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Article:

http://dx.doi.org/10.1016/j.lwt.2015.02.036

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1 BISCUIT DOUGH STRUCTURAL CHANGES DURING HEATING:
2 INFLUENCE OF SHORTENING AND CELLULOSE ETHER EMULSIONS

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The effect of using a variety of cellulose ether emulsions as a fat source, instead of a conventional shortening, on the structural changes occurring in a dough biscuit recipe during heating were studied. Linear viscoelastic properties and texture properties during heating were compared. In comparison to conventional shortenings, the cellulose ether emulsions are a healthier option characterized by lower fat content, lower saturated fatty acids content and the absence of trans fatty acids.

In the dough with shortening, temperature had the biggest influence on the viscoelastic properties, characterized by a decrease in viscoelasticity within the temperature range from 25°C to 45°C. In the cellulose ether emulsions dough the increase in temperature produced a slow linear decrease in the elastic and viscous moduli. At 80°C all dough showed similar viscoelastic behavior. The texture development during baking differed among the recipes. In the shortening dough the texture changes appeared since the first minute of heating, while in the emulsion dough, the effects were noticeable after 5 min of baking and varied according to the cellulose ether thermal gelation properties. A final crispy texture was obtained in all the recipes, although the cellulose emulsion biscuits required longer baking times.

**Keywords:** cellulose ether emulsion, fat replacer, biscuit baking, rheology, texture
1. Introduction

The reduction of fat in the everyday diet has become a public health issue and is an important concern for most consumers who wish to follow healthier habits (Zoulias, Oreopoulou & Tzia, 2002). In the topic of fat reduction, the most important concern is the reduction of saturated fat and the elimination of trans fatty acids from the diet. Although some controversy exists regarding the health effects of saturated fatty acids, the negative health effects of trans fatty acids have been accepted. While the debate regarding the exact health effects of these fatty acids (particularly saturated fatty acids) is unsolved what is clear is that food manufacturers have initiated a trend of moving away from ingredients containing saturated fatty acids and trans-fatty acids on the basis of consumer demand (Co & Marangoni, 2012). The World Health Organization recommends that fat consumption should be shifted towards unsaturated fats, as opposed to saturated-fats and trans-fats (W.H.O., 2004).

Fat sources higher in unsaturated fatty acids lack structure at room temperature. As a consequence, they can produce adverse effects in food products which often results in a reduction of product quality when used as a direct substitute for solid fats (Hughes, Marangoni, Wright, Rogers & Rush, 2009; Youssef & Barbut, 2009).

Nowadays, transesterification is the highly employ method to increase fatty acids saturation, as contrary to the partial hydrogenation process no trans fatty acids are formed. However, the ideal option should be the possibility to employ fat sources higher in unsaturated fatty acids such as edible oils, as such, without the need to increase its saturation.
In short-dough biscuits fat is an essential ingredient and it is the largest component after flour (Manohar & Rao, 1999). The major functions of fats in baked systems are imparting shortening, richness, and tenderness all which contribute to improve flavor and mouthfeel (Pareyt & Delcour, 2008). In general, higher percentages of fat produce more tender biscuit (Lai & Lin, 2006). While the water or sugar solution in the absence of shortening would interact with the flour proteins to create cohesive and extensive gluten. When shortening is present, the fat surrounds the proteins and the starch granules, isolates them, therefore breaking the continuity of the protein and starch structure (Ghotra, Dyal & Narine, 2002). This phenomenon results in eating properties after baking that are described as less hard, shorter, and more inclined to melt in the mouth.

Due to the important functionality of fat in biscuits, fat reduction without affecting quality properties and consumer acceptability is a challenging task. Different approaches have been carried out to reduce fat in biscuits with various levels of success. Fat replacement in biscuits has been studied by many different authors (Inglett, Warner & Newman, 1994; Rößle, Ktenioudaki, & Gallagher, 2011; Zbikowska & Rutkowska, 2008; Zoulias, Oreopoulou & Tzia, 2002; Zoulias, Oreopoulou & Kounalaki, 2002; Sudha, Srivastava, Vetrimani, & Leelavathi 2007; Laguna, Varela, Salvador, Sanz, & Fiszman, 2012; Laguna, Primo-Martin, Varela, Salvador & Sanz, 2014).

Hydroxypropyl methylcellulose (HPMC) has been used as fat replacer in baked goods, frozen desserts, dry mix sauces, dressings (Akoh, 1996) and gluten free breads (Sabanis & Tzia, 2011; Mariotti, Pagani & Lucisano, 2011). Methylcellulose (MC) and HPMC thermal gelation properties were also employed to reduce fat absorption in flour based batters; MC and HPMC confers the flour semisolid consistency at lower temperatures.
than frying temperatures and the formation of a barrier against moisture loss and oil absorption (Sanz et al., 2004, 2005ab). HPMC was used as fat replacer in short-dough biscuits by Laguna et al. (2014). In this first work, authors used a pre-hydrated HPMC solution, which was added to the dough as a fat replacer in the biscuit, however, they did not achieve shortening replacement higher than 15%, due to negative consumers’ score in acceptance.

Tarancón, Fiszman, Salvador & Tárrega (2013) investigate the use of a vegetable oil cellulose ether emulsion as shortening replacer. The consistency provided by the cellulose emulsion makes it possible to incorporate liquid oil in the biscuit recipe and provides a good consistency to manipulate the biscuits (laminate, cut and baked) in the same way that a full fat recipe would. In this way not only a reduction of the total amount of fat is accomplished but also a reduction of saturated fatty acids and the elimination of trans fatty acids. Biscuits prepared with the cellulose emulsions had good consumer acceptability (Tarancón, Fiszman, Salvador & Tárrega, 2013; Tarancón, Sanz, Salvador & Tárrega, 2014; Tarancón, Sanz, Fiszman & Tárrega, 2014).

The cellulose ethers methylcellulose (MC) and HPMC are employed to prepare the emulsions to have thermo-gelling ability. In solution these polymers are completely hydrated and with little polymer–polymer interaction other than simple entanglement. As the temperature is increased, an initial drop in viscosity is observed due to the decrease in the water hydration. When critical temperature is reached, sufficient dehydration occurs to promote polymer–polymer interactions instead of polymer–solvent interactions. As a consequence, these cellulose ether solutions start to gel. Upon cooling, the gelation process is completely reversed and the gel formed will revert to a sol state, recovering its original consistency. The temperature at which the gelation
process starts and the strength of the gel formed depends on; the type and degree of substitution of the cellulose, molecular weight, concentration and presence of electrolytes (Nishinari, Hofmann, Moritaka, Kohyama & Nishinari, 1997; Sarkar, 1979).

The thermal gelation properties of cellulose ethers appear to have an important role in the success of the cellulose emulsions as fat replacers, as emulsions without these property (xanthan gum emulsion) did not provide successful sensory properties. However, the role of the thermal properties of the cellulose ethers in texture development during biscuit baking has not been studied.

In a previous study the interaction of the cellulose ether with the other ingredients of a biscuit recipe was investigated at room temperature by the study of the rheological properties at small deformation (creep and viscoelastic properties). The cellulose emulsion conferred higher deformability to the dough and lower percentage of recovery in comparison to a control dough prepared with shortening, which was associated to higher spread and greater diameter in the final biscuits. The obtained biscuit texture properties could not be associated to the dough’s rheological properties at room temperature (Tarancón, Hernández, Salvador & Sanz, 2014).

The aim of this study is to investigate the structural changes occurring during baking in a conventional biscuit dough (prepared with shortening) and in doughs’ prepared with different cellulose emulsions as shortening replacers. First, the thermo-rheological properties of the different cellulose ether emulsions employed as shortening replacers and of the different biscuit dough (with shortening and with cellulose ethers) were studied. Second, the dough biscuit texture changes at different stages during baking was also investigated and related to rheological properties.
2. Materials and methods

2.1. Emulsion preparation

Oil-water-cellulose ether emulsions were prepared with three different cellulose ethers each with different thermo-gelling ability, supplied by The Dow Chemical Co. (E4M, K4M and A4M). They differ in their methoxyl and hydroxypropoxyl percentages. E4M and K4M are hydroxypropyl methylcellulose with 29.0% methoxyl and 9.3% hydroxypropoxyl (E4M) and 22.8% methoxyl and 8.1% hydroxypropoxyl (K4M), and A4M is a methylcellulose with 29.9% methoxyl. All have a viscosity around 4000 mPa.s at 2 % aqueous solution at 20ºC, A4M (3938 cps), E4M (5063 cps) and K4M (3183 cps) measured by The Dow Chemical Company following reference methods ASTM D1347 and ASTM D2363. The ingredients of the emulsion were sunflower oil with high level of oleic acid (Carrefour, Madrid, Spain), water and the different cellulose ethers. The proportions employed were sunflower oil 51%, water 47% and cellulose ether 2%. The cellulose ether was first dispersed in the oil using a Heidolph stirrer at the lowest speed for 5 min. The mixture was then hydrated by gradually adding the water at 10ºC while continuing to stir. The water temperature of 10 ºC was selected according to the specific hydration requirement of cellulose ethers. Stirring continued using a homogenizer (Ultraturrax T18, IKA, Germany) until the emulsion was obtained.

2.2. Biscuit dough preparation

The dough ingredients (flour weight basis) were shown in Table 1. The composition data of the soft wheat flour provided by the supplier is 11% protein, 0.6% ash;
alveograph parameters $P/L=0.27$, where $P=$maximum pressure required and $L=$extensibility, and $W=134$, where $W=$baking strength of the dough and of the shortening was 78.4% total fat, 51% saturated fatty acids, 20% monounsaturated fatty acids, 6% polyunsaturated fatty acids and < 2% trans fatty acids.

The shortening or shortening replacer, sugar, milk powder, leavening agents, salt, water and glycerol (in the case of biscuits made with the shortening replacer) were mixed using a mixer (Kenwood Ltd., U.K) for 1 min at low speed (60 rpm). The bowl was scraped down and the mixture was mixed again for 3 min at a higher speed (255 rpm). The flour was added and mixed in for 40 s at 60 rpm, then mixed for a further 20 s at 60 rpm after scraping down the bowl once more. The dough was sheeted with a sheeting machine (Parber, Vizcaya, España) and moulded to 10 mm in thickness. It should be noted that every manipulation of the dough induces some stress in the test piece, so to ensure dough stability the dough, they were kept in a refrigerator for 24h hours before each test. The test pieces were shaped by punching a tube into the dough and using a wire to cut the resulting cylinder (10mm thickness and 50 mm diameter) into pieces of a set height.

2.3. Biscuit baking

Three biscuits of 50 mm diameter and 10 mm thickness for each formulation were placed on a perforated tray and baked in a conventional oven (De Dietrich, France) for 1, 3, 5, 7, 9 and 12 min at 200ºC. The oven and the oven trays were always the same, the trays were placed at the same level in the oven and the number of biscuits baked was always the same. After cooling, the texture of biscuits was evaluated.
2.4. Linear viscoelastic properties

Viscoelastic properties of the emulsions and dough were measured. The samples were allowed to rest in the measurement cell for a 5 min equilibration time. The emulsions were measured using a 40 mm diameter serrated plate-plate sensor geometry with 1 mm gap. Biscuit dough was measured using a 20 mm diameter serrated plate-plate with 2 mm gap. To simulate the effect of heating in the samples structure, temperature sweeps were performed between 25 ºC to 80 ºC at a heating rate of 1.0 ºC/min and a strain amplitude was applied inside the linear viscoelastic region in all the temperature ranges and for all the samples, according to previous stress sweeps carried out between 25 ºC and 80 ºC. Vaseline oil was applied to the exposed surfaces of the samples to prevent sample drying during testing. Additionally, mechanical spectra in the linear region from 0.01 to 10 Hz between 25 ºC and 80 ºC were recorded in separate tests. The storage modulus (G’), the loss modulus (G’’) and tan δ were recorded. Two different batches for each formulation prepared on different days were employed and each test was conducted twice.

2.5. Texture analysis

The sheeted dough (10 mm in thickness) for the different formulations was analysed at different times. A TA-XT.plus texture analyser equipped with the Texture Exponent software (version 2.0.7.0. Stable Microsystems, Godalming, UK) was used. Penetration tests were conducted with the upper Volodkevich Bite Jaw, penetrating dough discs of 10 mm in thickness with a diameter of 50 mm to 2.5 mm at a test speed of 1 mm/s; a trigger force of 5g was set. Six baking times for each formulation was measured (0, 1, 3, 5, 7, 9 and 12 min). Two different batches of biscuits for each dough formulation,
prepared on different days, were always prepared and each test was conducted with three replicates of each formulation and batch.

2.6. Data analysis

One way-ANOVA was applied to study the effect of type of cellulose on the rheological parameters. Least significant differences were calculated using the Tukey test at a significance of $P<0.05$. These analyses were performed using XLSTAT 2009.4.03 (Addinsoft, Barcelona, Spain).

3. Results and discussion

3.1. Emulsion properties

3.1.1. Linear viscoelastic properties at 20ºC

Consistent and stable emulsions were formed with the three types of cellulose ethers evaluated. To better evaluate the effect of the different cellulose ethers in the emulsion properties, the linear viscoelastic properties were studied. The mechanical spectra of the fresh emulsions at 20ºC are shown in Figure 1A. The viscoelastic properties were very similar among the different emulsions. A big dependence with frequency was always found, and the transition area of the mechanical spectra, with the crossover among $G'$ and $G''$, was observed, in the available frequency range.

In Table 2 statistical differences among values of $G'$, $G''$ and $\tan \delta$ at 1 Hz are shown. Values of $G'$ and $G''$ were not significantly different among emulsion A4M and K4M, while emulsion E4M showed significantly lower values. The analysis of $\tan \delta$ values at 1 Hz revealed significantly higher values in emulsion K4M, implying lower predominance of the elastic component in this emulsion. Also, the frequency at which
the crossover among $G'$ and $G''$ occurred was slightly higher in emulsion K4M, which reflects a slightly lower elasticity (Figure 1A).

3.1.2. Viscoelastic changes during heating

To investigate the effect of the thermo-gelling ability of the cellulose ethers in the emulsion, the evolution of the viscoelastic functions with the increase in temperature was monitorized. In Figure 1C, values of $G'$ and $G''$ versus temperature, and of tan $\delta$ versus temperature (Figure 1D) are shown, respectively. Differences in the viscoelastic heating profile among emulsions were found. In all the emulsions an increase in elasticity occurred upon heating, but the temperature at which the increase in $G'$ (and decrease in tan $\delta$) appeared was dependent on the cellulose type. As expected, the lowest onset gelation temperature was found for cellulose A4M, which is associated to the higher methoxyl content of methylcellulose. E4M and K4M emulsions showed higher onset gelification temperatures. However, the heating profile of E4M emulsion was similar to emulsion A4M. In both E4M and A4M emulsions the viscoelastic functions remained stable with the increase in temperature, until the corresponding critical temperature was achieved, which determines a decrease in tan $\delta$ (40ºC for emulsion A4M and 50ºC for emulsion E4M). In the K4M emulsion, the change in the viscoelastic properties with temperature was more gradual and the change in the curve gradient was lower. At the end of the heating curve, the A4M emulsion showed the highest $G'$ values and the lowest values of tan $\delta$ (higher viscoelasticity), which indicates the formation of a stronger gel system.

The effect of frequency at 80ºC was also investigated. The sample structure was stabilized during 10 min at the end of the heating curve and after that a frequency sweep was applied (Figure 1B). Contrary to the spectra shown at 20ºC (Figure 1A), at 80ºC...
practically no frequency dependence of the viscoelastic functions was observed, with the values of $G'$ higher than $G''$ in all the available frequency studied in all samples, according to the observed thermal gelation. A4M emulsion showed significantly higher values of $G'$ than E4M and K4M emulsions. These two emulsions did not differ significantly in $G'$ values, but K4M emulsion showed significantly higher tan $\delta$ values, which implies lower emulsion viscoelasticity similar to the behavior found at 20ºC (Table 2).

3.2. Biscuit dough

3.2.1 Linear viscoelastic properties at 20ºC

The frequency sweep of the shortening dough in comparison to the different emulsion dough is shown in Figure 2 (A and B). In Table 3 values of $G'$, $G''$ and tan $\delta$ values at 1 Hz are shown. The control dough showed higher $G'$ and $G''$ than the emulsion dough, implying that the emulsion dough structure was softer (Figure 2A). The shortening dough showed significantly higher values of $G'$ and $G''$ at 1 Hz than the three samples of emulsion dough. No significant differences were found among the three emulsions, although values of $G'$ and $G''$ were slightly higher in the A4M emulsion dough. However, no significant differences were found in the values of tan $\delta$ at 1 Hz although the frequency dependence of the control and the emulsion dough was different as can be seen in Figure 2B. The control dough shows higher frequency dependence than emulsion dough samples with a marked increase in tan $\delta$, observed at higher frequencies. This reflects that the control dough is closer to the cross over point at lower frequencies, associated to the end of the plateau zone, than the emulsion dough, which revealed a tendency towards a more extended plateau region.
Values of $G'$ and $G''$ and $\tan \delta$ during heating of the different dough samples are shown in Figures 2 (C and D, respectively). The heating profiles were different among the control and the emulsion dough. In the control dough two main zones were observed. A first zone characterized by a pronounced decrease in $G'$ and $G''$. This decrease is associated to an increase in $\tan \delta$ (which implies a decrease in the predominance of the elastic component) until a maximum value in $\tan \delta$ was achieved at 42°C. This first zone is associated to fat melting (Roos, 1995). The second zone starting approximately around 45°C and it is characterized by a very low dependence of $G'$ and $G''$ values with temperature and by a decrease in $\tan \delta$.

However, all the emulsion dough samples show a similar heating profile characterized by a linear and slow decrease in both $G'$ and $G''$ when temperature was increasing, revealing the absence of fat (no fat melting in the first zone). The differences among celluloses found in the emulsion structures do not appear so evident after the interaction with the other biscuit ingredients. Although slight differences were found in the lines gradients, with the largest gradient found in the A4M dough, and the lowest in the E4M. The graph corresponding to $\tan \delta$ versus temperature shows small fluctuations in the $\tan \delta$ values. At the end of the heating sweep, values of $G'$ and $G''$ and $\tan \delta$ became very similar among the different dough types, although the control dough showed the highest $\tan \delta$.

The effect of frequency on the dough viscoelastic properties at 80°C are shown in Figure 2 (E and F). At 80°C the values of $G'$ and $G''$ of the control dough are higher than the values of the cellulose dough (Figure 2E), although the values at 1 Hz (Table 3) were not significantly different among the samples. The $\tan \delta$ values were also very
similar between the different dough types, and no significant differences were found (Figure 2F). These results indicate that the existing differences in the viscoelastic behavior at room temperature were minimized after heating, as all the dough samples have quite similar viscoelastic behavior after heating. Also, the differences observed among the cellulose types during emulsion heating appear only slightly evident during dough heating.

3.3. Evolution of biscuit texture during baking

The evolution of biscuit texture during baking of the shortening dough and the different emulsion dough is shown in Figure 3. The profiles of the penetration curves were measured before dough samples were introduced in the oven (0 min) and after different heating times: 1, 3, 5, 7, 9 and 12 min. The last time is the usual baking time employed in biscuit manufacturing.

Before the baking process, all dough types show a similar profile characterized by a typical penetration profile, with no breaking point, based only on the increase of the force over time until the displacement fixed corresponding to the indentation of the sample. No resistance of sample to penetration was observed. The control dough showed the highest penetration force, which is in accordance with the highest values of \( G' \) and \( G'' \) (Figure 2A) and with the lowest deformation capacity (Tarancón, Hernández, Salvador and Sanz, 2014). After 1 min baking a sudden increase in the penetration force was found in the control sample, while the behavior of the emulsion dough remains practically unchanged. After 3 min the penetration curve profiles of the emulsion dough showed no change respect to 1 min indicating a very low resistance to penetration, however, after 3 min the control sample presented a profile completely different with a shape of the curve corresponding to a more resistant system. The
gradient of a curve is a parameter which indicates the rigidity of the structure. For the
control sample the gradient increased quickly from 1 to 3 min implying the
development of a solid structure. As can be seen, the penetration force of control sample
was higher than the emulsion formulations until after 5 min baking.

At 1 and 3 min baking (Figure 3B and C, respectively) no differences in the force
curves among the emulsions dough were found, however, at 5 min (Figure 3D) an
increase in the force of the emulsions dough was observed, with the higher increase for
A4M emulsion dough respect the other cellulose emulsions. This greater increase on the
A4M dough could be due to the earlier onset of gelation of the A4M cellulose that
occurs at lower temperature than the hydroxypropyl methylcellulose samples (The Dow
Chemical Co., 1996). In Figure 3E, after 7 min baking, the emulsion dough forces begin
to equal that of the control force, although in the control biscuit we begin to see the
crispy character due to the appearance of many peaks of fracture. At 9 min baking, the
hydroxypropyl methylcellulose (E4M and K4M) emulsion biscuits presented
penetration forces similar to the control biscuits but without crispy character. A4M
emulsion biscuits presented a force peak much higher indicating greater hardness than
the other biscuits. Finally, after 12 min baking (Figure 3G) forces on emulsion biscuits
are similar and their profiles presented many peaks of fracture, indicating that at the end
of baking all samples presented a similar texture profile, although with a harder texture
in the emulsion biscuits.

In summary, texture evolution during baking reveals that biscuits with oil-water-
cellulose emulsions needed more time to set the structure (probably due to a higher
water retention of cellulose), and require longer baking times to develop a crispy
texture. Differences were observed among the different cellulose chemical substitutions,
associated to their level of methoxyl substitution and the emulsion gelation temperature.
The A4M dough showed the highest increase in force associated to its higher methoxyl substitution and lower gelation temperature, followed by E4M and K4M.

4. Conclusions

The structural changes occurring during dough baking were completely different between the shortening and the emulsion dough samples. In the shortening dough the changes during heating were mainly governed by the fat melting process, while in the emulsion dough these changes were associated to the cellulose thermo-gelling properties.

The shortening dough’s viscoelastic behavior was characterized by a pronounced decrease in the elastic and viscous component and in viscoelasticity within the temperature range between 25°C and 45°C, associated to fat melting. On the contrary, in the emulsion dough the effect of an increase in temperature only produced a slow linear decrease in \( G' \) and \( G'' \). Despite these differences, in both types of dough the final viscoelastic behavior achieved at 80°C was very similar.

As for the changes in texture during baking, also, completely different patterns of behavior were found among the shortening and the emulsion doughs. In the shortening dough the texture changes appeared after the first minute of baking, while in the emulsion dough the effects were only noticeable after 5 min of baking. At this stage differences were found among the cellulose ethers, with the A4M (methylcellulose) emulsion dough being the first that showed an increase in force, and the highest increase in force. This was associated to the lower gelation temperature and the highest strength of the gel formed by methylcellulose (A4M) in comparison to the hydroxypropyl methylcellulose samples (E4M and K4M). In all the dough samples a final texture composed of several breaking peaks was obtained, although the baking time required to obtain such a texture was much longer in the emulsion biscuits and their final texture
was harder than the control sample. In conclusion, the cellulose emulsions are successful as shortening replacers, although longer baking times should be employed to develop the traditional biscuit crispy texture.

Acknowledgements

The authors are grateful for the assistance of Phillip John Bentley with the English-language editing.

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FIGURE LEGENDS

Figure 1. G’ (closed symbols) and G’’ (open symbols) as a function of frequency for the different cellulose emulsions at 20ºC (A) and 80ºC (B) and the effect of increasing temperature on G’ and G’’ (C) and loss tangent (D) for the different cellulose emulsions at a heating rate of 1.0 ºC/min (circles: A4M, triangles: E4M and squares: K4M).

Figure 2. G’ (closed symbols) and G’’ (open symbols) and loss tangent as a function of frequency for the different doughs at 20ºC (A: G’, G’’ and B: loss tangent) and 80ºC (E: G’, G’’ and F: loss tangent) and the effect of increasing temperature on G’ and G’’ (C) and loss tangent (D) for the different doughs at a heating rate of 1.0 ºC/min (asterisk: control dough, circles: A4M dough, triangles: E4M dough and squares: K4M dough).

Figure 3. Profile of the penetration curves of the shortening dough and the different emulsion doughs after different baking times (0 min:A, 1 min: B, 3 min: C, 5 min: D, 7 min:E, 9 min: F and 12 min: G).