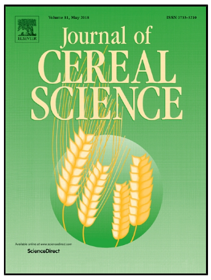
# Accepted Manuscript




Effect of intermittent high-intensity sonication and temperature on barley steeping for malt production

Gisandro Reis de Carvalho, Tiago Carregari Polachini, Roger Darros-Barbosa, José Bon Corbín, Javier Telis-Romero

| | |
|---|---|
| PII: | S0733-5210(18)30160-7 |
| DOI: | 10.1016/j.jcs.2018.06.005 |
| Reference: | YJCRS 2585 |
| To appear in: | *Journal of Cereal Science* |
| Received Date: | 21 February 2018 |
| Accepted Date: | 09 June 2018 |

Please cite this article as: Gisandro Reis de Carvalho, Tiago Carregari Polachini, Roger Darros-Barbosa, José Bon Corbín, Javier Telis-Romero, Effect of intermittent high-intensity sonication and temperature on barley steeping for malt production, *Journal of Cereal Science* (2018), doi: 10.1016 /j.jcs.2018.06.005

# Effect of intermittent high-intensity sonication and temperature on barley steeping for malt production

Gisandro Reis de Carvalho (CARVALHO, G. R.)[1]; Tiago Carregari Polachini (POLACHINI, T. C.)[1,2]; Roger Darros-Barbosa (DARROS-BARBOSA, R.)[1]; José Bon Corbín (BON, J.)[2]; Javier Telis-Romero (TELIS-ROMERO, J.)[1]*

[1] São Paulo State University (Unesp), Institute of Biosciences, Humanities and Exact Sciences (Ibilce), Campus São José do Rio Preto, Food Engineering and Technology Department – DETA, Cristóvão Colombo St. 2265, 15054-000, São José do Rio Preto, Brazil.

[2] Grupo de Análisis y Simulación de Procesos Agroalimentarios (ASPA), Departamento de Tecnología de Alimentos, Universitat Politècnica de València (UPV), C/ Camino de Vera, s/n, 46071, Valencia, Spain.

*Corresponding author.

E-mail address: jtelisromero@gmail.com

Phone number: + 55 (17) 3221-2251

Fax number: +55 (17) 3221-2250

**Abstract**

Barley malt production comprises three main steps: steeping, germination, and drying. Ultrasound technology has been widely studied to find ways to improve mass transfer in food processes and, consequently, to reduce process times. So, this study evaluated the effect of temperature and the intermittent application of ultrasound on the steps involved in barley hydration. The barley hydration was carried out at 10, 15, 20, and 25 °C with and without the application of 0.75 W/mL and 1.5 W/mL of nominal power density at 20 kHz. The ultrasonic energy delivered was measured in the same conditions as the steeping process using a calorimetric method, taking distinct differential volume measurements throughout the hydration medium. The ultrasonic energy delivered presented average values of 51.1 W at 750 W and 84.7 W at 1500 W nominal power. Ultrasound application increased both water uptake rates and equilibrium moisture content as shown by the Peleg and Weibull-exponential model parameters, with the latter showing better adjustment ($R^2_{adj}$>0.953 and *NRMSE*<5%). Applying ultrasound also significantly reduced the time required to achieve the conventional moisture level required for barley germination: 29% and 44% at controlled temperatures of 20 $^{o}$C and 25 $^{o}$C, respectively.

**Keywords:** hydration; malt; high-intensity ultrasound; non-conventional technologies.

## 1. Introduction

Barley is the most important grain for the malting industry, widely used for the production of beer, whiskey, barley wines, malt extracts, and other products (Yaldagard et al., 2008). This is mainly due to the technological properties and flavoring characteristics that barley malt can confer to foodstuffs. These attributes are a consequence of a series of reactions that occur during a careful and protracted malting process.

In general, the malting process comprises three main steps: steeping or hydration, germination, and drying. During conventional steeping, the grain is soaked in water for 24 to 36 hours in order to increase moisture levels to 40-46% (w.b.) (Brookes et al., 1976). After steeping, the grains take approximately 3 to 6 days to germinate. During hydration and especially during germination, water promotes the transport of gibberellic acid inside the grains which leads to the production of enzymes such as α- and β-amylase. This process causes changes in the structure of the grain, influencing the quality of the malt (Mayolle et al., 2012; Montanuci et al., 2015). Afterwards, the green malt is dried to reduce the water content to 4-5% (w.b.). This interrupts the biochemical reactions and develops the malt's flavor (Samaras et al., 2005). The time-temperature binomial of this last operation is highly dependent on the hydration process, since the sensitivity of the enzymes may change in accordance with the moisture content of the grain (Lewis and Young, 2001).

The hydration process is an essential unit operation for dried products like barley, as it defines the product's properties and subsequent uses, such as cooking, extraction, fermenting, germinating, and eating (Patero and Augusto, 2015). Concerning the malt quality, controlling the amount of barley water absorbed is imperative to improving the malting process. However, water absorption by the grain may be influenced not only by factors such as barley composition and grain structure, but also by water temperature during steeping and the specific steeping methods employed (Montanuci et al., 2013).

As a way of accelerating grain hydration, high-intensity ultrasound (US) provides a novel technological solution to improve mass transfer processes. This technology has many applications in the food industry (Cárcel et al., 2012; Garcia-Noguera et al., 2010; García-Pérez et al., 2007). Although high-intensity ultrasound already plays an important role in the hydration of seeds and cereals such as beans and sorghum (Ghafoor et al., 2014; Patero and Augusto, 2015) there are still few studies that specifically look at barley steeping (Miano et al., 2015; Yaldagard et al., 2008).

High-intensity ultrasound is able to enhance mass transfer through mechanisms related to both solid and liquid phases, concerning barley and water respectively, in the specific case of barley hydration. Solids under the effect of acoustic waves might behave like sponges due to a rapid series of alternating contractions and expansions (García-Pérez et al., 2007). The same effect can induce the formation of micro channels inside grain and reduce the resistance to water absorption (Ghafoor et al., 2014). Ultrasound can also promote micro-agitation in solid–fluid interfaces (Liang, 1999), which can reduce the external resistance and increase the water transport into the barley grain.

The operating temperature is also an important factor when dealing with grain hydration. Resio et al. (2006) reported faster hydration and a slightly higher moisture content saturation point in amaranth grains when soaked at higher temperatures at a studied range of 30–60 ºC, although at the highest temperature the grains are supposed to lose solids to the hydration medium. The temperature dependence of steeping is probably related to a higher agitation state of water molecules at higher temperatures, and this trend was also observed in conventional barley grain steeping at a range of 10–35 ºC (Montanuci et al., 2013; Borges et al., 2017). However, the temperature should be controlled when the hydration process is assisted by high-intensity ultrasound in order to prevent the system over-heating and any other undesirable effects on either the malt or the operating conditions.

The absorption of part of the acoustic energy into heat, which is proportional to the intensity and attenuation of the ultrasonic waves traveling through the medium, should be investigated to avoid undesirable effects (Raso et al., 1999). Generally, this determination has been made through the measurements of punctual temperature variations during the first periods of ultrasound application in different processes (Polachini et al., 2017; Margulis and Margulis, 2003; Chivate and Pandit, 1995). However, the energy is dissipated throughout the liquid and may not reach all of the material of interest. The study by Fan et al. (2017) reinforced the need for information about a specific acoustic field as a function of different positions of the ultrasonic device.

Therefore, the present work was intended to provide an alternative approach to obtaining average acoustic energy for whole volume elements using the calorimetric method. Additionally, this study evaluated the effects of high-intensity ultrasound at different controlled temperatures during the hydration process of whole barley kernels, by measuring the water uptake and modeling the rate of water uptake as a function of time, temperature, and ultrasound power intensity. The characterization of this process is essential for the correct design of hydration processes assisted by ultrasound.

## 2. Materials and methods

### *2.1. Raw materials*

Barley (*Hordeum vulgare*, variety Shakira) kernels, kindly donated by the Agroindustrial Cooperative (Guarapuava, Paraná, Brazil), were used for the hydration experiments. Initial moisture levels were determined using AOAC methods (AOAC, 2005), oven-drying at 105 °C, in three replicates. The initial moisture level was 10.01 ± 0.65 g/100 g (d.b.). The grains were stored in a refrigerated low-humidity room.

*2.2. Ultrasound-assisted hydration experiments*

The ultrasound-assisted hydration experiments were carried out in the system schematized in Figure 1. Approximately 100 g of barley kernels were placed in a perforated stainless-steel sample holder, inside a jacketed stainless-steel vessel containing 1000 mL of water. Constant-temperature water circulated in the jacket side of the vessel, provided by an ultra-thermostatic circulated water bath (MA-184, Marconi, Piracicaba, Brazil), to control the hydration temperature.

A Vibra-Cell VCX-1500 ultrasound processor horn type (Sonics & Materials, Newton, USA), with a 25 mm diameter axial sonotrode operating at 20 kHz frequency and 1500 W maximum nominal power, was used to assist in the steeping treatments. The ultrasonic tip of the sonotrode was axially placed 1.5 cm above the sample in the center of the stainless-steel vessel (Fig. 1).

After each specific steeping period (1, 2, 3, 4, 6, 8, 10, 14, and 24 hours), the sample holder was removed from the vessel, drained to remove excess superficial water, weighed, and placed back into the hydration vessel in order to determine the water uptake. Ultrasound acoustic waves were continuously applied for 30 minutes every time a sample was placed in the hydration unit, and then interrupted until it was removed for weighing. This resulted in a total of 4.5 hours of ultrasound application during the 24 hours. The water content ($X$) was calculated by mass balance, with the initial sample mass ($m_0$), the initial moisture ($X_0$), and the sample weight ($m$) as a function of hydration time ($t$). For this calculation, increases in the weight of the sample were considered to be exclusively due to the mass of water absorbed by the grain. Additionally, the dissolution of soluble solids towards the medium was negligible (Patero and Augusto, 2015).

The steeping experiments were carried out at four different temperatures (controlled at 10, 15, 20, and 25 $^{o}$C), in accordance with the upper temperature limits required for barley germination and malting industry practices (Brookes et al., 1976). The influence of high-intensity ultrasound nominal power on barley hydration was evaluated by applying three different levels (0, 750, and

1500 W). For each condition, the steeping experiment was carried out in duplicate according to an experimental factorial design.

*2.3. Ultrasonic energy measurements*

Prior to the experiments, the ultrasonic energy actually delivered ($P_{US}$) in the hydration experiments was determined using the calorimetric method adapted from Cárcel et al. (2012). The ultrasonic energy was obtained for the hydration unit presented in Fig. 2a, in which an energy balance was applied for the time interval of the application of ultrasound acoustic waves from time $t_0$ to $t_{US}$ and average water temperature from $T_0$ to $T_{US}$. The temperatures were measured by means of thermocouples placed in the water and connected to the data acquisition module (NI 9213, National Instruments, Austin, USA). LabView software (National Instruments, Austin, USA) was used to record the temperature on a computer (Fig. 1). The temperature inside the medium was recorded (in triplicate) during the 90 seconds ($t_{US}$) of ultrasound application (Raso et al., 1999) in 57 positions (four radial positions, including $r$ = 0; six angular positions, and three axial positions, as showed in Fig. 2b). Temperature recordings were carried out in the same conditions as the hydration experiments at 10, 15, 20, and 25 °C and high-intensity ultrasound at 750 and 1500 W nominal power. From the measured temperatures at each point, a mean volumetric temperature of the hydration water was calculated by Eq. (1) using the rectangular integration:

$$T = \frac{1}{V}\int T_i dV_i \qquad (1)$$

Considering that energy exchanges between the hydration water and the jacketed vessel were negligible for the short time of temperature recording, the accumulated energy accounted for the acoustic power ($P_{US}$) by Eq. (2).

$$P_{US} = \rho V c_p \frac{dT}{dt} \qquad (2)$$

As expected, the similar linear profile of temperature versus time was observed in the hydration medium, and the values of d*T*/d*t* were acquired by linear fitting. The thermophysical properties of the hydration water were obtained using the Eqs. (2) and (3) reported by Carvalho et al. (2015). These values were considered constant at the mean temperature during heating.

$$\rho = 995.3 - 8.442 \times 10^{-3} T^2 \qquad (2)$$

$$c_p = 3.112 + T^{3.406 \times 10^{-3}} \qquad (3)$$

From the ultrasonic energy ($P_{US}$) values, the ultrasonic density was also calculated according to Eq. (4):

$$D_{US} = \frac{P_{US}}{V} \qquad (4)$$

*2.4. Mathematical modeling for ultrasound-assisted barley hydration*

Two empirical models were used to describe the hydration of barley assisted by ultrasound. The Peleg model (Peleg, 1988) represented by Eq. (5) describes the kinetics of moisture sorption that asymptotically approaches the equilibrium:

$$X = X_0 + \frac{t}{k_1 + k_2 t} \qquad (5)$$

where $k_1$ is the Peleg model's constant rate, or the inverse of the hydration rate, and $k_2$ is the constant of capacity of the Peleg model which is related to the equilibrium moisture content ($X_{eq}=X_0+1/k_2$).

The Weibull-type exponential model with three parameters describes hydration processes as probabilistic events, which may present some variability such as mass gains or losses (Cunningham et al., 2007; Cunha et al., 1998). It is represented by Eq. (6):

$$\theta = 1 - \exp\left(-\left(\frac{t}{\beta}\right)^{\alpha}\right) \qquad (6)$$

where $\theta = (X-X_0)/(X_{eq}-X_0)$, $\alpha$ is the shape parameter, correlated to the process initial rate, and $\beta$ is the scale parameter which corresponds to the time required for the sample to reach 63% of the equilibrium moisture content.

*2.5. Statistical analysis*

The models were fitted to the experimental data of water uptake versus hydration time by non-linear regression using the Solver tool of Microsoft Excel® (Microsoft Corporation, Redmond, USA). The parameters were identified using the generalized reduced gradient method (GRC Non-linear) of the same software. The effectiveness of the fit was evaluated using the adjusted determination coefficient ($R^2_{adj}$), residual plot analysis, and the normalized root mean square error (*NRMSE*) in order to determine the reliability of both models.

The influence of temperature and ultrasound nominal power on all ultrasound-assisted hydration treatments was evaluated by the analysis of variance (ANOVA) with a confidence interval of 95%, by using the software Minitab v.16.1.1 (Minitab Inc., State College, USA).

**3. Results and Discussion**

*3.1. Ultrasonic energy*

It is known that some energy could be dissipated during electrical conversion into effective acoustic energy. Therefore, the delivered ultrasonic energy was measured using the calorimetric method concerning the entire medium of hydration to provide a better estimation of the acoustic fields in the entire reactor. This provides important data to make adequate conclusions about the cavitation, not only at a specific point, but for the whole liquid.

The linear increase of temperature with sonication time provided values of d$T$/d$t$ to obtain $P_{US}$ of 51.12 W at 750 W and 84.68 W at 1500 W nominal power, as indicated in Fig. 3. The resulting average ultrasonic power density obtained by the relationship between the ultrasonic energy delivered and the volume of water (1000 mL) varied from 0.0495 to 0.0522 W/mL and from 0.0829 to 0.0864 W/mL at 750 W and 1500 W, respectively, at the range of temperatures studied. As this work took into account energy transfer throughout the liquid medium (not only at the point of maximum cavitation), relative lower delivered energy was obtained compared to other studies (Polachini et al., 2017; Ozuna et al., 2014). The increase in temperature was less intense as the temperature recordings were taken closer to the reactor wall and away from the sonotrode tip, bringing down the maximum conversion rates that are usually concentrated right below the sonotrode. At the position of maximum increase, temperature increases would result in average acoustic powers of 75.17 W at 750 W and 123.26 W at 1500 W if only this punctual increase is taken into account. However, the resulting actual ultrasonic density was similar or higher than other similar studies involving hydration assisted by bath type ultrasound (Patero and Augusto, 2015; Yildirim et al., 2011), even considering the integral method used to obtain real acoustic power.

The results indicated that acoustic energy was significantly affected by the intensity of applied nominal power, but not affected by the temperature of the water. The average conversion ratio between the delivered ultrasonic energy and the nominal power ($P_{US}/P_N$) was 6.82% for 750 W and 5.64% for 1500 W nominal power, showing a reduction in the conversion ratio with the increase in the nominal power. This low conversion ratio is due to high volumes treated with high-action and, consequently, to the presence of many regions of low-action ultrasound. As all these regions were considered in the calculation, the little punctual $dT/dt$ near to the reactor wall caused a decrease in the resulting average $dT/dt$. The reduction in the yield of conversion can be explained by the higher energy losses to the medium due to attenuation and higher levels of cavitation at higher nominal ultrasonic power. Gogate et al. (2011) stated that, above a certain power limit, a number of cavitation bubbles might cluster near the tip of the sonotrode. This can cause disturbances in this region of the medium, leading to difficulties for energy transfer processes.

According to Sala et al. (1995), reaction rates in ultrasound-assisted processes may be reduced as the medium temperature increases due to the exponential increase in vapor pressure and the reducing number of microbubbles formed, resulting in fewer collapses due to cavitation and less energy transmitted. However, Raso et al. (1999) found that the amount of energy delivered by the ultrasound waves can be reduced drastically above 70 ºC. This was found to be true in the current study, which observed no influence of temperature on the transmitted energy at a range of temperatures from 10–25 ºC.  Although temperature might not affect energy conversion into acoustic power, it had a significant positive effect on mass transfer as reported by the grain hydration. This means that the yield of power conversion is not affected if hydration is enhanced by increasing temperature.

*3.2. Ultrasound-assisted barley hydration*

The experimental steeping curves of barley are shown in Figure 4, together with the fitted mathematical models. The increase in temperature and ultrasonic power applied for the total of 4.5 hours reduced the time required to reach the necessary moisture levels for the germination of barley seeds by conventional processes (approximately 75 g/100 g d.b.) (Mayolle et al., 2012). After 24 hours, the steeping performed at 10 °C did not reach 75 g/100 g (d.b.) of moisture, even with the application of intermittent high-intensity ultrasound. At 15 °C, the hydration performed without the application of high-intensity ultrasound also failed to reach the necessary moisture level for germination, even after 24 hours (Fig. 4). However, by applying high-intensity ultrasound at 0.0511 W/mL actual volumetric power at a temperature of 15 $^{o}$C, 24 hours of hydration was enough for the barley seeds to reach the required moisture for germination, while only 20 hours was needed at 0.0847 W/mL actual volumetric power. The steeping conducted at

20 °C assisted by 0.0511 W/mL and 0.0847 W/mL of acoustic density showed reductions of 29.2% and 37.5% respectively in the amount of time required to reach the right moisture levels without sonication. At 25 °C, the time reduction was even higher: 33.3% and 44.5% quicker with the application of ultrasound at 0.0511 W/mL and 0.0847 W/mL respectively.

The results of this study have shown a significant reduction in the amount of time required for barley steeping by applying high-intensity ultrasound. Montanuci et al. (2013) showed that hydrated barley reached 75 g/100 g (d.b.) moisture only at 20 °C or above, when investigating hydration in five cultivars of barley during a period of 32 hours using a mechanically stirred medium (sample and water) at temperatures between 10 and 35 °C. Based on this data, ultrasound can be used as an efficient tool to enhance the mass transfer and reduce the conventional steeping time instead of using energy to heat and/or to maintain the average temperature. At industrial plants which are already supplied with warm water, the time previously required for the hydration phase can be reduced by almost half by using power ultrasound. It is also worth reiterating the statement of Miano et al. (2005) that the ultrasonic treatment is not only capable of reducing the required time for steeping without affecting the germination and vigor of the seeds, but it can also improve the germination rate of the seeds.

Regarding the fitting procedure, the residual plots for the Peleg model and the Weibull-exponential model are presented in Figure 5. In general, both models were well-fitted to the experimental values. The fitting performance can also be verified through the statistical parameters $R^2_{adj}$ and *NRMSE* presented in Table 1. The Weibull-exponential model, in particular, was better fitted as it resulted in lower *NRMSE* and higher adjusted determination coefficients, even presenting more parameters than the Peleg model. The Peleg model showed higher negative values for shorter times and a tendency to increase until reaching a maximum positive value and then decreasing for longer times of hydration. The Weibull-exponential model showed similar behavior, but with residual values lower than the Peleg model. Although these models presented different degrees of accuracy, the interpretation of the respective parameters provides important information.

The constant rate parameter values of the Peleg model are in close agreement with the ones published by Montanuci et al. (2013) for five different varieties of barley seeds, which varied from 0.0273 to 0.0670 h·100 g/g (d.b.) at a range of temperatures between 10 and 35 ºC without ultrasound. This parameter ($k_1$), related to the initial rate of hydration, was not significantly influenced by the temperature of the process ($p$=0.075). The application of high-intensity ultrasound reduced $k_1$ ($p$<0.001), or in other words the application of ultrasound increased the initial rate of hydration. The constant of capacity parameter ($k_2$) behaved as reported by these same authors. It was affected by temperature and the intensity of the ultrasound applied, in such a way that the higher the temperature combined with the application of ultrasound, the lower the value of $k_2$ ($p < 0.001$). Moreover, the hydration of other grains such as sorghum assisted by acoustic ultrasonic density of 0.026 W/mL was able to reduce the constant rate ($k_1$) of the Peleg model from 0.029 to 0.025 h·100 g/g (d.b.) and reduce the capacity constant ($k_2$) from 0.0286 to 0.02574 100 g/g (d.b.) at 25 °C (Patero and Augusto, 2015). The fitting parameters are also in accordance with Ghafoor et al. (2014), who applied ultrasound to the hydration of navy beans at 16 °C, describing a reduction in the Peleg model's $k_1$ from 0.029 to 0.016 h·100 g/g (d.b.) for the control and sonicated processes respectively.

Regarding the Weibull-exponential model, the shape parameter ($\alpha$), related to the initial rate of water absorption, was affected ($p$<0.001) by temperature and the application of high-intensity ultrasound. The parameter increased with rising temperatures and decreased with the increasing ultrasonic power. Similarly, as shown by the Peleg model, applying ultrasound increases the initial rate of water absorption. The scale parameter ($\beta$), which represents the time needed to absorb 63% of the total water absorbed at the equilibrium (Cunningham et al., 2007), was not significantly affected by the application of high-intensity ultrasound ($p$>0.781), but decreased as the temperature was increased ($p$<0.001). As the treatments assisted by ultrasound caused an increase in the grain's equilibrium moisture content, the required time to absorb 63% of $X_{eq}$ is supposed to be the same. However, temperature seemed to have a positive effect on the water absorption rate. The resulting parameters are in accordance with the ones reported by Montanuci et al. (2015), who also fitted the experimental data of barley hydration to the Weibull-exponential model. They obtained values from 0.41 to 0.59 for the shape parameter ($\alpha$) and values of 5.83 to 8.87 h for the scale parameter ($\beta$) at the same range of hydration temperatures.

The values of equilibrium moisture ($X_{eq}$) estimated by the Weibull-exponential model were higher than the values estimated by the Peleg model for all experimental conditions. The effectiveness of fit ($R^2_{adj}$ and *NRMSE*) indicated that the values of $X_{eq}$ estimated by the Weibull-exponential model are more reliable than their Peleg counterparts. Both the increase in process temperature and in the intensity of the applied ultrasound resulted in higher ($p$<0.05) equilibrium moisture. The observed increase ranged from 5.50% to 14.93%, with greater differences at lower temperatures studied. The main complex mechanism involved in conventional non-stirred barley steeping is water diffusion, which controls grain hydration. However, ultrasound has shown itself to be a promising technology for enhancing water transport and $X_{eq}$. The alternative compression and expansion cycles act similarly to a sponge, which contracts and expands repeatedly as ultrasonic waves travel through the tissue. This phenomenon can keep the micro-channels free, facilitating mass transfer (Garcia-Noguera et al., 2010; Gafhoor et al., 2014; Patero and Augusto, 2015). In addition, the wave propagation can generate micro-channels in the tissue due to the mechanical stress and this can improve the hydration. Nevertheless, further investigation is encouraged concerning the possible effects of high-intensity ultrasonic waves on enzyme formation and germination rates in barley malt production.

## 4. Conclusions

This study investigated the effect of temperature and high-intensity ultrasound on barley hydration. The ultrasonic energy delivered was determined using the calorimetric method taking into account different points of the reactor. The average acoustic power was not significantly affected by temperature in the range investigated, however reductions (6.82% to 5.64% on average) in the conversion ratio were observed as the nominal power increased from 750 to 1500 W, possibly due to higher attenuation of the ultrasonic waves at higher intensities. Ultrasound-assisted barley hydration showed higher water uptake rates at the beginning of the process and then asymptotic behavior towards maximum moisture levels for longer hydration times, corresponding to the equilibrium moisture. The water uptake rate and the equilibrium moisture content increased with the ultrasound power, as showed by the Peleg and Weibull-exponential models. The Weibull-exponential model presented a better adjustment ($R^2_{adj}$>0.953 and *NRMSE*<5%) to the data for barley hydration assisted by ultrasound, although the Peleg model also represents an alternative as it is so easy to use. Ultrasound application reduced the time required to achieve the necessary moisture level for barley germination from 29 to 44% at relatively low temperatures (20 and 25 °C respectively), compared to the treatment without ultrasound, indicative of an alternative technology for improving barley hydration.

## Nomenclature

| | | |
|---|---|---|
| $c_p$ | Water heat capacity | J/kg K |
| $D_{US}$ | Acoustic density | W/mL |
| $k_1$ | Constant rate parameter of the Peleg model | h·100 g/g d.b. |
| $k_2$ | Constant capacity parameter of the Peleg model | 100 g/g d.b. |
| $m$ | Mass of sample | g |
| $m_0$ | Initial mass of sample | g |
| *NRMSE* | Normalized root-mean-square error | % |
| $p$ | *p*-value | - |
| $P_N$ | Nominal power of the ultrasound generator | W |
| $P_{US}$ | Ultrasonic energy delivered | W |
| $R^2_{adj}$ | Adjusted determination coefficient | - |
| $T$ | Average temperature of water | ºC |

| | | |
|---|---|---|
| $t$ | Time | s, h |
| $T_0$ | Initial temperature of the water | ºC |
| $t_0$ | Time at the beginning of the first interval | S |
| $T_{US}$ | Water temperature at the end of the first interval | ºC |
| $t_{us}$ | Time at the end of first interval | S |
| $V$ | Volume of the hydration unit | $m^3$ |
| $X$ | Moisture content | g/100 g d.b. |
| $X_0$ | Initial moisture content | g/100 g d.b. |
| $X_{eq}$ | Equilibrium moisture | g/100 g d.b. |
| $\alpha$ | Shape parameter of the Weibull-exponential model | |
| $\rho$ | Water density | $kg/m^3$ |
| $\beta$ | Scale parameter of the Weibull-exponential model | h |

## Acknowledgements

The authors are thankful to FAPESP (Fundação de Amparo à Pesquisa do Estado de São Paulo, Project Numbers 2013/17497-5; 2017/06518-2); and CAPES (Coordenação de Aperfeiçoamento de Pessoal de Nível Superior) for the financial support provided to carry out this research.

## References

AOAC. (2005). Official methods of analysis of AOAC International. AOAC International.

Borges, C. W. C.; Jorge, L. M. M.; Jorge, R. M. M. (2017). Kinetic modeling and thermodynamic properties of soybean cultivar (BRS257) during hydration process. Journal of Food Process Engineering 40(6), 1-8.

Brookes, P.A., Lovett, D.A., MacWilliam, I.C. (1976). The steeping of barley. A review of the metabolic consequences of water uptake, and their practical implications. Journal of the Institute of Brewing 82, 14-26.

Cárcel, J.A., García-Pérez, J.V., Benedito, J., Mulet, A. (2012). Food process innovation through new technologies: Use of ultrasound. Journal of Food Engineering 110, 200-207.

Carvalho, G.R., Chenlo, F., Moreira, R., Telis-Romero, J., (2015). Physicothermal Properties of Aqueous Sodium Chloride Solutions. Journal of Food Process Engineering 38, 234-242.

Chivate, M. M., Pandit, A. B. (1995). Quantification of cavitation intensity in fluid bulk. Ultrasonics Sonochemistry 2, S19-S25.

Cunha, L. M. Oliveira, F. A. R. Oliveira, J. C. (1998). Optimal experimental design for estimating the kinetic parameters of processes described by the Weibull probability distribution function. Journal of Food Engineering 37, 175-191.

Cunningham, S.E., McMinn, W.A.M., Magee, T.R.A., Richardson, P.S. (2007). Modelling water absorption of pasta during soaking. Journal of Food Engineering 82, 600-607.

Fan, D., Huang, L., Li, B. Huang, J., Zhao, J., Yan, B., Zhou, W., Zhang, W., Zhang, H. (2017). Acoustic intensity in ultrasound field and ultrasound-assisted gelling of surimi. LWT - Food Science and Technology 75, 497-504

Garcia-Noguera, J., Oliveira, F.I.P., Gallão, M.I., Weller, C.L., Rodrigues, S., Fernandes, F.A.N. (2010). Ultrasound-Assisted Osmotic Dehydration of Strawberries: Effect of Pretreatment Time and Ultrasonic Frequency. Drying Technology 28, 294-303.

García-Pérez, J.V., Cárcel, J.A., Benedito, J., Mulet, A. (2007). Power Ultrasound Mass Transfer Enhancement in Food Drying. Food and Bioproducts Processing 85, 247-254.

Ghafoor, M., Misra, N.N., Mahadevan, K., Tiwari, B.K. (2014). Ultrasound assisted hydration of navy beans (Phaseolus vulgaris). Ultrasonics Sonochemistry 21, 409-414.

Gogate, P. R., Sutkar, V. S., Pandit, A. B. (2011). Sonochemical reactors: Important design and scale up considerations with a special emphasis on heterogeneous systems. Chemical Engineering Journal 166 (3), 1066-1082.

Lewis, M. J.; Young, T. W. (2001). Brewing. Springer Science & Business Media, 2 ed.

Liang, H. (1993). Modelling of ultrasound assisted and osmotically induced diffusion in plant tissue. Ph.D. Thesis. Purdue University, USA.

Margulis, M. A., Margulis, I. M. (2003). Calorimetric method for measurement of acoustic power absorbed in a volume of a liquid. Ultrasonics Sonochemistry 10, 343-345.

Mayolle, J.E., Lullien-Pellerin, V., Corbineau, F., Boivin, P., Guillard, V. (2012). Water diffusion and enzyme activities during malting of barley grains: A relationship assessment. Journal of Food Engineering 109, 358-365.

Miano, A.C., Forti, V.A., Abud, H.F., Gomes-Junior, F.G., Cicero, S.M., Augusto, P.E.D. (2015). Effect of ultrasound technology on barley seed germination and vigour. Seed Science and Technology 43, 297-302.

Montanuci, F.D., Jorge, L.M.d.M., Jorge, R.M.M. (2013). Kinetic, thermodynamic properties, and optimization of barley hydration. Food Science and Technology (Campinas) 33, 690-698.

Montanuci, F.D., Jorge, L.M.M., Jorge, R.M.M. (2015). Effect of time and temperature on the hydration process of barley grains. Heat and Mass Transfer 51, 363-372.

Ozuna, C., Puig, A., Garcia-Perez, J. V., Cárcel, J. A, (2014). Ultrassonically enhanced desalting of cod (Gadus morhua). Mass Transport kinetics and structural changes. LWT - Food Science and Technology, 59 (1), 130-137.

Patero, T., Augusto, P.E.D. (2015). Ultrasound (US) enhances the hydration of sorghum (Sorghum bicolor) grains. Ultrasonics Sonochemistry 23, 11-15.

Peleg, M. (1988). An Empirical Model for the Description of Moisture Sorption Curves. Journal of Food Science 53, 1216-1217.

Polachini, T. C., Carvalho, G. R., Telis-Romero, J. (2017). Determination of acoustic fields in acidic suspensions of peanut shell during pretreatment with high-intensity ultrasound. Brazilian Journal of Chemical Engineering 34(2), 385-394.

Raso, J., Mañas, P., Pagán, R., Sala, F.J. (1999). Influence of different factors on the output power transferred into medium by ultrasound. Ultrasonics Sonochemistry 5, 157-162.

Resio, A. C.; Aguerre, R. J.; Suarez, C. (2006). Hydration kinetics of amaranth grain. Journal of Food Engineering 72(3), 247-253.

Sala, F.J., Burgos, J., Condón, S., Lopez, P., Raso, J. (1995). Effect of heat and ultrasound on microorganisms and enzymes, in: Gould, G.W. (Ed.), New Methods of Food Preservation. Springer US, Boston, MA, pp. 176-204.

Samaras, T. S.; Camburn, P. A.; Chandra, S. X.; Gordon, M. H.; Ames, J. M. (2005). Antioxidant properties of kilned and roasted malts. Journal of Agricultural and Food Chemistry 53(20), 8068-8074.

Yaldagard, M., Mortazavi, S.A., Tabatabaie, F. (2008). Application of Ultrasonic Waves as a Priming Technique for Accelerating and Enhancing the Germination of Barley Seed: Optimization of Method by the Taguchi Approach. Journal of the Institute of Brewing 114, 14-21.

Yildirim, A. Öner, M. D. Bayram, M. (2011). Fitting Fick´s model to analyze water diffusion into chickpeas during soaking with ultrasound treatment. Journal of Food Engineering 104, 134-142.

Table 1 – Parameters of the Peleg model and the Weibull exponential model adjusted to the experimental data of high-intensity ultrasound-assisted barley hydration.

Peleg's model

| ***T*** (°C) | $D_{US}$ (W/mL) | $k_1$ (h·100 g/g d.b.) | $k_2$ (100 g/g d.b.) | $X_{eq}$ (g/100 g d.b.) | $R^2_{adj}$ (-) | ***NRMSE*** (%) |
|---|---|---|---|---|---|---|
| | 0 | 0.0462 ± 0.0037$^{Aa}$ | 0.0165 ± 0.0000$^{Aa}$ | 70.72 ± 0.05$^{Db}$ | 0.895 | 8.29 |
| 10 | 0.0511 | 0.0411 ± 0.0016$^{Ab}$ | 0.0152 ± 0.0004$^{Ab}$ | 75.81 ± 1.79$^{Da}$ | 0.954 | 5.66 |
| | 0.0847 | 0.0336 ± 0.0008$^{Ac}$ | 0.0152 ± 0.0002$^{Ab}$ | 75.80 ± 0.91$^{Da}$ | 0.958 | 5.49 |
| | 0 | 0.0488 ± 0.0017$^{Aa}$ | 0.0152 ± 0.0002$^{Ba}$ | 75.62 ± 0.68$^{Cb}$ | 0.935 | 6.61 |
| 15 | 0.0511 | 0.0379 ± 0.0026$^{Ab}$ | 0.0141 ± 0.0003$^{Bb}$ | 80.91 ± 1.71$^{Ca}$ | 0.955 | 5.41 |
| | 0.0847 | 0.0363 ± 0.0014$^{Ac}$ | 0.0138 ± 0.0001$^{Bb}$ | 82.54 ± 0.64$^{Ca}$ | 0.957 | 5.47 |
| | 0 | 0.0407 ± 0.0012$^{Aa}$ | 0.0141 ± 0.0001$^{Ca}$ | 80.98 ± 0.50$^{Bb}$ | 0.933 | 6.33 |
| 20 | 0.0511 | 0.0398 ± 0.0002$^{Ab}$ | 0.0126 ± 0.0002$^{Cb}$ | 89.29 ± 1.37$^{Ba}$ | 0.953 | 5.72 |
| | 0.0847 | 0.0362 ± 0.0013$^{Ac}$ | 0.0122 ± 0.0004$^{Cb}$ | 91.95 ± 2.70$^{Ba}$ | 0.958 | 5.47 |
| | 0 | 0.0459 ± 0.0011$^{Aa}$ | 0.0122 ± 0.0003$^{Da}$ | 91.89 ± 1.91$^{Ab}$ | 0.948 | 5.98 |
| 25 | 0.0511 | 0.0357 ± 0.0009$^{Ab}$ | 0.0119 ± 0.0002$^{Db}$ | 94.28 ± 1.41$^{Aa}$ | 0.963 | 5.16 |
| | 0.0847 | 0.0338 ± 0.006$^{Ac}$ | 0.0116 ± 0.0002$^{Db}$ | 96.05 ± 1.32$^{Aa}$ | 0.969 | 4.75 |
| Temperature effects | | $^{A}p$-value = 0.075 | $^{ABCD}p$-value < 0.001 | $^{ABCD}p$-value < 0.001 | | |
| | Power effects | $^{abc}p$-value < 0.001 | $^{ab}p$-value < 0.001 | $^{ab}p$-value < 0.001 | | |

Weibull exponential model

| ***T*** (°C) | $D_{US}$ (W/mL) | $\alpha$ (-) | $\beta$ (h) | $X_{eq}$ (g/100 g d.b.) | $R^2_{adj}$ (-) | ***NRMSE*** (%) |
|---|---|---|---|---|---|---|
| | 0 | 0.480 ± 0.044$^{Ca}$ | 8.36 ± 0.02$^{ABa}$ | 80.34 ± 1.31$^{Dc}$ | 0.953 | 5.00 |
| 10 | 0.0511 | 0.448 ± 0.018$^{Cb}$ | 10.18 ± 0.29$^{ABa}$ | 89.12 ± 1.44$^{Db}$ | 0.992 | 2.07 |
| | 0.0847 | 0.377 ± 0.008$^{Cb}$ | 9.84 ± 0.13$^{ABa}$ | 91.90 ± 1.12$^{Da}$ | 0.997 | 1.09 |
| | 0 | 0.524 ± 0.020$^{Ba}$ | 9.75 ± 0.19$^{BCa}$ | 85.98 ± 1.01$^{Cc}$ | 0.976 | 3.74 |
| 15 | 0.0511 | 0.465 ± 0.021$^{Bb}$ | 8.69 ± 0.06$^{BCa}$ | 92.50 ± 2.96$^{Cb}$ | 0.988 | 1.91 |
| | 0.0847 | 0.454 ± 0.005$^{Bb}$ | 8.38 ± 0.07$^{BCa}$ | 94.38 ± 0.71$^{Ca}$ | 0.994 | 1.84 |
| | 0 | 0.477 ± 0.007$^{Ba}$ | 8.36 ± 0.05$^{Ca}$ | 91.91 ± 0.98$^{Bc}$ | 0.972 | 3.42 |
| 20 | 0.0511 | 0.516 ± 0.012$^{Bb}$ | 8.32 ± 0.04$^{Ca}$ | 98.70 ± 0.41$^{Bb}$ | 0.989 | 2.79 |
| | 0.0847 | 0.490 ± 0.040$^{Bb}$ | 8.80 ± 1.01$^{Ca}$ | 104.00 ± 0.57$^{Ba}$ | 0.992 | 2.18 |
| | 0 | 0.612 ± 0.002$^{Aa}$ | 8.29 ± 0.03$^{Da}$ | 97.24 ± 1.70$^{Ac}$ | 0.976 | 4.03 |
| 25 | 0.0511 | 0.524 ± 0.007$^{Ab}$ | 7.73 ± 0.03$^{Da}$ | 102.45 ± 1.03$^{Ab}$ | 0.992 | 2.12 |
| | 0.0847 | 0.513 ± 0.010$^{Ab}$ | 7.46 ± 0.05$^{Da}$ | 104.23 ± 0.58$^{Aa}$ | 0.996 | 1.57 |
| Temperature effects | | $^{ABC}p$-value < 0.001 | $^{ABCD}p$-value < 0.001 | $^{ABCD}p$-value < 0.001 | | |
| | Power effects | $^{ab}p$-value < 0.001 | $^{a}p$-value = 0.781 | $^{abc}p$-value < 0.001 | | |

*Capital and small letters represent significant (different letter) or non-significant (same letter) differences between the model parameters with relation to temperature (capital) and actual acoustic density (small), respectively, with a confidence interval of 95%.

**Figure Captions**

Figure 1 – Experimental set-up for barley hydration and for measuring the ultrasonic energy delivered.

Figure 2 – (a) Schematic of energy balance to determine the ultrasonic energy; (b) Thermocouples positions inside the hydration vessel.

Figure 3 – Ultrasonic energy delivered (mean ± SD of the experiments in triplicate) as function of nominal power and temperature.

Figure 4 – Experimental data (mean ± SD of the experiments in duplicate) of barley hydration without ultrasound (■), with ultrasound at 0.0511 W/mL (□) and 0.0847 W/mL (○) actual power density; and fitted models: Peleg (grey line) and Weibull exponential (dashed line) and moisture content of 75 g/100 g d.b. necessary for barley germination after conventional hydration (horizontal line).

Figure 5 – Residual plot of the Peleg model and the Weibull exponential model for barley hydration without ultrasound (■), at 0.0511 W/mL (□) and 0.0847 W/mL (○) actual power density.

Figure 1

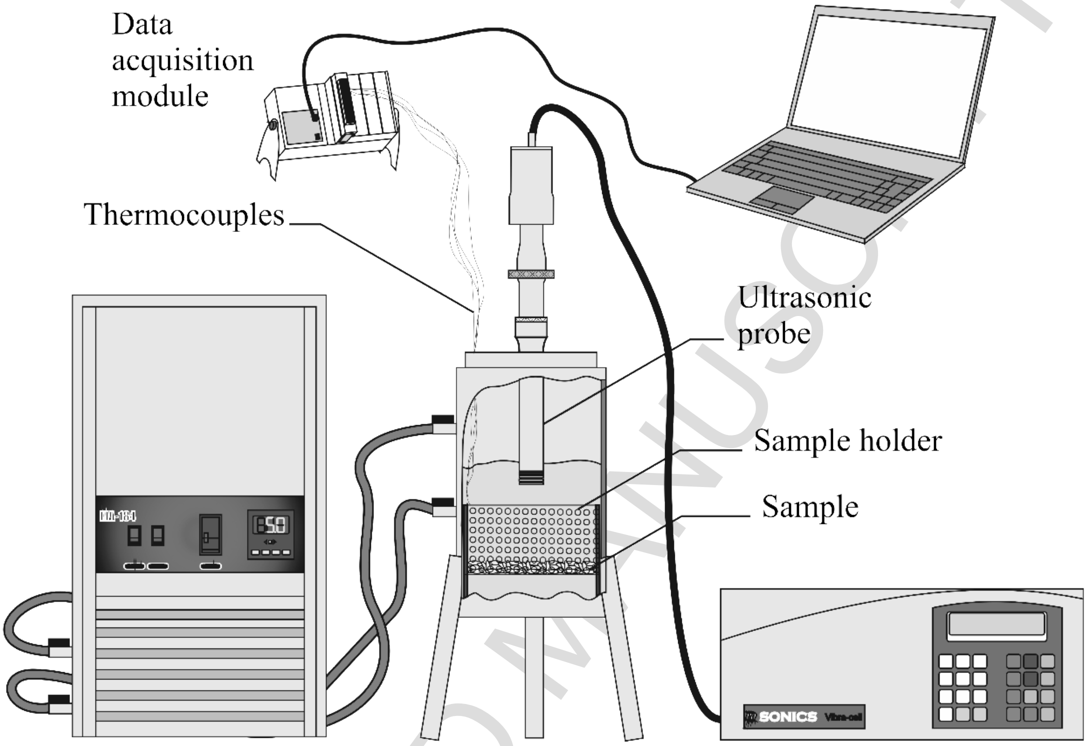
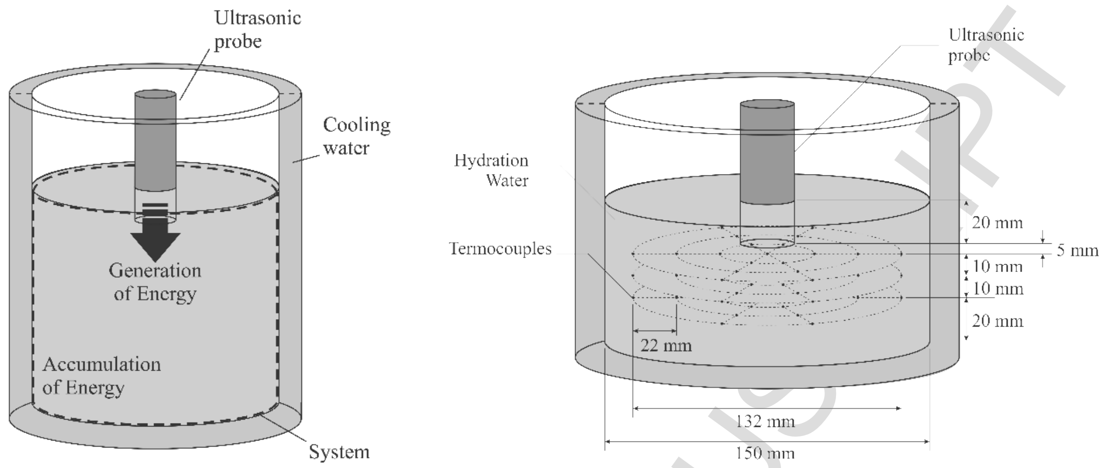
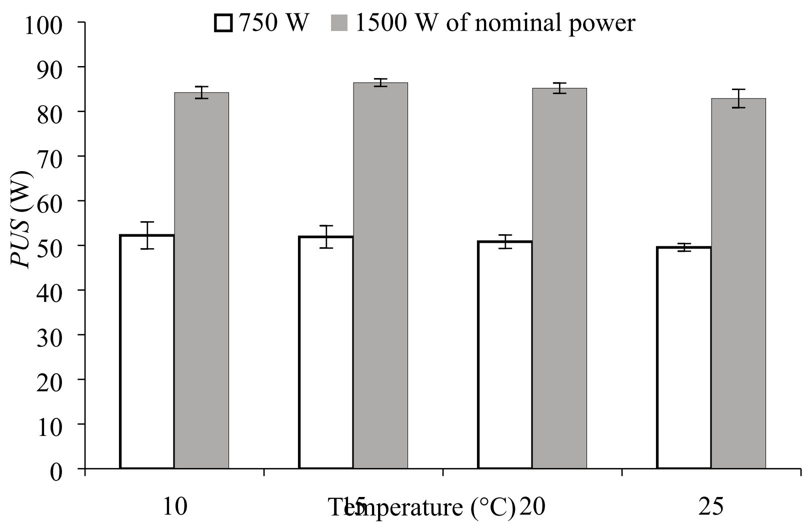

Data
acquisition
module
Thermocouples
Ultrasonic
probe
Sample holder
Sample
Ultrathermostatized
bath
Stainless steel
jacketed vessel
Ultrasonic
generator
SONICS

Figure 2


Ultrasonic probe
Cooling water
Generation of Energy
Accumulation of Energy
System
Ultrasonic probe
Hydration Water
Termocouples
20 mm
5 mm
10 mm
10 mm
20 mm
22 mm
132 mm
150 mm

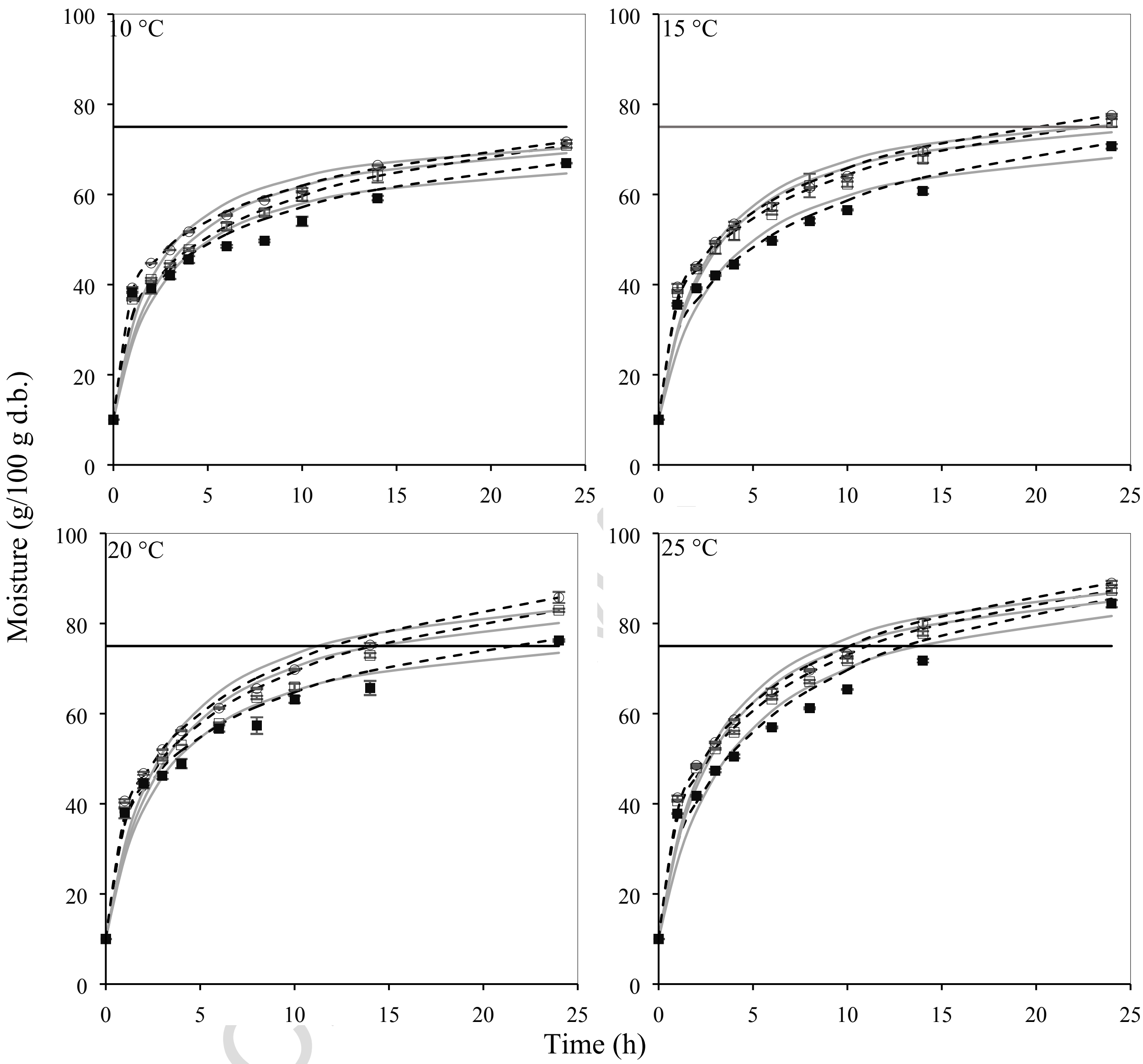
(a) (b)

Figure 3

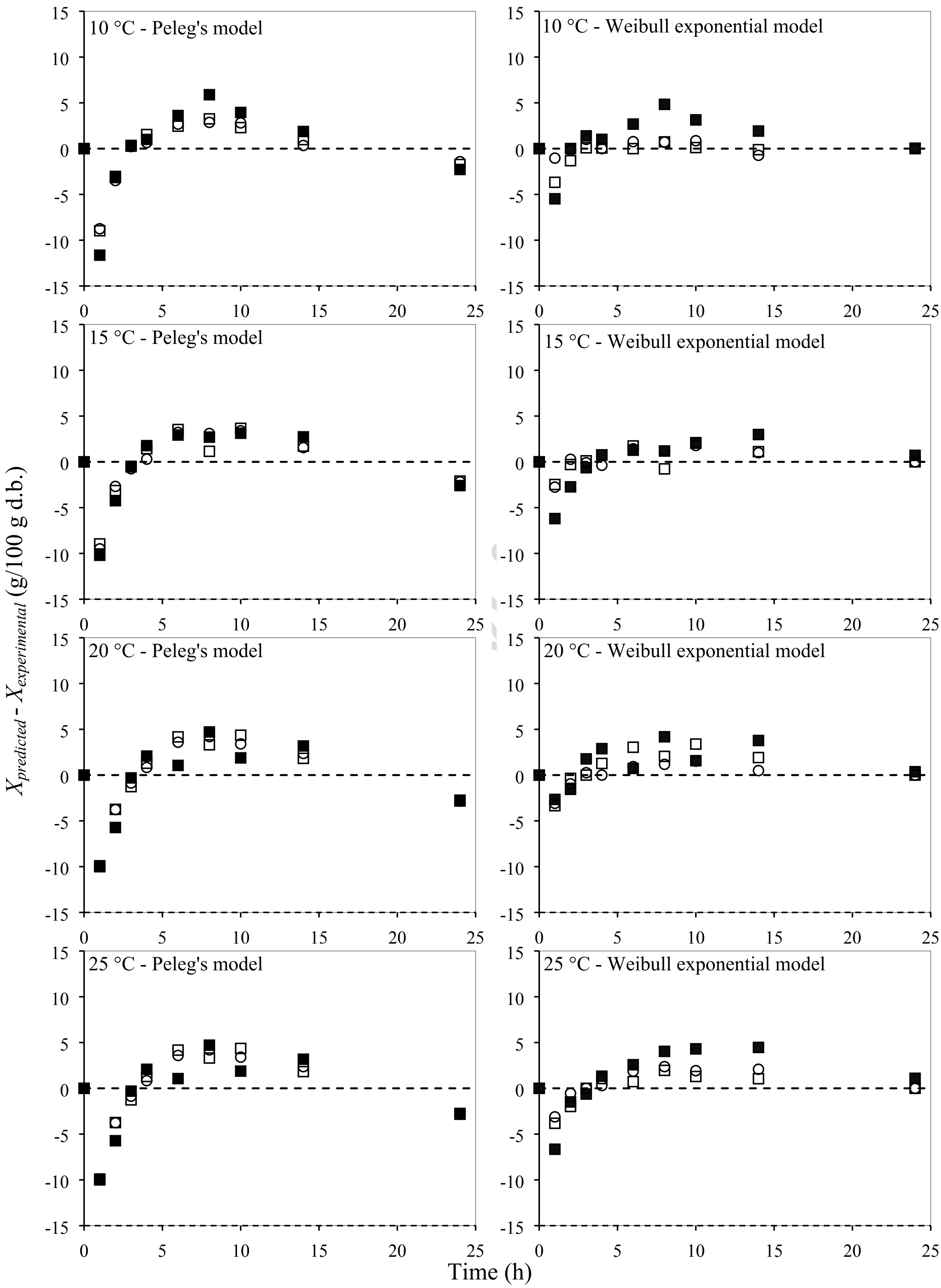


750 W
1500 W of nominal power
PUS (W)
0
10
20
30
40
50
60
70
80
90
100
10
15
20
25
Temperature (°C)

553 Figure 4


10 °C
15 °C
20 °C
25 °C
Moisture (g/100 g d.b.)
Time (h)
0
20
40
60
80
100
0
5
10
15
20
25

562 Figure 5




563

**Highlights**

> The effect of temperature and input power on barley hydration was studied.

> A new approach to determine the acoustic fields was reported.

> Hydration processes were well-fitted to both the Weibull and Peleg models.

> Ultrasound enhanced both water uptake and equilibrium moisture content.

> Ultrasound reduced the hydration time required for barley germination by up to 44%.