temperatures used with respect to the physical-chemical parameters and bioactive compounds, and the temperature of 60 ºC led to the best relationship between drying time and preservation of the characteristics of black rice grains.

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