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PRESSURE GRADIENTS APPLICATION FOR ACCELERATING CORN HYDRATION

APLICACIÓN DE GRADIENTES DE PRESIÓN PARA ACELERAR LA HIDRATACIÓN DE MAÍZ

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Abstract

Three varieties of corn kernels were hydrated under pressure gradients in the range of 96 to 136 cm Hg, and at atmospheric pressure for comparison. Hydration at atmospheric pressure was modeled by the first order kinetics model and by the Fick's second law of diffusion. Rate constants of 0.192, 0.184 and 0.141 h^{-1}, and water effective diffusivity of 5.4 $\times 10^{-11}$, 3.6 $\times 10^{-11}$, and 1.9 $\times 10^{-11}$ m^2 s^{-1} were obtained for blue, yellow and white corn kernel, respectively. Significant polynomial equations described the corn kernels hydration under pressure gradients, as a function of vacuum pressure, vacuum time and hydration time. Hydration rates of the studied grains were significantly enhanced using the pressure gradients, compared to the conventional method. Pressure gradient soaking required 60% less time than the conventional soaking, to reach a water content of 45% in corn kernels. The hydration extent of corn kernels determined the levels of solutes leaching, in both soaking treatments. Minimum hydration levels for initiating leaching of soluble solids were observed.

Keywords: corn, soaking, water diffusion, pressure gradients, hydration models.

Resumen

Se hidrataron tres variedades de granos de maíz bajo gradientes de presión en el rango de 96 a 136 cm Hg, y a presión atmosférica como comparación. La hidratación a presión atmosférica se modeló con los modelos cinético de primer orden y con la segunda ley de Fick de difusión. Se obtuvieron constantes de velocidad de 0.192, 0.184 and 0.141 h^{-1}, y de difusividad efectiva de agua de 5.4 $\times 10^{-11}$, 3.6 $\times 10^{-11}$, y 1.9 $\times 10^{-11}$ m^2 s^{-1} para los granos de maíz azul, amarillo y blanco, respectivamente. La hidratación de los granos de maíz bajo gradientes de presión se describió con ecuaciones polinomiales en función de la presión y tiempo de vacío y del tiempo de hidratación. La velocidad de hidratación de los granos se mejoró utilizando gradientes de presión en comparación al método convencional. Con el remojo bajo gradientes de presión se requirió un 60% menos tiempo para alcanzar un 45% de humedad que con el tratamiento convencional. El grado de hidratación de los granos de maíz determinó los niveles de solutos lixiviados en ambos tratamientos. Se observaron niveles mínimos de hidratación para el inicio de la lixiviación de sólidos solubles.

Palabras clave: maíz, remojo, difusión de agua, gradientes de presión, modelos de hidratación.

1 Introduction

Hydration of cereals and legumes is an important first step in many processes, before passing to following stages (Miano *et al*., 2017). The common practice is to soak the grains in water at room temperature until they reach a desired hydration level. However, it could take up to 16-36 hours to obtain a complete hydrated grain, which besides being a limiting processing factor, may favor leakage of solutes, and cause microbial proliferation or development of unpleasant odors (Joshi *et al*., 2010; Naviglio *et al*., 2013). Due to the soaking slowness of some grains, several methods have been proposed.

High temperature has been used to decrease the soaking time of corn, wheat, amaranth, rice, soybeans, and cowpea (Agarry *et al*., 2014; Maskan, 2001; Calzetta Resio *et al*., 2006; Bello *et al*., 2004; Gowen *et al*., 2007; Fracasso *et al*., 2014; Demirhan & Özbek, 2015). Application of high pressures (>10 MPa), ultrahigh pressures (275-690 MPa), cyclically

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pressurized soaking (0-1000 kPa) and pressure gradients (0-200 kPa) were reported to shorten the hydration of corn kernels, chickpea, cannelini beans, and tepary beans, respectively (Gunasekaran & Farkas, 1988; Naviglio *et al*., 2013; Ibarz *et al*., 2004; Zanella *et al*., 2014). Quick hydration has also been achieved in rough rice, navy beans, sorgo and corn kernels by application of ultrasound (~ 25-40 kHz, 25-50 W/L) (Wambura *et al*., 2008; Ghafoor *et al*., 2014; Patero & Augusto, 2014; Miano *et al*., 2017).

Different combinations of soaking variables, such as pressure- temperature (200 - 500 MPa, 20- 50 ºC) or ultrasound-temperature (100-300 W, 87-97 °C) have been effective treatments for shortening the soaking time of japonica rice grains and chickpea, respectively (Huang *et al*., 2009; Yildirim *et al*., 2015). Even chemical compounds may facilitate water uptake, like in the case of amaranth grains and use of SO_2 (Calzetta Resio *et al*., 2006).

All the above-mentioned soaking methods have demonstrated to be effective for reducing the processing time compared with the conventional soaking method. However, unfavorable conditions might reduce their effectiveness. For instance, if temperature is too high it may produce excessive leakage of solutes, very high pressure may inhibit the swelling of starch of the grains and reduce de hydration capacity. Certain soaking methods are expensive, high energy consuming, or not easy to implement and to scale-up (Ibarz *et al*., 2004; Bello *et al*., 2004). It should be mention that the type of grain, variety, and its inherent characteristics play an important role in the effectiveness of the hydration and that there is no one best soaking method for all the different grains.

Hydration by pressure gradients, most known as vacuum impregnation (VI), has been used to efficiently introduce water or other external solution in porous matrices (Valdez *et al*. 2013). VI is a relatively simple operation that involves a vacuum pulse application to a solid product- solution system, followed by a relaxation stage at atmospheric pressure. In these consecutive steps the occluded gases are expelled from the porous structure, which is then occupied by the external liquid during the relaxation step, under the action of the generated pressure gradient (Zanella *et al*., 2014). It has been reported that hydration of beans can be accelerated when the relaxation or hydration step is carried out at over atmospheric pressures that create higher pressure gradients (Zanella *et al*., 2014).

The objective of this work was to evaluate the effect of pressure gradients on the hydration time of three corn varieties, and to compare it with the hydration at atmospheric pressure.

2 Materials and methods

2.1 Raw material

Three varieties identified as blue corn landrace (Don Goyo, from State of Mexico), yellow corn hybrid (Asgrow 773, from Sinaloa), and white corn hybrid (QPM, Quality Protein Maize, from Zacatecas) were used in this study. Blue maize (d= 0.79± 0.001 cm) is considered as flour corn and yellow (d= 0.79± 0.002 cm) and white (d= 0.79± 0.002 cm) corn hybrids as dent corn. All maize varieties were harvested during 2016 and after harvest, the grains were dried (~13% moisture) and stored at 4°C until further use. The corn samples used in the soaking tests were hand selected, removing broken or damaged pieces.

2.2 Soaking treatments

A sample of each variety of corn grain (~15 g) was placed in distilled water at ambient temperature (T= 24±2°C), using a corn to water ratio of 1 to 10 (w/w). Then, the soaking process was carried out at either atmospheric pressure (717 cm Hg) or under pressure gradients. In these last treatments, the corn kernel-water systems were placed in a hard-cast aluminum pressure vessel (H = 16 3/4 inches, D = 12¼ inches) with capacity of 23 L. Two stainless steel manometers were attached to the pressure vessel, which was connected to the vacuum pump.

Experiments at atmospheric pressure were carried out for soaking periods up to 26 hours in three replicates. In the pressure gradient process, the corn kernel-water systems were placed in the pressure vessel at different soaking pressure gradients in the range between 96 - 136 cm Hg. These pressure gradients resulted from the consecutive application of a vacuum pulse (p_v, 20 - 60 cm Hg gauge) and an overpressure of 76 cm Hg gauge to the soaking systems. Soaking conditions came from the combination of vacuum pressure (p_v), vacuum application time (t_v), and soaking time (t_s) given by a central composite experimental design (Table 1) (Design-Expert Software v.9.0.6.2, 2015). Hydrated samples were drained for 1 min, and then dried with absorbent paper. Immediately afterwards, the samples were weighed (Ohaus Pioneer, Mod PA 224C, Bradford MA, USA).

Table 1. Independent variables and levels in the central composite design for corn kernels soaking.

Independent variables	Symbol	Actual factor levels				
		1	2	3	4	5
Vacuum application time (min)	t_v	2	4	7	9.9	12
Vacuum pressure pulse (cm Hg)	p_v	20	28.1	40	51.9	60
Soaking time (h)	t_s	0.5	1.2	2.2	3.3	4

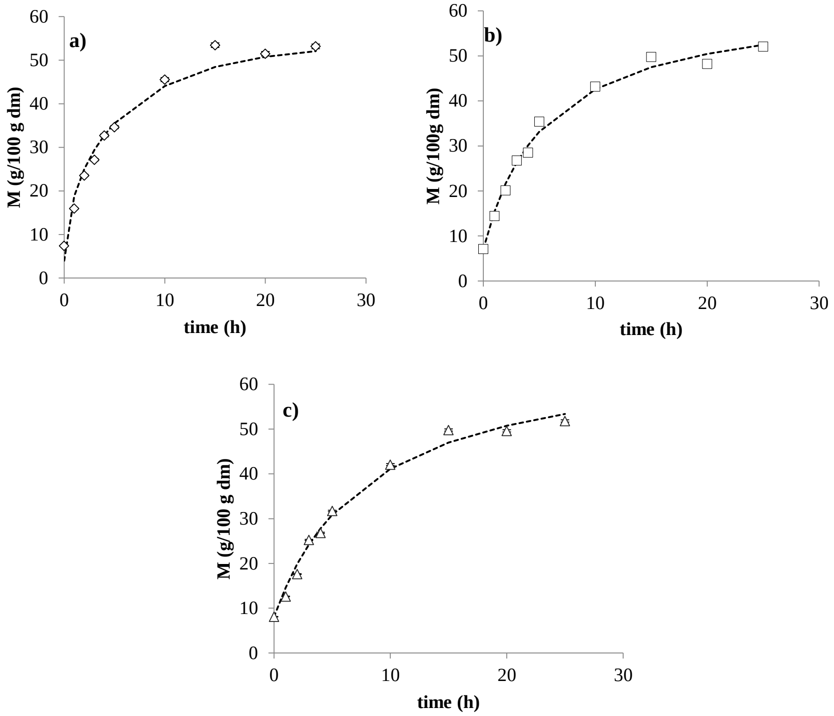

Fig. 1. Moisture gain of a) blue corn, b) yellow corn, and c) white corn kernels during soaking at atmospheric pressure.

2.3 *Absorbed water*

The initial and final moisture of each corn kernels sample was determined using the standard method 44-40-01 (AACC 1999). The difference in weight of the hydrated grains and the initial dry matter was used to calculate the absorbed water (M).

$$M = \frac{W_F - W}{W_O} \times 100 \qquad (1)$$

where M- moisture gained (g water/ 100 dm); W_O - initial weight and W_F - final weight of the samples.

2.4 *Leached solids*

The soluble solids leached from the grains after soaking in water, were determined by the difference in solids weight before and after the soaking process.

$$SL = \frac{W_{SO} - W_{SF}}{W_{SO}} \times 100 \qquad (2)$$

where SL- leached dried solids (g dried solids/ 100 g dm); W_{SO}- initial weight of solids and W_{SF} - final weight of solids.

3 Results and discussion

3.1 *Soaking at atmospheric pressure*

3.1.1 Moisture gain

The increasing trend of moisture gain by corn kernels versus time, during soaking at atmospheric pressure, is shown in Fig. 1. The hydration curves follow a typical behavior: at the beginning of the process the rate of water absorption (dM/dt) is high and decreases with soaking time until it reaches zero. At this point, the equilibrium moisture content values for blue, yellow and white corn kernels are around 52.7, 50.0, and 50.4 g water/100 g dm, respectively, which were reached at around 15 h of soaking.

Thus, in this process the water content increased up to 6.2.4- 7.3 times its original value, which is similar to hydration levels reported for corn and other grains of high starch content (Bello *et al*., 2004; Miano, *et al*., 2017; Patero & Augusto, 2015).

The first order kinetics model and the Fick's second law of diffusion for a sphere were applied for modeling the water uptake of the corn kernels, eq. 3 and eq. 4, respectively (Bello *et al*., 2004; Virgen-Navarro *et al*., 2016; Mendoza-Madrigal *et al*., 2017; Miano *et al*, 2017).

$$M = M_{eq} - (M_{eq} - M_o)e^{-kt} \tag{3}$$

$$M = M_{eq} - (M_{eq} - M_o)\left(\frac{6}{\pi^2}\right)\sum_{n=1}^{\infty}\left(\frac{1}{n^2}\right)e^{-D_{eff}\frac{\pi^2 n^2}{r^2}t} \tag{4}$$

where M- moisture content at a specific time (g /100 g dm), M_{eq}- equilibrium moisture content (g /100 g dm), M_o- initial moisture (g /100 g dm), t- time (h), k- first order rate constant (1/h), D_{eff} - effective moisture diffusivity (m^2/s), r - equivalent radius of the corn grains (m), and n - number of terms in the series.

The parameters of the fitted models for describing the hydration kinetics of the corn kernels are summarized in Table 2. The sum of the first six terms in the series (eq. 4) were used to calculate the effective diffusion coefficient of water.

The coefficient of determination (R^2_{adj}) corresponding to the first order model is higher than the R^2_{adj} of the diffusion model, on the other hand, the mean relative error (MRE) and root-mean-square deviation (RMSD) values are smaller. It can be seen that the equilibrium moisture content values predicted by the diffusion model are slightly over-estimated, contrarily to the first order kinetic model, whose predicted values are quite close to the experimental ones. These differences may be partially due to the assumptions that the diffusion coefficient is independent of the water content (constant diffusion coefficient), as well as the swelling and non-sphericity of the grains (Ghafoor *et al*., 2014). Although both models describe the behavior of the hydration kinetic curves (high R^2_{adj}), the first order kinetics model is the most appropriate to predict water uptake of the three corn kernels.

The first order rate constants and the water effective diffusion coefficient values indicate that blue corn kernels take up water more rapidly than the yellow and white corn kernels. The effective diffusion coefficients of the studied corn varieties (1.93 - 5.42×10^{-11} m^2/s) are in the range of the reported values for similar corn varieties (3.28×10^{-11} - 5.69×10^{-11} m^2/s), (Agarry *et al*., 2014), but clearly lower than those reported for corn kernels hydrated at higher temperatures (5.66×10^{-10} - 19.10×10^{-10} m^2/s) (Marques *et al*., 2014).

The difference in the hydration rate of the studied grains may be due to the differences in internal structure, the thickness of the pericarp, the space between the endosperm and embryo, and the proportion of the chemical compounds in each grain (Miano *et al*., 2017; Ramos *et al*., 2004).

Table 2. Kinetic and statistical parameters of the fitted models to describe hydration of corn kernels at atmospheric pressure and room temperature.

Model	Corn variety	Regression parameters	R^2_{adj}	MRE[a] (%)	RMSD[b]
First order	Blue	k = 0.192 h-1 ; Me = 53.6%	0.994	2.6	1.29
	Yellow	k = 0.184 h^{-1} ; Me = 51.3%	0.993	2.86	1.36
	White	k = 0.141 h^{-1} ; Me = 53.4%	0.994	3.89	1.27
Fick	Blue	$D_{eff} = 5.4\times10^{-11}$ m^2/s ; Me = 53.6%	0.969	9.72	2.84
	Yellow	$D_{eff} = 3.6\times10^{-11}$ m^2/s ; Me = 57.8%	0.974	9.24	2.53
	White	$D_{eff} = 1.9\times10^{-11}$ m^2/s ; Me = 68.9%	0.97	10.35	2.78

[a] Mean relative error (MRE) = $100/N * \sum|M_{experimental} - M_{predicted}|/M_{predicted}$; [b] Root-mean-square deviation $RMSD = \sqrt{\frac{1}{N-M}\sum[(M_{experimental} - M_{predicted})^2}$; where N- number of data and M- number of regression parameters of the model.

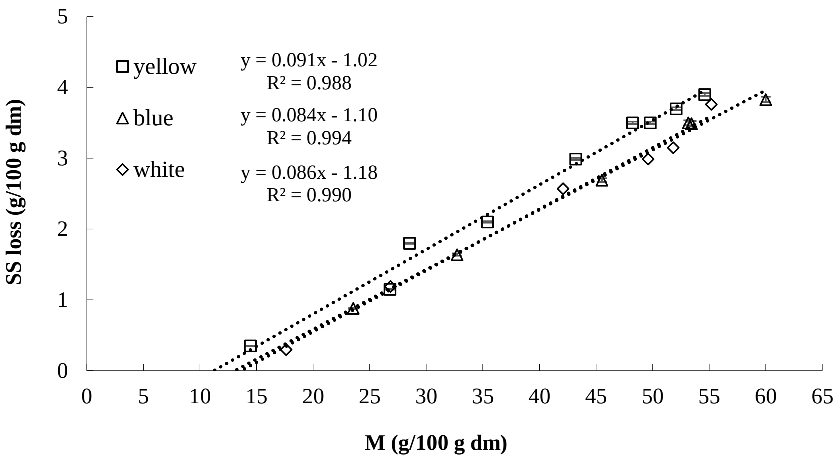

Fig. 2. Soluble solids loss of corn kernels as a function of moisture content during soaking at atmospheric pressure.

3.1.2 Leached solids

The soluble solid loss (SSL) data were plotted as a function of the corresponding moisture content, for the soaking treatment at atmospheric pressure (Fig. 2). A strong linear correlation ($R^2 \geq 0.990$) was obtained between SSL and the hydration level of the corn kernels.

The slope of the straight lines is a measure of the corn kernels facility to lose solutes, which is in descending order: yellow corn>white corn>blue corn. On the other hand, the interception of the straight lines with the M-axis (SSL= 0), corresponds to the minimum moisture level required to trigger the solutes solubilization present in the grains. The leaching of soluble solids starts when the yellow, blue and white corn kernels reach a moisture content of 11.1, 13.0 and 13.8%, respectively, since at this point, the hydration of grains tend to swell them allowing the leaching of soluble solutes (Calzetta Resio *et al*., 2005).

3.2 Soaking under pressure gradients

3.2.1 Moisture gain

Analysis and regression modeling of the obtained hydration data (Table 3) and the ratio soluble solid loss to moisture content (SSL/M) (data not shown) were conducted using Design Expert v.9.0.6.2, 2015 (Stat-Ease Inc., Minneapolis, MN, USA).

The developed models did not show lack of fit and presented high determination coefficients (Table 4), which explain about 87.9%, 95.0% and 92.9% in the variability of the water uptake of blue (M_B), yellow (M_Y), and white (M_W) corn kernels, respectively. The models for M_W and M_B include the linear effect of t_v ($p < 0.05$), while the linear effect of p_v was significant ($p < 0.05$) in M_Y and M_B. A significant correlation of the interaction $t_v t_s$ ($p < 0.05$) with the response M_W was observed. Only the soaking time had significant linear effect ($p < 0.05$) on water uptake of the three corn varieties. Among the coefficient values, those corresponding to the soaking time are considerably higher than the other coefficients (Table 4), which confirms the importance of this variable in the hydration of the grains.

Although the applied vacuum pressure and time of application did not have a significant effect on the water uptake of all the grains, the vacuum application step is required to expel the air entrapped in their internal structure. Otherwise, if gas is present in the internal voids, it will expand after the soaking period under pressure, driving out the absorbed water (Martínez Monteagudo *et al*., 2006; Valdez Fragoso *et al*., 2013).

The response surfaces showing the linear effect of soaking time (ts) are depicted in Fig. 3, where it is clearly seen that the moisture levels of the corn kernels increase with soaking time, at any vacuum application time. Under these soaking conditions, moisture content increased approximately from 25 to 40%, 26 to 39%, and 23 to 47% in the blue, yellow, and white corn kernels, respectively. These results show that in the first 0.5 h of soaking under pressure gradients, the hydration levels are around 2-3 times higher than the those levels attained with the soaking process at atmospheric pressure (Fig. 1).

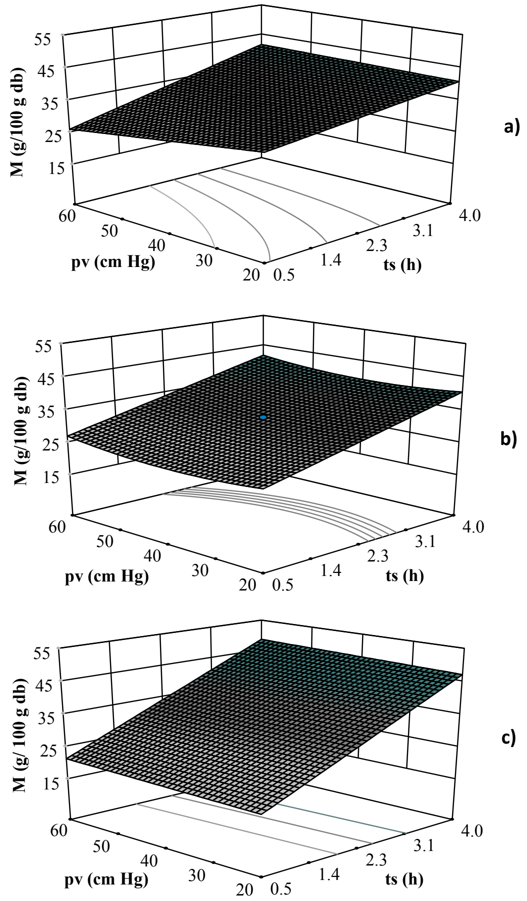

Fig. 3. Water absorption of a) blue, b) yellow and c) white corn kernels, at $t_v = 2$ min.

In this case, water flows by molecular diffusion, whereas in the former case the hydrodynamic mechanism drives bulk water incorporation into the corn kernels (Valdez-Fragoso *et al* ., 2013).

The highest hydration levels attained in the studied corn kernels (39-47%) (Fig 3.) are similar to those obtained in corn under high pressure soaking (10.5-70 MPa) (Gunasekaran & Farkas. 1988). The reached values with this treatment are around 5 % lower than the equilibrium moisture content (50 %) obtained with the atmospheric soaking treatment (Fig. 1) and the reported in a hydration study of corn kernels by using ultrasound (50 %) (Miano *et al*., 2017). However, the atmospheric and ultrasound soaking treatments were considerably longer (14 h) than the pressure gradient treatment (4 h).

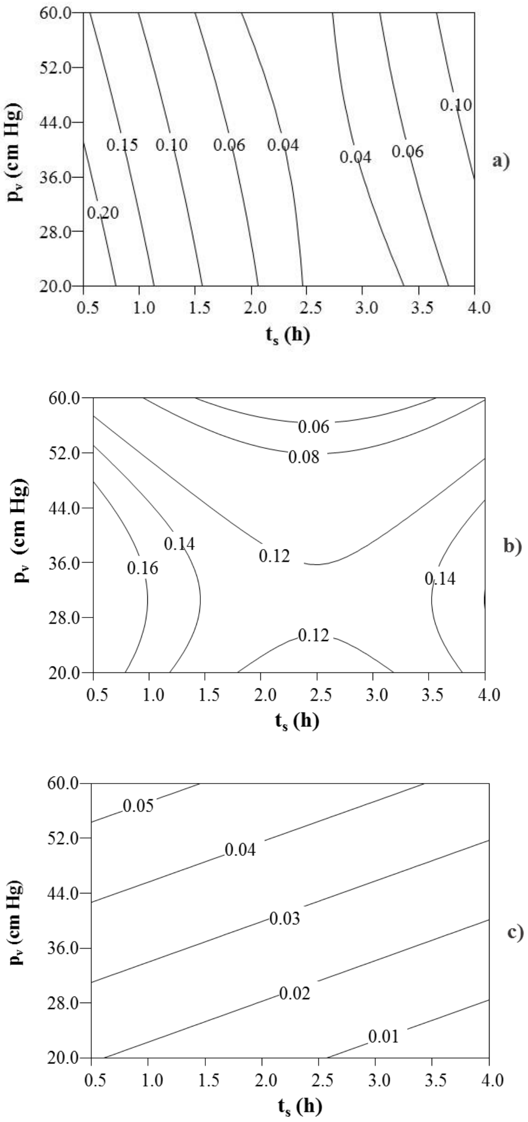

Fig. 4. Contour plots of the ratio SSL/M of a) blue, b) yellow, and c) white corn kernels, obtained at $t_v = 2$ min.

Moisture content up to 50 and 45 % have been found in white and yellow corn kernels, hydrated at 60 °C (Agarry *et al*., 2014), which is consistent with the effect of temperature described by Arrhenius equation.

In terms of energy and equipment cost, soaking corn kernels by application of pressure gradients, seems to be more advantageous than ultrasound, warm water soaking, and high-pressure hydration, considering the hydration levels and soaking time.

Table 3. Absorbed water of three corn kernels varieties, soaked under pressure gradients.

Run	t_v (min)	p_v (cm Hg)	t_s (h)	Moisture (g/100 g dm)		
				Blue kernel	Yellow kernel	White kernel
1	9.9	28.1	3.3	37.92	36.3	37.6
2	7	40	2.2	33.52	32.78	33.71
3	12	40	2.2	34.86	34.74	35.23
4	7	40	4	41.92	40.16	42.44
5	7	40	2.2	34.2	31.42	34.68
6	4.03	51.9	1.2	31.34	28.71	29.92
7	2	40	2.2	35.23	32.84	34.04
8	7	40	0.5	27.51	24.31	21.68
9	9.9	51.9	33	37.5	38.39	38.61
10	4	28.1	1.2	34.57	29.31	25.93
11	7	20	2.2	37.14	33.78	35.88
12	7	40	2.2	35.56	32.18	32.52
13	9.9	28.1	1.2	35.17	28.11	27.56
14	7	40	2.2	35.15	33.1	33.4
15	7	40	2.2	34.6	32.38	32.21
16	7	60	2.2	33.86	35.82	33.67
17	4	51.9	3.3	39.07	36.28	42.61
18	4	28.1	3.3	39.62	36.55	41.43
19	9.9	51.9	1.2	31.38	28.32	30.95

t_v-vacuum aplication time; p_v - vacuum pressure level; t_s- soaking time.

Table 4. Regression polynomials to describe moisture gain and the ratio soluble solids loss/moisture of the three corn varieties, soaked under pressure gradients.

Model[a]	Lack of fit	R^2
$MB = 37.39 - 0.08t_v \mathbf{- 0.22p_v + 0.78t_s + 0.06p_vt_s}$	ns	0.903
$MY = 28.57 + 0.08t_v - 0.33p_v + \mathbf{4.20t_s + 4.61 \times 10^{-3}p_v^2}$	ns	0.953
$MW = 13.53 + 0.87t_v + 0.03p_v + \mathbf{8.65t_s - 0.42t_vt_s}$	ns	0.933
$(SSL/M)B = 0.53 - 0.02t_v - 5.02 \times 10^{-3}p_v \mathbf{- 0.24t_s}$ $\mathbf{+3.14 \times 10^{-4}t_vp_v + 1.09 \times 10^{-3}p_vt_s + 9.83 \times 10^{-4}t_v^2 + 0.04t_s^2}$	ns	0.935
$(SSL/M)Y = 0.28 - 0.03t_v \mathbf{+ 3.42 \times 10^{-3}p_v} - 0.12t_s$ $+1.04 \times 10^{-3}p_vt_s + \mathbf{1.79 \times 10^{-3}t_v^2 - 9.45 \times 10^{-5}p_v^2 + 0.01t_s^2}$	ns	0.825
$(SSL/M)W = -0.08 + \mathbf{0.01t_v} + 2.23 \times 10^{-3}p_v \mathbf{- 0.02t_s}$ $\mathbf{-1.97 \times 10^{-4}t_vp_v - 1.52 \times 10^{-3}t_vt_s - 4.36 \times 10^{-4}p_vt_s}$	ns	0.777

[a] significant models (p<0.01) in terms of actual factors; bold terms are significant (p<0.1); subscripts B, Y and W are the corresponding blue, yellow and white corn kernel

3.2.2 Leached solids

The variation of the SSL/M ratio with the variables of soaking under pressure gradients is described by the polynomials given in Table 3. In contrast to the models for moisture gain M, the SSL/M models have lower determination coefficients and include more terms as well as some interaction effects. These differences in polynomials might be ascribed to the complexity of the leaching phenomenom, which is partially due to the high molecular size of the solutes, the physical and chemical properties of the pericarp, and the mass transfer mechanism.

The contour plots of Fig. 4 present the SSL/M ratio as a function of ts and pv. It can be seen that blue and yellow corn kernels lose solutes more easily than white kernels, and that the behavior is very different for each grain. The highest SSL/M values for blue, yellow, and white corn kernels are around 0.20, 0.16

and 0.05 g SSL/g dm, respectively, which are reached during the first 30 min of soaking. At this point, moisture gain is around 52 %, 34%, and 48% of the final hydration levels achieved by blue, yellow, and white corn kernels, respectively (Fig. 3). Under such hydration conditions the grains swell considerably, accelerating the leaching of solids (Calzetta Resio *et al.*, 2005).

Conclusions

- Application of pressure gradients considerably reduced the hydration time of the studied varieties of corn kernels, compared to the atmospheric soaking treatment
- The hydration kinetics of corn kernels under atmospheric pressure and pressure gradients conditions was mathematically modelled.
- The soluble solids loss was linearly correlated with the hydration level of corn kernels hydrated at atmospheric pressure.
- Considerable leaching of soluble solutes occurs at the initial period of the hydration under pressure gradients.

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