Acta Scientiarum

http://periodicos.uem.br/ojs/acta
ISSN on-line: 1807-8672
Doi: 10.4025/actascitechnol.v47i1.69773

SCIENCE, FOOD TECHNOLOGY AND FOOD ENGINEERING

Black oat, rye, and wheat cereals: kinetics and changes during hydrothermal processing

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ABSTRACT. The grain hydration process is fundamental in cereal processing but can modify their physical and chemical properties. However, there is a gap in understanding the principles of mass transport (water) and its changes during the hydration phases, especially in rye, which has yet to be studied. A kinetic study was carried out to investigate moisture transfer and changes in starch during oats, rye, and wheat hydration. During the process it was observed that the mass transfer was directly affected by the system's temperature, which indicates changes in the absorption capacity over time. The tracer pigment indicated that water entered the grains by diffusion through the grain coating and capillarity through the fissures. Thermal analyses revealed that the starch remained unchanged at hydration temperatures below 50°C. Mathematical models describe the moisture gain in the different conditions studied, while the generalized model allows the experimental moisture to be predicted. This study contributes to understanding the time/temperature relationship in cereal hydration by presenting a kinetic model and examining changes in starch. This provides valuable insights to optimize the industrial operation of this stage and guides new project development.

Keywords: gelatinization; water uptake; water diffusion; hydration rate.

Received on September 27, 2023.
Accepted on April 11, 2024.

Introduction

Cereals are essential for human nutrition and must be stored dry for preservation, so they must be rehydrated before processing or consumption (Miano & Augusto, 2018). This hydration step is essential for germination, cooking, component extraction, fermentation, and malting, among others (Oladele, Osundahunsi, Agbetoye, & Augusto, 2018).

Mathematical modeling is a fundamental tool in understanding the hydration process, where, through empirical and phenomenological models, the water absorption behavior in the grain can be explained (Marques, Jorge, & Jorge, 2014; Montanuci, Jorge, & Jorge, 2014; Nicolin, Jorge, & Jorge, 2015). From this understanding, possibilities can be evaluated for the intensification of the process, and can be used as a basis for cereal processing industries.

When choosing the model, one must consider the fit of the equation to the experimental data and the ease of execution (Kashiri, Kashaninejad, & Aghajani., 2010; Turhan, Sayar, & Gunasekaran, 2002; Yildirim, Öner, & Bayram, 2010). With these premises, for this study, the following was used: Peleg's empirical model due to its good fit and wide use; the phenomenological model of Omoto-Jorge, for its non-complex application; the Nicolin-Jorge model, which is based on the previous model, but considers that the mass transfer coefficient varies linearly with moisture; and phenomenological model of diffusion, because it is widely used and with good results.

The Peleg model is the most used in the hydration of food products, even though it is an empirical equation with only two parameters, k_1, which is equivalent to the hydration rate, and k_2, which is the equilibrium moisture content (Peleg, 1988). The Omoto-Jorge model (Omoto et al., 2009) is based on group parameters in which the mass transfer coefficient was considered constant, has an analytical solution, and presents a satisfactory fit for grain hydration modeling; it is a phenomenological model developed from the transient mass balance for water inside the grain (Balbinoti, Jorge, & Jorge., 2018a, 2018b; Chacón Alvarez, Jorge, & Jorge, 2020; Mattioda, Jorge, & Jorge, 2019a; Silva, Jorge, & Jorge et al., 2019). The Nicolin-Jorge model (Nicolin, Neto, Paraíso, Jorge, & Jorge, 2015) considers the linear dependence of the mass transfer coefficient of the Omoto-Jorge model, taking into account that this coefficient tends to zero in the steady state. The diffusivity, determined by a specific model, represents the amount of water diffused by an area subject to a concentration gradient (Crank, 1975), allowing to relate experimental data to the laws of physics (Mattioda, Jorge, & Jorge, 2018).

With these premises, this study focused on understanding the hydration kinetics of cereals and their changes in grains, starches, and how much the binomial temperature and time influence the absorption of water and, consequently, its optimization. These data will serve as basis for the oat, rye, and wheat processing industries, both for process calculations, and for the development of worksites.

Material and methods

Material

The samples used in this study, from the 2019 harvest, were rye, cultivar IPR 89, category S2, donated by COPERCAMPOS, located in the State of Santa Catarina, Brazil (latitude -27.34237, longitude -51.21059), black oats, cultivar EMBRAPA and cultivar Supera wheat, donated by Alvorada Farm, located in the State of Paraná, Brazil (latitude -25.15767, longitude -50.53942).

Starch extraction for thermogravimetric analysis

The grains (100 g) were ground (Marconi Knife Grinder, MA 630/01/Brazil), suspended in distilled water in a proportion of 1:2 (grains: water), and the mixture was sieved (150 mesh), suspending the filtrated in 100 mL of water for 12 hours under refrigeration (4°C ± 1). The supernatant was discarded and the decanted fraction was again suspended in 100 mL of distilled water, the mixture was centrifuged, separating the starch, the supernatant was discarded again, and the starch mass was dried for 24 hours in an oven with air circulation at 38°C (Nova Ética, model 400/6ND, Brazil). The dried material was ground with a pestle, sieved (200 mesh), and stored in a desiccator until analysis (Bultosa, Hall, & Taylor, 2002).

Thermal characterization of starch

The gelatinization of the extracted starch was characterized using differential scanning calorimetry (DSC 8500, Perkin Elmer, EUA). The starch was suspended in water (1 starch:4 distilled water), homogenized with vortex, and inserted into a 50 μL aluminum pan, hermetically closed. The heating rate was 10°C min.$^{-1}$ between temperatures from 25 to 120°C. The event temperatures and enthalpy changes were identified with the help of Pyris software (PerkinElmer Inc.; EUA, Version 11.1).

Starch mass degradation occurred by thermogravimetric analysis (TGA) (TGA-4000 by Perkin Elmer), with a temperature between 30 and 600°C under a heating rate of 10°C min.$^{-1}$ with the use of nitrogen gas, making the atmosphere inert. The generated data were evaluated in the Origin Pro 2018 software.

Hydration experiments

The hydration experiment was carried out by heating 100 g of grains in 300 ml of distilled water at 10, 20, 30, 40, and 50°C ± 0.2, in a thermostatic bath (TECNAL/model TE/184/Brazil), with the sampling in triplicates taking place every 30 minutes in the first 3 hours and after 13 hours, between these periods the collection took place every hour. The moisture content of the hydrated samples was determined by gravimetry (ESTUFA CB Retilinia-002/2-Brasil) at 105°C until the sample reaches constant weight (Association of Official Analytical Collaboration [AOAC], 1995).

Water absorption behavior during the hydration process

A tracer was used, a molecule that behaves like the molecule of interest, which in this case is water. For this purpose, methylene blue in water (0.15 g 100 mL^{-1}) was chosen. In this solution mixture, grains were added in a 1:3 ratio (grain: water with dye) and kept for 17 hours at 50°C in a thermostatic bath (TECNAL/model TE/184/Brazil). The grains were sampled at different times and cut longitudinally with a scalpel for image acquisition.

Leached solids

The leached solids result from the interaction of immersion water with hydrophilic molecules within the grain matrix, allowing a kinetic evaluation of the release. For quantification, the hydration medium was evaluated in triplicate, by refractometry analysis (RL3, PZOG Warszawa). The results expressed in °Brix, and the measurement times were the same used in the hydration experiments. To evaluate de solid release kinetic was used the kinetic zero-order model (Equation 1 and 2) (Sayar, Turhan, & Köksel, 2011).

$$\frac{dM_{ds}}{dt} = k_{ds} \quad (1)$$

$$M_{ds} = k_{ds}t \quad (2)$$

Where M_{ds} is the amount of solid released, k_{ds} is the solids release rate constant, and t is the time.

Mathematical modeling of the hydration process

Peleg (1988) developed an empirical model that describes the phenomenon of water absorption in grains (Equation 3).

$$\mathrm{M(t)} = \mathrm{M_0} + \frac{\mathrm{t}}{(\mathrm{k_1}+\mathrm{k_2}.\mathrm{t})} \quad (3)$$

Where M(t) is the moisture on a wet basis (%) as a function of time t (min.); M_0 is the initial grain moisture content; and the model parameters k_1 (min.%-^{1}) and k_2 (%-^{1}), are respectively related to the mass transfer rate and the maximum water absorption capacity. The equilibrium moisture content (M_e) can be determined by considering $t \rightarrow \infty$ and obtaining a relationship between M_e and k_2.

The Diffusion model (Fick, 1855) is a phenomenological model used to describe moisture absorption in the hydration of grains. Limitations due to geometry, isotropy, volume, and capillarity changes are disregarded (Crank, 1975; Miano & Augusto, 2018) (Equation 4).

$$\frac{\partial \mathrm{M}}{\partial \mathrm{t}} = \mathrm{D}\left(\frac{\partial^2 \mathrm{M}}{\partial \mathrm{r}^2} + \frac{2}{\mathrm{r}}\frac{\partial \mathrm{M}}{\partial \mathrm{r}}\right) \quad (4)$$

Where D is the diffusion coefficient (m^2/s), M is the moisture ($\%_{wb}$), r is the radius (m), and t is the time (s). To obtain an analytical solution, the following will be considered: the spherical shape of the grain; diffusion will be independent of water concentration; the increase in grain volume by hydration will not be considered; the surface will instantly reach equilibrium moisture; and the process will be controlled only by the diffusion mechanism.

The Omoto-Jorge model (Omoto et al., 2009) used the experimental values of moisture on a wet basis (M_{wb}) and grain density (ρ_g), allowing the estimation of water concentration in grains (ρ_A) (Equation 5).

$$\rho_{\mathrm{A}} = \mathrm{M_{wb}}\rho_{\mathrm{g}} \quad (5)$$

The variation of the water mass in the grain ($\rho_A V$) as a function of time (t) is determined as a function of the mass water flow (N_A) (g cm^2 $min.^{-1}$) and the area (A) (Equation 6).

$$\frac{\mathrm{d}\rho_{\mathrm{A}}}{\mathrm{dt}} = \mathrm{N_A}.\mathrm{A} \quad (6)$$

Equation 6 is the basis of the Omoto-Jorge model and was obtained from the mass balance in the transient state of hydration. For this purpose, homogeneous water concentration, constant volume, and spherical geometry were considered. Equation 7 defines N_A and can be applied to Equation 6, generating Equation 8.

$$\mathrm{N_A} = K_S(\rho_{\mathrm{Aeq}} - \rho_{\mathrm{A0}}) \quad (7)$$

$$\frac{\mathrm{d}(\rho_{\mathrm{A}}\mathrm{V})}{\mathrm{dt}} = \frac{3K_S}{\mathrm{r_0}}(\rho_{\mathrm{Aeq}} - \rho_{\mathrm{A0}}) \quad (8)$$

The Omoto-Jorge model (Omoto et al., 2009) presents the following parameters: coefficient of mass transfer by convection, represented by K_S (cm $min.^{-1}$), and the concentration of water in the grain at equilibrium, ρ_{Aeq} (g mL^{-1}) as a function of time t (min.), r_0 (cm) is the initial radius, and ρ_{A0} (g mL^{-1}) is the water concentration in the grain at the initial time. Based on other studies (Balbinoti et al., 2018b, 2018a; Chacón Alvarez et al., 2020; Mattioda et al., 2019a; Silva et al., 2019), the geometry of the grains was considered spherical.

The Nicolin-Jorge model (Nicolin, Marques, Balbinoti, Jorge, & Jorge, 2017) considered the linear dependence of the mass transfer coefficient of moisture (Equation 9).

$$\frac{d(\rho_A)}{dt} = \frac{A}{V}(a - b\rho_{A0})(\rho_{Aeq} - \rho_{A0}) \tag{9}$$

Where A is the surface area of the grain (cm^2), V is the volume (cm^3), ρ_{A0} (g cm^{-3}) is the water concentration in the grain at the initial time, ρ_{Aeq} (g cm^{-3}) is the water concentration at equilibrium. When comparing Equation 8 with 9, it can be seen that K_S was considered a linear function; with this, a (cm min.$^{-1}$) and b (cm^4 g^{-1} min.$^{-1}$) are constants. Considering that moisture is uniformly distributed throughout hydration, the solution obtained is (Equation 10):

$$\rho_A(t) = \frac{-a + \rho_{eq} K_1 \exp(K_2 t)}{b + K_1 \exp(K_2 t)} \tag{10}$$

The constants K_1 (cm^4 g^{-1} min.$^{-1}$) and K_2 (min.$^{-1}$) can be defined according to Equations 11 and 12, remembering that the spherical geometry of the grains is considered.

$$K_1 = \frac{a + b\rho_{A0}}{\rho_{eq} - \rho_{A0}} \tag{11}$$

$$K_2 = \frac{3}{r}(a + b\rho_{eq}) \tag{12}$$

The models will be analyzed and the one which presents the best fit between the experimental and predicted data will be generalized.

Analysis of fit of mathematical models

The parameters of the models were obtained by nonlinear regression using the Levenberg–Marquardt algorithm software Origin 8.6. Regression analysis was carried out for each replicate, thus obtaining mean value and standard deviation. The quality of the set of equations was evaluated according to the magnitude of the coefficient of determination (R^2), average relative error (P), and the square root of the average error (RMSE).

Results and discussion

Extraction and thermal characterization of starch from raw grains

Starch is the main constituent of the endosperm of grains, being the most used part in the flour production and an important component in the malt production. By extracting the starch from raw grain by an aqueous extraction for oat, rye and wheat, yields of 11.20, 12.80, and 30.43% were obtained, respectively.

The purpose of the DSC study was to determine the gelatinization range of the different starches, that is, their temperatures at the beginning (T_0), peak (T_p), and end of gelatinization (T_f), as well as the enthalpy (TABLE 1). Such information is essential to determine the temperature limit in grain hydration since, for this study, the starch must not be gelatinized.

Table 1. Gelatination temperature for oat, rye and wheat starch by DSC analysis.

Cereal	T_0 (°C)	T_p (°C)	T_f (°C)	ΔH_g (J g^{-1})
Oat	54.25^b ± 0.29	57.55^b ± 0.35	60.97^b ± 0.36	0.8934^a ± 0.1428
Rye	53.22^b ± 0.47	56.88^c ± 0.69	59.75^b ± 1.12	1.2112^a ± 0.2913
Wheat	56.38^a ± 0.57	61.24^a ± 0.29	65.14^a ± 0.73	2.2867^a ± 0.1030

Mean value ± Standard deviation. Values followed by the same letters in a given column did not differ significantly according to Tukey's test (5%).

Thermal analysis of oat grain starch showed values similar to those reported in some studies (Majzoobi, Saberi, Farahnaky, & Mesbahi, 2014). On the other hand, Zhu (2017) described enthalpy values approximately ten times higher and a T_p 6.91°C higher than that found in this study. Such similarities, as well as discrepancies, may be due to the diversity of existing oat cultivars.

Verwimp, Vandeputte, Marrant, and Delcour (2004) and Gomand, Verwimp, Goesaert, and Delcour (2011) evaluated rye by DSC and found values for the start of gelatinization at 51.4 and 48.6°C, the end at 59.75 and 64.1°C, and enthalpy variation of 12.4 and 14.2 J g^{-1} respectively. Despite the difference in enthalpy variation values, the starch gelatinization temperature values found in this analysis are in the same temperature range as those refer to by the authors.

For wheat, starch gelatinization showed initial and peak values similar to the study by Cieśla and Eliasson (2003), but higher than that of Jacobs, Eerlingen, Rouseu, Colonna and Delcour (1998) and Mattioda et al. (2018). A value that draws attention is the interval between the beginning and end of this thermal event, in which the authors report values between 8 and 10°C, demonstrating that, despite the difference, it is within the same interval. The enthalpy variation is similar to found by Mattioda et al. (2018). In addition to possible reactions, starch degradation processes were evaluated through TGA (Table 2).

Table 2. Thermal degradation of oat, rye and wheat starch.

	First Peak		Second Peak	
Cereal	Temperature (°C)	Mass loss (%)	Temperature (°C)	Mass loss (%)
Oat	$76.06^b \pm 0.02$	$5.50^a \pm 1.05$	$317.27^c \pm 0.17$	$70.37^a \pm 2.40$
Rye	$81.78^a \pm 0.06$	$6.05^a \pm 0.28$	$322.33^a \pm 0.03$	$71.85^a \pm 0.61$
Wheat	$71.16^c \pm 0.08$	$5.64^a \pm 0.99$	$319.85^b \pm 0.10$	$71.48^a \pm 2.60$

Mean value ± Standard deviation. Values followed by the same letters in a given column did not differ significantly according to Tukey's test (5%).

The thermal starch degradation of the three cereals raw occurred in two stages. The first stage addressed temperatures below 120°C, with the greatest variation occurring in temperatures of 76.6; 81.78; and 71.16°C, with a mass loss of 5.5; 6.05; and 5.64% for oat, rye and wheat respectively. The mass loss is related to the evaporation of the residual water present in the starch. The second mass loss is related to starch pyrolysis, with more significant losses, being 70.37; 71.84, and 71.48%, at temperatures of 317.27, 322.33, and 319.85°C for oat, rye and wheat respectively. Those two distinct peaks indicate the purity of the starch since there is no degradation throughout the run that could indicate the presence of other components, thus validating the DSC analysis that describes the thermal events related to starch. Other authors have noted that the number of peaks and the temperature ranges in the starch analysis were similar (Hornung et al., 2017; Lima, Cabral, Neto, Tavares, & Pierucci, 2012; Pan et al., 2019).

Hydration process

Although the grains in this study are cereals, their kinetics show different behaviors, with rye (Figure 1b) having the highest water absorption per time, followed by oat (Figure 1a) and wheat (Figure 1c). The lower hydration of wheat grains can be explained by the size of the grains, which have an initial average volume of 0.0186, 0.019, and 0.030 cm^3 for oat, rye and wheat, respectively. It is known that the smaller the grain, the greater the percentage of water absorption (Montanuci et al., 2014), but it is also known that morphology, and grain composition, among others (Bewley, Bradford, Hilhorst, & Nonogaki., 2013), influence such phenomenon, which is why rye reaches higher values than oat, even with similar size.

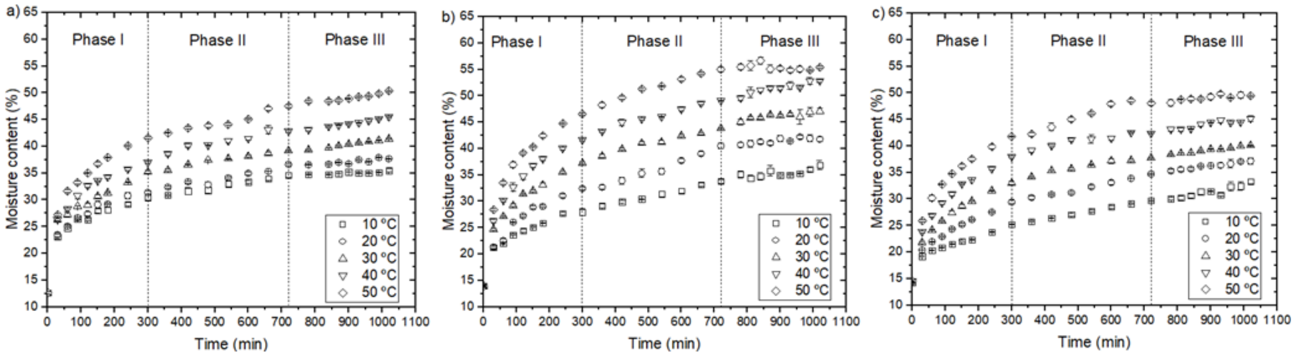

Figure 1. Kinetic behavior in oat (A), rye (B) and wheat (C) under different hydrothermal conditions (10 – 50°C), during 1020 min.

The experimental data were divided into three phases (Table 3) according to the mechanisms of water transport to the grain, following the study by Borges, Jorge and Jorge (2017), starting from an initial average moisture content of 12.65, 13.96 and 14.43% for oat, rye and wheat, respectively.

Phase I comprised the first 300 minutes of hydration due to its rapid water absorption, while phase II covered the interval from 300 to 720 minutes, where water absorption occurs with reduced speed compared to the previous phase. Phase III comprises the last 300 minutes, from 720 to 1020 minutes, with a low absorption rate, showing a strong tendency towards saturation equilibrium. Final moisture, after 1020 minutes of hydration, increased with temperature rise. Another observed behavior was that grain size influences moisture gain because rye, which has a smaller size and, consequently, a larger contact surface between the grain and water, has a higher hydration rate than wheat.

Table 3. Water absorbed by oat, rye and wheat in each phase.

Cereal	Temperature (°C)	Phase I (%w.b.)	Phase II (%w.b.)	Phase III (%w.b.)	Final Moisture (%w.b.)
Oat	10	17.60 ^{e} ± 0.53	4.19 ^{bc} ± 0.61	0.89 ^{b} ± 0.20	35.50 ^{e} ± 0.52
	20	18.77 ^{d} ± 0.21	5.22 ^{ab} ± 0.11	1.07 ^{b} ± 0.32	37.69 ^{d} ± 0.25
	30	22.67 ^{c} ± 0.27	3.98 ^{c} ± 0.25	2.21 ^{a} ± 0.24	41.40 ^{c} ± 0.13
	40	24.00 ^{b} ± 0.35	5.83 ^{a} ± 0.24	2.68 ^{a} ± 0.30	45.51 ^{b} ± 0.12
	50	29.32 ^{a} ± 0.20	5.92 ^{a} ± 0.48	2.81 ^{a} ± 0.29	50.32 ^{a} ± 0.12
Rye	10	14.34 ^{e} ± 0.60	5.85 ^{c} ± 0.31	3.11 ^{a} ± 0.44	36.90 ^{e} ± 0.84
	20	19.25 ^{d} ± 0.22	8.10 ^{a} ± 0.70	1.25 ^{b} ± 0.14	41.79 ^{d} ± 0.43
	30	22.52 ^{c} ± 0.43	6.57 ^{bc} ± 0.24	3.26 ^{a} ± 0.44	47.09 ^{c} ± 0.42
	40	27.11 ^{b} ± 0.07	7.43 ^{ab} ± 0.12	3.77 ^{a} ± 0.23	52.85 ^{b} ± 0.29
	50	32.79 ^{a} ± 0.19	8.51 ^{a} ± 0.55	0.38 ^{c} ± 0.18	55.42 ^{a} ± 0.25
Wheat	10	10.98 ^{e} ± 0.12	4.45 ^{d} ± 0.12	3.58 ^{a} ± 0.33	33.28 ^{e} ± 0.45
	20	15.03 ^{d} ± 0.33	5.31 ^{b} ± 0.23	1.52 ^{b} ± 0.24	36.24 ^{d} ± 0.33
	30	18.69 ^{c} ± 0.43	4.74 ^{cd} ± 0.19	1.60 ^{b} ± 0.22	39.33 ^{c} ± 0.23
	40	23.44 ^{b} ± 0.15	4.38 ^{b} ± 0.19	1.85 ^{b} ± 0.28	44.21 ^{b} ± 0.37
	50	27.15 ^{a} ± 0.33	6.26 ^{a} ± 0.43	1.21 ^{b} ± 0.32	49.25 ^{a} ± 0.11

Mean value ± Standard deviation. Means followed by the same letters in the columns did not differ significantly using the Tukey test (5%) for each cereal.

Evaluation of water transport regime throughout the hydration

Hydration aims to transport water into the interior of the cereal. To help understanding this process, the grains were submerged in a water solution of methylene blue dye. Through monitoring pigmentation in the grain over time, the mechanism of water transport can be evaluated (Figure 2).

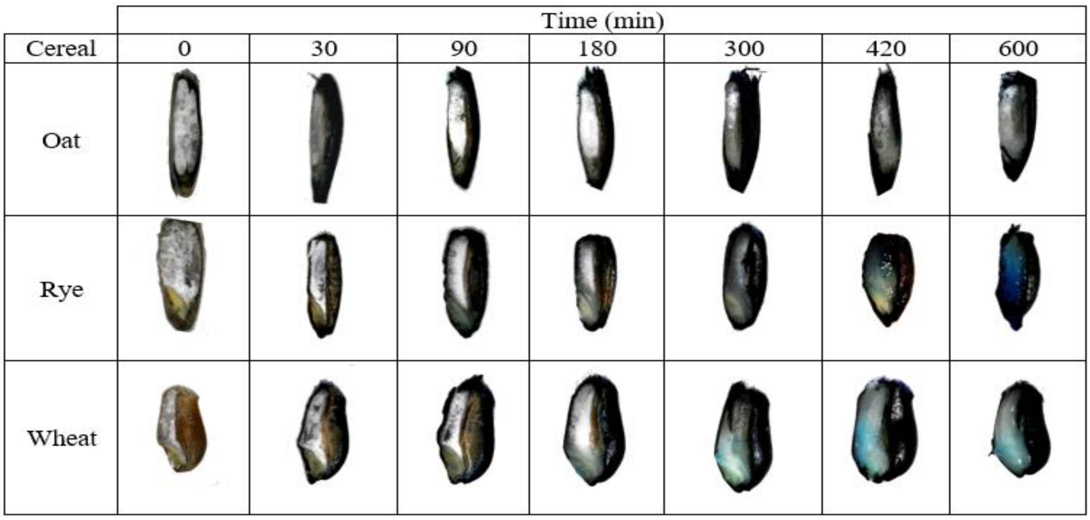

Figure 2. Kinetics of leached solids.

The three cereals have similar behavior in the mass transfer process: in 90 minutes, the water breaks the outermost barriers of the grain (pericarp), starting through the micropyle present in the grain germ, after 180 minutes, the blue color reaches a large part of the germ and a small part of the endosperm, which becomes fully colored after 600 minutes. The path taken by water is similar to other studies, such as that of oat (Anthero, Lima, Cleto, Jorge, & Jorge, 2019), barley (Chacón Alvarez et al., 2020) and wheat (Mattioda et al., 2018).

Kinetics of leached solid

In the hydration of grains, it is common to lose solids by leaching. These solids usually have starch, proteins, and antioxidants, among others (Lerma-García, Herrero-Martínez, Simó-Alfonso, Mendonça, & Ramis-Ramos, 2009), and the hydration time, as well as the temperature, can influence the amount of lost solids (Han & Lim, 2009; Miah., Haque, Douglass, & Clarke, 2002; Panda & Shrivastava, 2019). A kinetic study of the solids lost in the hydration water was carried out (Table 4).

Table 4. Values of the constant k_{ds} and of the soluble solid content in the water of oat, rye and wheat in the hydration at 50°C.

Cereal	Soluble Solids (°Brix)	k_{ds} (°Brix min^{-1}) (x10^{-3})	R^2
Oat	1.0	1.2	0.951
Rye	2.4	2.5	0.981
Wheat	1.8	1.7	0.976

The hydration water began to show solids after 240 minutes for rye, and 300 minutes for the other cereals, which corroborates the study carried out with the methylene blue tracer, which shows that after 180 minutes the water reaches the starchy part of the grain. After 300 minutes, there was greater incorporation of water in the endosperm, with possible significant leaching of solids. Another feature to be highlighted is that oat completed their loss in 540 minutes, while the other cereals continued the leaching process until the end of the experiment (1020 minutes).

This study revealed that rye had the highest rate of total and per minute solids loss, followed by wheat and oat. The solids loss model (Equation 2) had a good fit indicated by the coefficient of determination (R^2).

Modeling of hydration kinetics

The Peleg's empirical model, the phenomenological models of Diffusion, Omoto-Jorge, and its variation of Nicolin-Jorge were adjusted to the experimental data for oat, rye, and wheat, as well as their deviations were estimated (Table 5).

Table 5. Deviation of Peleg, Fick, Omoto-Jorge and Nicolin-Jorge models for oat, rye and wheat grains.

Model	Oat Deviation (%)	Rye Deviation (%)	Wheat Deviation (%)
Peleg	10	10	7
Fick	10	7	4.5
Omoto-Jorge	30	25	20
Nicolin-Jorge	15	10	10

The evaluation of the deviations between experimental and model data in the hydration of cereals showed that wheat presented the lowest average deviations (10.4%), followed by rye (13%) and black oats (16.25%). Regarding the models, the Diffusion showed the lowest average deviation (7.2%), followed by the Peleg (9%), the Nicolin-Jorge (11.7%), and the Omoto-Jorge (25%). The linear dependence of the mass transfer coefficients assists significantly in the representation of the experimental data, and this is proven by the smaller variations in the Nicolin-Jorge model about the Omoto-Jorge model. Another relevant point is that the values found in the experimental data were higher than those calculated in the lowest moisture values at the beginning of hydration. That indicates the limitation that the models present to describe the initial hydration process, that is, the high gain of initial water level cannot be well described by the models.

Analyzing the hydration of these grains one can see the effect of temperature on the hydration process, which is expressed numerically by the model parameters (Table 6).

The mass transfer is influenced by the temperature of the system (Nascimento, Balbinoti, Jorge, & Jorge, 2022). In Peleg's model, the drop in the value of the parameter k_1 (Figure 6a) at higher temperatures is one more section of data that proves this influence with the increase in temperature. Temperature, since this parameter is inversely proportional to the moisture gain. Similar behavior was found in other grains, including the analysis of oat carried out by Anthero et al. (2019), and of wheat by Mattioda et al. (2018). The values of the k_2 parameter, which are also influenced by temperature variation, are useful for determining the equilibrium moisture content. The value found mathematically was similar to that found after 24 hours of hydration, with the difference calculated experimentally being 4.1% lower for oat, 2.6% lower for rye, and 2.2% for wheat.

Based on the laws of physics, the Fick model determines the diffusivity of water through a concentration gradient. With its parameters, we can see that the increase in diffusivity is temperature dependent, with oat having the highest diffusivity in the temperature of 10°C, between 10 and 30°C the variation is slight, but the slightest variation with the increase in temperature, since wheat had the greatest increase in diffusion related to temperature.

Table 6. Obtained model parameters under different hydrothermal conditions.

Temperature		Peleg		Fick	Omoto	Nicolin	
	(°C)	k_1 (min.%$^{-1}$)	k_2 (%$^{-1}$) (x10^{-2})	D_{ef} (m^2 s^{-1}) (x10^{-12})	*ks* (cm min.$^{-1}$) (x10^{-4})	a (cm min.$^{-1}$) (x10^{-4})	-b (cm^{4}·g^{-1} min.$^{-1}$) (x10^{-3})
Oat	10	3.38^{a}±0.19	4.42^{a}±0.03	9.88^{c}±0.57	1.77^{c}±0.17	6.18^{a}±0.98	1.47^{a}±0.29
	20	3.37^{a}±0.11	4.07^{b}±0.02	10.75^{bc}±0.59	1.77^{c}±0.15	5.41^{a}±0.69	1.18^{abc}±0.20
	30	2.75^{b}±0.03	3.32^{c}±0.01	11.27^{b}±0.60	1.79^{bc}±0.15	4.88^{b}±0.73	0.92^{c}±0.19
	40	2.60^{b}±0.10	2.94^{d}±0.01	12.99^{a}±0.79	2.02^{ab}±0.18	5.82^{ab}±0.68	1.05^{bc}±0.16
	50	2.15^{c}±0.02	2.51^{e}±0.00	13.57^{a}±0.82	2.12^{a}±0.18	5.90^{ab}±0.64	0.97^{bc}±0.14
Rye	10	6.16^{a}±0.09	4.02^{a}±0.02	7.12^{e}±0.23	1.53^{d}±0.12	4.78^{d}±0.84	0.97^{bc}±0.23
	20	4.91^{b}±0.10	3.06^{b}±0.02	7.49^{d}±0.24	1.75^{cd}±0.12	4.86^{d}±0.54	0.84^{b}±0.13
	30	3.78^{c}±0.10	2.82^{c}±0.05	11.73^{c}±0.32	2.07^{c}±0.16	5.57^{bc}±0.59	0.91^{b}±0.13
	40	2.88^{d}±0.09	2.42^{d}±0.00	12.72^{b}±0.50	2.37^{b}±0.20	6.50^{b}±0.63	0.98^{b}±0.06
	50	1.85^{e}±0.00	2.21^{e}±0.00	12.72^{a}±0.50	3.23^{a}±0.29	9.08^{a}±0.56	0.91^{a}±0.06
Wheat	10	10.28^{a}±0.19	4.84^{a}±0.05	7.07^{e}±0.36	1.21^{d}±0.07	3.65^{e}±0.57	0.78^{d}±0.17
	20	6.88^{b}±0.17	3.90^{b}±0.04	10.13^{d}±0.39	1.75^{cd}±0.11	5.35^{d}±0.57	1.03^{cd}±0.15
	30	4.34^{c}±0.18	3.57^{c}±0.01	11.73^{c}±0.32	2.10^{c}±0.15	6.92^{cde}±0.64	1.32^{bc}±0.15
	40	3.16^{d}±0.07	3.06^{d}±0.02	14.31^{b}±0.45	2.86^{ab}±0.26	9.12^{b}±0.54	1.59^{ab}±0.12
	50	2.53^{e}±0.07	2.64^{e}±0.02	19.46^{a}±0.58	4.80$^{±a}$0.35	11.50a±0.94	1.73^{a}±0.20

Mean value ± Standard deviation. Means followed by the same letters in the columns did not differ significantly using the Tukey test (5%) for each cereal.

Fitting the experimental data to the Omoto-Jorge model allows obtaining the k_s parameters, which refer to mass transfer. The k_s values did not show an increase related to the increase in temperature for the oat and rye grains. In contrast, the wheat suffered an increase, showing its dependence and indicating a greater mass transfer in the grain. Similar k_s characteristics were found in other studies (Borges et al., 2017; Marques et al., 2014; Omoto et al., 2009), which reaffirms the dependence of this parameter on temperature.

The increase in the a and -b parameters of the Nicolin-Jorge model directly indicate the influence of temperature on the mass transfer of rye and wheat, whereas oats showed a more stable behavior of these parameters, considers the linear dependence of the mass transfer coefficient and considers it a tendency to zero when approaching the steady state of hydration. The behavior of parameters a and b for oats was similar to that of rice, whereas rye and wheat were similar to that of corn and (Nicolin et al., 2017; Nicolin, et al., 2015). The quality of the fit of the models was determined by the relative mean error value (P), mean error value (RMSE) and coefficient of determination (R^2) (Table 7).

Table 7. fit analysis of the models under the different hydrothermal conditions.

	Temperature	Oat			Rye			Wheat		
Model	(°C)	P	RMSE	R^2	P	RMSE	R^2	P	RMSE	R^2
	10	2.921	1.161	0.994	3.701	1.346	0.950	3.761	1.137	0.950
	20	3.093	1.291	0.991	3.545	1.506	0.977	2.953	1.046	0.977
Peleg	30	3.327	1.540	0.994	3.189	1.404	0.986	2.164	0.822	0.991
	40	2.955	1.441	0.993	3.070	1.511	0.989	2.030	0.864	0.994
	50	3.195	1.629	0.993	2.496	1.412	0.995	2.209	1.124	0.994
	10	9.306	3.053	0.951	2.216	0.812	0.979	1.821	0.728	0.957
	20	5.510	2.158	0.952	2.592	0.999	0.982	1.421	0.589	0.977
Fick	30	5.707	2.443	0.945	1.289	0.584	0.990	1.325	0.536	0.987
	40	1.497	0.717	0.952	1.639	0.792	0.990	1.790	0.772	0.985
	50	4.743	2.418	0.936	2.487	1.373	0.978	1.223	0.614	0.990
Omoto-Jorge	10	9.102	0.035	0.737	7.525	0.032	0.821	7.285	0.028	0.857
	20	8.414	0.033	0.817	8.495	0.039	0.900	6.931	0.029	0.901
	30	9.890	0.046	0.824	9.609	0.046	0.869	9.609	0.046	0.868
	40	8.658	0.050	0.812	8.376	0.049	0.846	8.376	0.049	0.825
	50	6.619	0.043	0.920	6.372	0.042	0.855	6.372	0.042	0.927
Nicolin-Jorge	10	4.867	0.021	0.902	4.716	0.022	0.909	3.688	0.016	0.940
	20	4.078	0.018	0.943	3.386	0.017	0.964.	2.677	0.013	0.969
	30	5.131	0.025	0.925	3.389	0.017	0.970	2.455	0.013	0.972
	40	4.284	0.021	0.948	3.123	0.020	0.967	1.682	0.011	0.987
	50	4.312	0.024	0.957	2.246	0.013	0.985	2.109	0.013	0.983

For a model to be considered acceptable when describing a process, the value of the mean relative error (P) and the root mean error value (RMSE) must be less than 10% and 5%, respectively (Maiorano, Mancini, & Reyneri, 2010), and when P is less than 5%, a better fit is assigned to the model (Lomauro, Bakshi, & Labuza, 1985). The determination coefficient (R^2) to be considered with good representativeness must present values above 0.92 (Madamba, Driscoll, & Buckle, 1996; Maiorano et al., 2010). When evaluating the model's fit, it is noticed that, as with the deviations, the Omoto-Jorge model was the least satisfactory, while its derivation, that is, the Nicolin-Jorge model, presented values that were more consistent with those found experimentally. The hydration behavior of oat presents the least satisfactory results, indicating the difficulty of explaining this process in this grain. Through these results, one can see that the Peleg model is the one that best describes the experimental hydration data for the three grains since it presents RMSE and P values below 5% and R^2 above 0.92.

Generalized model

The generalized model provides information on hydration as a function of time and temperature, and due to the better fit of Peleg's model, this model was generalized. As already observed in other studies, to generalize a model, temperature-dependent parameters can be related to the equation, which can be the equation of the line found for the parameters or a relationship with the Arrhenius equation, and those with no dependency is replaced by the average value found (Anthero et al., 2019; Balbinoti et al., 2018b; Borges, Jorge, & Jorge, 2019; Franco Junior, Morais, Silva, Oliveira, & Martins, 2020; Marques et al., 2014; Mattioda et al., 2019a, 2019b; Silva et al., 2019)

In the generalized model of Peleg for oat, rye, and wheat, the parameters dependent on the temperature variation were used and were related to the equation of the straight line found when associating the parameters k_1 and k_2 with the temperature (Table 8). The deviations from the models can be seen in Figure 2.

Table 8. Adjusted parameters for generalized peleg model for oat, rye and wheat grain.

Cereal	k_1	R^2	k_2	R^2
Oat	-0.0477T+4.4746	0.930	-0.0003T+0.043	0.993
Rye	-0.1064T+7.1093	0.996	-0.0004T+0.418	0.911
Wheat	-0.1921T+11.204	0.915	-0.0005T+0.052	0.966

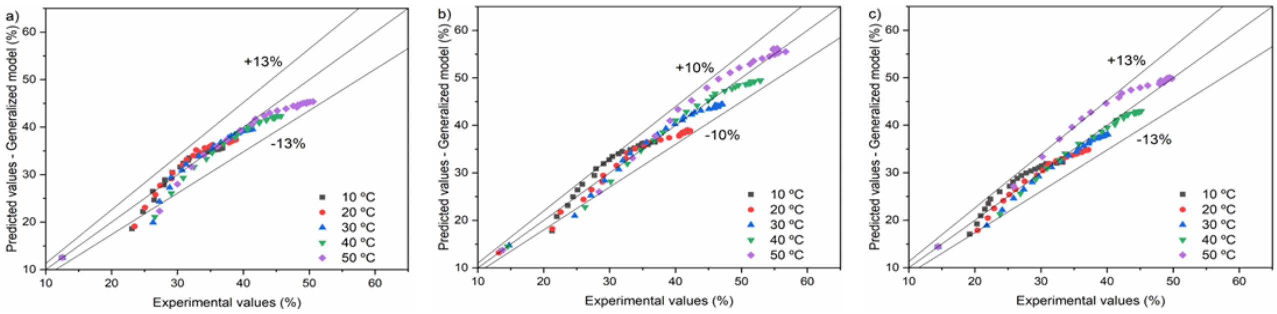

Figure 2. Correlation of the experimental moisture and predicted by generalized Peleg model for oat (a), rye (b) and wheat (c).

Generalized models predict experimental moisture at any time during the hydration process, at any temperatures investigated. Compared with the experimental data, they presented a deviation of 13% for oat and wheat, whereas rye presented a deviation of 10%. Wheat showed a smaller deviation when compared to the studies by Mattioda, Jorge, and Jorge (2019b), which was 14%.

As in the Peleg model, the data for the Generalized Peleg Model presented a good fit that was justified by the high values of the coefficient of determination (R^2), satisfactory values of the square root of the mean error (RMSE) in view of the fact that the mean relative error (P) less than 10% and in most reaching values less than 5% (Table 9).

Table 9. Fit analysis of the generalized Peleg models.

CEREAL	P	RMSE	R^2
Oat	6.721	3.208	0.962
Rye	3.970	1.813	0.971
Wheat	3.992	1.746	0.963

Conclusion

Through this study, it was possible to characterize the hydration of three different grain crops: black oats, rye, and wheat. The maximum temperature limit was 50°C, since the thermal analysis of the starch indicated that below these temperatures the starch is not degraded, which is ideal for several products from these cereals.

Three phases can be seen in the hydration kinetics of cereals, which occurred at similar times. However, rye showed a higher water absorption rate, which is probably associated with grain size. The achieved moisture values in the hydration process are appropriate levels for cereal germination, component extraction, fermentation, and malting, among others.

The analysis with methylene blue indicated that the water entry into the grains was due to capillarity through the hilum and diffusion through the grain lining. The hydration water shows solids after 240 minutes for rye and 300 minutes for the other grains. Another feature to note is that oats completed the loss in 540 minutes, while the other grains continued the leaching process until the end of the experiment (1020 minutes). The leached solids may contain important compounds of interest such as antioxidants.

The Peleg; Diffusion; Omoto-Jorge; and Nicolin-Jorge models were fitted to the experimental hydration data. In general, the models had excellent fit quality, except for the Omoto-Jorge model for the observed temperature of 30 and 40°C, which had an unsatisfactory coefficient of determination. Based on the statistical parameters (R^2, RMSE, and P), the most appropriate model for representing the experimental data in this study was the Peleg model. It was selected to be generalized, thus being able to represent the moisture for different times and temperatures.

Acknowledgments

The authors would like to thank the Coordination for the Improvement of Higher-Level Personnel (CAPES) and the National Council for Scientific and Technological Development (CNPq) grant numbers: 312215/2017-7 and 315598/2020-4. We also thank the scientific cooperation of the Analytical Center of the Department of Chemical Engineering (UFPR) and to the donors of the cereals used in this study.

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