ULTRASOUND STUDY OF WHEAT FLOUR PROPERTIES

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Running title: Ultrasound flour analysis

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ABSTRACT

In this work, the wheat flour properties are investigated using ultrasound techniques. Moreover, the flour samples were characterized also by means of well established techniques such as protein content, Alveograph and Mixolab®. A set of 35 dough samples, made of wheat flours with diverse physical and quality properties, were studied. The obtained results shown that ultrasound measurements can detect changes in the dough consistency induced by proteins and also by gelatinization of the starch. Furthermore, ultrasound measurements can be related to parameters indicative of the proteolytic degradation or softening of the dough due to protease activity. Thus, ultrasound can be considered a low cost and rapid tool, complementary to conventional test, for wheat flour characterization.

Key words: flour, bread dough, ultrasound, NDT, rheology.
1. Introduction

In many bakeries, especially in medium-small plants, the quality control of the bread dough is based on sensorial tests. A trained operator stresses the dough sample in order to determine its strength and elasticity. There are other methods with more scientific basis, such as fundamental and empirical rheology, which have been extensively reported in the literature [1-6]. Fundamental rheology can give information about the structure and fundamental physical properties of the dough while empirical rheology may offer useful practical information for the mixing and baking process. Furthermore, determining the dough properties is a method to study the characteristics of the flour that have been used in its preparation.

Ultrasound can also provide relevant information to the flour and bread dough characterization. Acoustic properties of materials are related to their elasticity, consistency and other physical properties. Thus, ultrasound parameters could be related to properties of the dough that could be relevant to the bakery industry. In addition, ultrasound systems are usually fast, relatively cheap and hygienic. Therefore, ultrasound can be considered a low cost complementary analysis in order to characterize both the bread dough and the flour used to prepare it.

There are some published works that deals with the ultrasound analysis of flour or bread dough. Some of these works show the effect that on the ultrasound parameters has the change of some properties of the bread dough or its processing, such as its moisture, mixing time and rest time [7-9]. The acoustic properties during that post-mixing stage of the dough are also shown in [10-13]. Moreover, the influence of gas bubbles in the acoustic properties of bread dough have been also studied [14]. Recent works study the acoustic parameters of dough with the operation conditions of extrusion
processes [15]. Moreover, different aspects of the ultrasound analysis of bread dough are also shown in our previous works. The early articles mainly deal with the sensitivity to ultrasound to dough water content, flour characteristic and mixing work input [16]. In [17] the time-dependence of the mechanical properties of dough for flour strength evaluation are studied. Moreover, the capability of ultrasound to discriminate fours to different purposes are shown in [18]. Recently, ultrasound analysis have been used to determine the properties of gluten free dough, such as rice flour dough [19]. These studies mainly deal with the analysis of the properties of the bread dough, at specific stages of preparation, with the variation/addition of some ingredients or with changes in the procedure of production.

However, in our knowledge there are little published about ultrasound study of the properties of flour. Only in our previous work [20] the use of ultrasound to determine some properties of the flour, mainly related to its quality, have been reported. In this work, the extended and detailed study of the flour features are performed with the analysis of 35 flours with diverse physical and quality attributes, tested by means of different well established techniques such as protein content, Zeleny sedimentation index, thousand kernel weight, hectolitre weight, Alveograph parameters, Mixolab® parameters and also by ultrasound techniques.

2. Materials and methods

Materials

Thirty five bread wheat samples from different cultivars obtained from 2008 Spanish crop were provided by Spanish Association of Cereal Chemists (AETC). After appropriate cleaning, a 500 g sample lot of wheat kernels were tempered to 16.5 % moisture in a Chopin conditioner by adding the necessary amount of water. For the
tempering, wheat kernels were kept at 20–25°C for 16 hours and then milled in a laboratory mill (Chopin Tripette et Renaud, Paris, France) to obtain straight grade flour.

**Conventional measurements**

All flour samples were tested for thousand-kernel weight (TKW) by counting the number of seeds in 20 g of grain and reported as dry basis. Samples were also analyzed for moisture content, protein (N x 5.7) and hectoliter weight following the Approved methods 44-15A, 46-11A, and 55-10, respectively [21]. The Zeleny test indicative of the wheat proteins quality were determined according Approved method 56-60 [21]. The alveograph test (Chopin Technologies, Paris, France) following the Approved Method 54-30 [21] were run to assess the viscoelastic behaviour of the dough. The alveograph parameters were automatically recorded by Alveolink-NG software (Chopin Technologies, Paris, France) including maximum over-pressure or tenacity (P) needed to blow the dough bubble, the dough extensibility (L), the work input needed to inflate the dough or the deformation or energy (W) and the deformation curve (P/L) [22].

Dough mixing and pasting behaviour of the wheat flour samples were studied using the Mixolab® device (Chopin Technologies, Paris, France) following the ICC Standard Method 173 [23] using 50 g of flour for each measurement. This device measures the torque (expressed in Nm) in real time produced by passage of dough between the two kneading arms. For the assays, 50 g of rice flour were placed into the Mixolab® bowl and mixed with the amount of water needed for obtaining a dough consistency of 1.1 Nm, calculated for each flour in preliminary assays. Parameters obtained from the recorded curve give information about the protein stability subjected to mechanical and thermal constraint and both the gelatinization and gelling of starch. Those parameters included initial maximum consistency (Nm) (C1), stability (min) or
time until the loss of consistency is lower than 11% of the maximum consistency reached during the mixing, amplitude (Nm) or the bandwidth at C1 related to dough elasticity [24], minimum torque (Nm) or the minimum value of torque (Nm) produced by dough passage subjected to mechanical and thermal constraints (C2), peak torque (Nm) or the maximum torque produced during the heating stage (C3), the minimum torque during the heating period (Nm) (C4) and the torque (Nm) obtained after cooling at 50°C (C5). More information about recorded parameters can be found in [25; 26]. Results are the average of duplicate measurements.

Ultrasound measurements

Flour (10 g) was mixed with the required amount of water, according to the water absorption determined in the Mixolab®, for obtaining wheat doughs with relative constant consistency that were used for further ultrasound measurements.

The experimental set-up is depicted in Fig. 1. It consists of a signal generator (HP33120A for the generation of a 3 cycles of sine burst 100 kHz excitation signal), a power amplifier (AG series amplifier, T&C Power Conversion, Inc. Rochester, NY), two ultrasonic transducers with a resonant frequency of 100 kHz (Panametrics-Olympus, Japan) and a digital oscilloscope (LeCroy LT344). The resonant frequency of the shear transducers is in the low kHz range (low frequency) in order to reach the smaller possible attenuation. An additional amplifier with 60 dB gain (Panametrics-Olympus, Japan) is connected to the receiver transducer so as to amplify the weak received signals. A dough sample is placed between the two carefully aligned transducers. The phase and the amplitude of the transmitted signal at the receiver can be compared with those of the transmitter.
However, as changes in velocity and more attenuation are introduced at every interface between different acoustical impedance materials, measurements at several sample thicknesses have been carried out in order to keep the same number and type of interfaces. The multiple thickness measurements also allow the velocity and attenuation to be determined by eliminating baseline offsets in the time and amplitude measurements [14]. The methodology was as follows: the transducers were approached until a measurable received signal were observed in the oscilloscope and a first reference measurement was performed, then, without removing the dough from between the transducers’ surface, the dough was slowly compressed to a several sample thicknesses, at which the measurement of the amplitude and the variation in the time of flight to the reference measurement were carried out. The measurements were repeated for three subsamples of each dough sample. The attenuation and velocity values are the average of the obtained in the three measurements. In Fig. 2 (left) the measured amplitude as a function of sample thickness for an arbitrary/typical dough sample of 240 dB/cm attenuation is shown as example of a multiple thickness measurement. As can be seen, the decay of the amplitude with thickness fits with an exponential curve (R²=0.9998) which is in accordance with the theoretical expression of the amplitude of the signal at the receiving transducer $A$ placed at a distance $x$ related to the attenuation coefficient of the dough $\alpha$, by:

$$A_{(x)} = C + A_0 e^{-\alpha x}$$  

Eq. 1

where $C$ is a constant and $A_0$ is the amplitude of the ultrasound signal at position $x=0$. Thus, the attenuation value can be obtained accurately from Eq. 1 avoiding the effect of spurious phenomena and interface effects. The values obtained in the same experiment for the variation of time of flight with the distance between transducers can be observed...
in Fig. 2 (right), for the same sample with 150 m/s of velocity value. As is shown, the variation of $TOF$ is decreasing linearly with distance, fitting a linear regression with a coefficient of $R^2 = 0.9981$ of the following expression:

$$TOF = \frac{1}{v} \cdot x$$  \hspace{1cm} \text{Eq. 2}

where $TOF$ is the time of flight in s, $x$ the distance in m and $v$ the ultrasound velocity in m/s. The ultrasound velocity $v$ is obtained straightforward from that equation. As mentioned previously, using the multiple thickness measurement not only the effect of the interfaces is compensated, but also the effect of occasional interferences to the measurements can be thus minimized by the averaging of measurements that this measurement procedure performs.

Measurements were performed at room temperature. Dough temperature was measured to ensure differences in temperature were not influencing the results. Measurements were performed at room temperature and dough temperature was 30 ± 1°C.

**Statistical analysis**

Multivariate analysis (stepwise regressions) was performed using Statgraphics V.7.1 program (Bitstream, Cambridge, MN). Multivariate data handling provides information on the significant correlations within the different physical and rheological parameters. Principal component analysis (PCA) was also performed to determine the number of principal components that significantly ($p < 0.05$) discriminated wheat varieties.

**3. Results and Discussion**

First, ultrasound attenuation and velocity were measured for all dough samples, which had relative constant consistency to dismiss the effect of the water absorption on
the attenuation and velocity. Previous studies showed that both velocity and attenuation are very sensitive to dough water content [16; 20]. The values of velocity and attenuation for the doughs obtained from the 35 wheat varieties are plotted against each other in Fig. 3. As can be seen, the points tend to a diagonal disposition, which could mean that the attenuation and velocity in dough samples are inversely proportional parameters. Moreover, the dough samples intended for cookies, identified with G# in the plot, tend to the up left corner of the graph, with high values of attenuation and relatively low values of velocity. This is in agreement with previous works, where was explained that this disposition is related to weak flours [20]. On the opposite corner, with the medium-low values of attenuation and the highest values of velocity within the plotted range, many of the doughs intended for sliced bread can be found, identified with M# in the plot, which is known that are the strongest flours of the batch of samples. Furthermore, it can be seen also in Fig. 3 that the doughs intended for common bread production, labelled P# in the plot, tend to be situated along the diagonal of the plot, often overlapped with dough made of flour intended for other purposes. This may indicate the wide range of consistency that the doughs produced with bread flour from different varieties can attain, which can even be similar that those belonging to other kinds of flour.

The range of values obtained for each parameter used to assign the conventional properties is described in Table 1 and 2. Ultrasound attenuation and velocity were compared to conventional properties of both the flour and the dough samples by means of correlation analysis. The obtained results are shown in Table 3. The wide variety of wheats employed result in a set of flour samples with very diverse physical and rheological characteristics, which vary from one sample to other substantially.
Conventional parameters associated to the proteins quality (located on the left top of Table 3) were highly correlated. Regarding the ultrasound parameters, statistically significant (p<0.05) positive correlation were obtained between ultrasound velocity and the alveograph parameter W or the input energy of deformation. This result agree with previous findings of [20] that associated strong flours (high W) with high velocity values. It should be remarked that velocity was also highly correlated with the energy of deformation after two hours resting (W2h), parameter indicative of the proteolytic degradation or softening of the dough due to protease activity [27]. Therefore, the ultrasound measurement could be a useful and rapid tool to identify protease damaged flours.

Positive correlations were also found between velocity and some parameters from the Mixolab®, on the left bottom of Table 3. Particularly significant (p<0.05) were the correlations for development time, dough stability during mixing and dough softening (C2) thermally induced, the later has been attributed to the weakening of the protein network [26]. The combined effect of the mechanical shear stress and the temperature constraint produced a decrease in the dough consistency that has been related with the beginning of the protein destabilization and unfolding. Following the same tendency described above, the attenuation of the doughs subjected to mild heating showed the opposite behaviour than the velocity, and was negatively correlated with the dough weakening (C2) and the temperature at which that minimum consistency (T at C2) was reached.

Conversely, negative significant correlation was found between the velocity and the temperature at which the maximum viscosity during the starch gelatinization is reached (T at C3). In opposition, positive significant correlation was observed between the attenuation and the temperature at C3. Therefore, ultrasound measurements can
detect changes in the dough consistency induced by proteins but also by gelatinization of the starch.

The obtained levels of correlation are statistically significant and give some information to the physical properties of the dough samples. Moreover, the ultrasound parameters can be considered complementary measurements to the conventional parameters, which can help to the characterization of flour and bread dough when a future practical application of that ultrasonic measurement system intended for flour and dough characterization in industrial plants are desired.

To study the possible discrimination of the wheat doughs and to obtain the optimum classification of the batch of samples a principal component analysis was performed. The results of that analysis can be observed in Fig. 4, where the dough samples are identified with the value of the alveograph W parameter. The first and second principal components explained 35% and 13% of the variation respectively. As mentioned before, the dough samples tested were made from wheat varieties that can be grouped into three different kinds of flour, for cookies, bread and sliced bread preparation. In Fig. 4, dough samples with W values over 100 mostly appear in the left side of the plot, while samples with W under that value are in the right side. Thus, the alveograph W parameter can be used to discriminate doughs in two main groups, one with W<100, which can be considered for cookies preparation and other with W>100, that can be considered suitable for several types of bread production.

The level of correlation of the ultrasound measurements with conventional parameters were again studied but for each group of samples separately. The obtained results are shown in Table 4. As can be seen, the levels of correlation found are generally higher when the statistical analysis is performed on samples divided into two groups of similar physico-chemical properties than when all the set of samples are in
one group. For the dough samples elaborated using flours with W>100, usually used to produce common bread and sliced bread, a relatively high level of correlation between ultrasound attenuation and dough stability, pasting temperature and the temperature at the maximum viscosity during the starch gelatinization (T at C3) is found. Better correlation is obtained between these conventional parameters and ultrasound velocity. Furthermore, ultrasound velocity can also be correlated with conventional parameters which do not offer significant correlation with attenuation, such as the development time. The ultrasound velocity, thus, seems to be more sensitive than the attenuation to the properties of the dough when testing samples with relatively high consistency, prepared using strong flours.

On the other hand, for the dough samples elaborated using flours with W<100, mostly intended for cookies preparation, the ultrasound attenuation shows a relatively high correlation with development time, dough stability and absorption. Moreover, ultrasound attenuation also seems highly sensitive to C3 that corresponds to the maximum consistency of the dough after starch gelatinization. This correlation found in the flours with W<100 could be due to the predominant role of the starch in this type of flours that are very weak in proteins. Ultrasound velocity only presents higher correlation than attenuation with the temperature at minimum consistency (T at C2). Therefore, ultrasound attenuation generally presents more sensitivity than velocity to dough properties in samples with relatively low consistency, elaborated with weak flours.

Therefore, ultrasound could be more sensitive to some properties of flour and dough if the samples are classified in groups with certain similarity in some of their properties.
4. Conclusions

Ultrasound can be used as alternative low cost tool to characterize rapidly the quality of the wheat flours. Velocity shows very good correlation with the parameters that are associated to proteins quality, whereas for weak flours would be more appropriate to use the attenuation that seems to be better correlated to starch changes due to gelatinization.

The velocity was also highly correlated with parameters connected to the proteolytic degradation or softening of the dough due to protease activity. Therefore, the ultrasound measurement could be a useful and rapid tool to identify protease damaged flours.

Therefore, ultrasound could be more sensitive to some properties of flour and dough if the samples are classified into groups with certain similarity in some of their properties.

Acknowledgements

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References


Figure captions

Fig. 1: Ultrasound setup.

Fig. 2: Ultrasound amplitude (left) and time of flight (right) as a function of sample thickness for a dough sample with ultrasound attenuation and velocity of 240 dB/cm and 150 m/s respectively. The exponential regression (left) and linear regression (right) and their values of $R^2$ are also shown.

Fig. 3: Ultrasound attenuation and velocity for the batch of dough samples.

Fig. 4: PCA loadings of the set of dough samples. PC1 and PC2 loadings. Each dough sample is labeled with its Alveograph W value.
Tables

Table 1. Moisture content, TKW and alveographic parameters (P, L, W, and the values after two hours resting) of wheat samples.

Table 2. Mixolab characteristics of flours from different wheat varieties.

Table 3: Coefficient of significant correlations (P<0.05) between flour conventional parameters and ultrasound measurements.

Table 4: Coefficient of significant correlations (P<0.05) between flour conventional parameters and ultrasound measurements for flours with W>100 and W<100.
Figure 1.
Figure 2

\[ R^2 = 0.9998 \]

\[ R^2 = 0.9981 \]
Figure 3.
Figure 4.
Table 1. Moisture content, TKW and alveographic parameters (P, L, W, and the values after two hours resting) of wheat samples.

<table>
<thead>
<tr>
<th></th>
<th>Moisture content (%)</th>
<th>Protein (%)</th>
<th>TKW (g)</th>
<th>P (mm H₂O)</th>
<th>L (mm)</th>
<th>W (x 10⁻⁴ J)</th>
<th>P/L</th>
<th>W₂h (x 10⁻⁴ J)</th>
<th>L₂h (mm)</th>
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<td>35</td>
<td>35</td>
<td>35</td>
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<td>35</td>
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<tr>
<td>Average</td>
<td>9.6</td>
<td>12.2</td>
<td>34</td>
<td>55</td>
<td>92</td>
<td>161</td>
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<td>87</td>
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<td>6</td>
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<td>12</td>
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<td>Minimum</td>
<td>7.9</td>
<td>8.6</td>
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<td>18</td>
<td>36</td>
<td>42</td>
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<td>107</td>
<td>110</td>
<td>346</td>
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<td>302</td>
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Table 2. Mixolab characteristics of flours from different wheat varieties.

<table>
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<th></th>
<th>Development time (min)</th>
<th>C1 (Nm)</th>
<th>Stability (min)</th>
<th>Amplitude (Nm)</th>
<th>T at C2 (°C)</th>
<th>C2 (Nm)</th>
<th>Stability T pasting (°C)</th>
<th>C3 (Nm)</th>
<th>T at C3 (°C)</th>
<th>C4 (Nm)</th>
<th>C5 (Nm)</th>
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<td>Average</td>
<td>3.8</td>
<td>1.12</td>
<td>6.3</td>
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<td>53.7</td>
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<td>0.00</td>
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<td>0.03</td>
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<td>1.5</td>
<td>0.05</td>
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<td>12.6</td>
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<td>0.87</td>
<td>10.8</td>
<td>1.27</td>
<td>3.56</td>
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Table 3: Coefficient of significant correlations (P<0.05) between flour conventional parameters and ultrasound measurements.

<table>
<thead>
<tr>
<th></th>
<th>W (x 10^4 J)</th>
<th>P/L (x 10^-4 J)</th>
<th>W2h (mm)</th>
<th>L2h (mm)</th>
<th>Development time (min)</th>
<th>stability (min)</th>
<th>C2 (Nm)</th>
<th>pasting T° (°C)</th>
<th>C3 (Nm)</th>
<th>T at C3 (°C)</th>
<th>C4 (Nm)</th>
<th>C5 (Nm)</th>
<th>Attenuation (dB/cm)</th>
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<td>0.710</td>
<td>0.807</td>
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<td>0.759</td>
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<td>W (x 10^-4 J)</td>
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Table 4: Coefficient of significant correlations (P<0.05) between flour conventional parameters and ultrasound measurements for flours with W>100 and W<100.

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<th>W&gt;100</th>
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<th>W&lt;100</th>
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<td>Attenuation (dB/cm)</td>
<td>Velocity (m/s)</td>
<td>Attenuation (dB/cm)</td>
<td>Velocity (m/s)</td>
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<td>Development time (min)</td>
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<td>0.517</td>
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<td>Stability (min)</td>
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<td>0.523</td>
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<td>0.503</td>
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<tr>
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<td>-0.498</td>
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<td>0.561</td>
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<td>C3 (Nm)</td>
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