

A New Method to Study Simple Shear Processing of Wheat Gluten-Starch Mixtures

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ABSTRACT

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This article introduces a new method that uses a shearing device to study the effect of simple shear on the overall properties of pasta-like products made from commercial wheat gluten-starch (GS) blends. The shear-processed GS samples had a lower cooking loss (CL) and a higher swelling index (SI) than unprocessed materials, suggesting the presence of a gluten phase surrounding starch granules. Pictures of dough microstructure by confocal scanning laser microscopy (CSLM) showed the distribution of proteins in the shear-processed samples. This study revealed that simple shear processing could result in a product with relevant cook-

ing properties as compared with those of commercial pasta. Increasing gluten content in GS mixtures led to a decrease in CL and an increase in maximum cutting stress of processed samples, whereas no clear correlation was found for SI values of sheared products. It was concluded that the new shearing device is unique in its capability to study the effect of pure shear deformation on dough development and properties at mechanical energy and shear stress levels relevant to industrial processing techniques like pasta extrusion.

Traditionally, dough is made by combining flour, water, and energy through mixing. The addition of sufficient mechanical energy provides the distribution and hydration of flour particles, allowing the formation of a continuous protein matrix, holding starch and other components together (Campos et al 1997). During dough processing like mixing and extrusion, wheat constituents are not only mixed with other ingredients but are also subjected to various shearing regimes. Bloksma and Bushuk (1988) stated that mixing could be divided into three distinct stages: distribution of material, hydration, and energy input to stretch and align protein molecules. The energy input due to mixing, which generally involves shear and extensional deformation, is an important aspect of protein development. Mixing energy alone however does not completely characterize the mixing process. The nature of the mixing action is also important but is more difficult to specify. Recently, Jongen et al (2003) using simulation study, reported that dough mixing in kneaders consists of a combination of various flow patterns such as shearing and elongation. Moreover, they pointed out that the way in which these deformations are provided to the material during mixing is of crucial importance.

There is hardly any literature on the effect of a constant strain deformation using high shear stresses on dough properties. Information in literature (Janssen et al 1996a,b; Safari-Ardi and Phan-Thien 1998; Uthayakumaran et al 2000, 2002; Autio et al 2001; Dhanasekharan et al 2001; Newberry et al 2002; Tronsmo et al 2003) concerning the effect of shear on dough properties is often limited to the characterization of dough rheological properties under small amplitude oscillatory shear measurements with a shear stress range of 0.2–0.8 kPa. Recently, Schluentz et al (2000) and Lee et al (2001) produced partially developed dough using a well-defined shear or extensional deformation from undeveloped dough. They concluded that shearing or extensional deformation alone using a rheometer is not able to produce dough of a quality

comparable to dough produced through a combination of shear and extensional deformation using a farinograph mixer. These investigations, however, are limited to the application of significantly lower shear stresses (0.8 kPa) and mechanical energy input compared with those normally used in dough mixing and pasta extrusion. Therefore, it is likely that due to the different work input used, the material has been processed differently. Therefore, we developed a new shearing device that provides the possibility to study simple shear deformation on wheat gluten-starch (GS) mixtures. The cell is able to apply shear rates varying from 24 to 250 sec⁻¹ to high viscous materials, thereby allowing high shear stresses up to 50 kPa. These stresses are comparable to shear stress values obtained in extrusion trials performed by Leroux et al (1995). A study by Van den Einde et al (2004, 2005) showed that comparable product changes could be obtained by shear cell and extrusion trials in case of starch breakdown. We therefore concluded that the shear cell is a unique device to study changes of product properties under high shear conditions, which might help to improve the understanding of the effects of extrusion or mixing on pasta product properties.

A dough formula simplified to flour and water still encompasses a complex series of flour components and their interactions. Therefore a model mixture of gluten and prime starch (GS) is used, not only to allow us to control the protein content but also to reduce the complex interactions of the flour constituents such as water-soluble pentosans and soluble proteins including enzymes (Petrofsky and Hoseney 1995).

The goals of this study were therefore to 1) demonstrate the usefulness of a pilot-scale shearing device to investigate simple shear deformation of wheat GS dough; and 2) investigate the effect of different protein/starch ratios on the overall properties of pasta-like products obtained from GS mixtures by a well-defined simple shear deformation.

MATERIALS AND METHODS

Materials

Commercial wheat gluten and starch were obtained from Roquette Freres (France). Moisture content of the starch and gluten were 12.3 and 6.5% (dm), respectively, determined by Approved Method 44-15A (AACC 2000). According to the manufacturer, protein and fat content of starch sample were max. 0.4 and 0.1% (w/w), respectively. Protein, starch, and fat content of gluten were max. 86.0, 10.0, and 3.0% (w/w), respectively.

Commercial Italian spaghetti with a composition (w/w) of starch 75.2%, protein 12.7%, moisture 8.9%, and fat 1.7% was used as a reference product.

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*The e-Xtra logo stands for "electronic extra" and indicates that the online version contains a color version of Fig. 8 not included in the print edition.

Preparation of GS Blends

Starch was blended with different amounts of gluten to achieve 0, 5, 8, 11, 14, and 17% gluten based on dry weight of starch. All samples were adjusted to 40% (db) moisture before shear processing. Aside from the point that the moisture content in the current study provides a good handling of the material in the shearing device, it also is within the range of water content normally used in pasta extrusion (40–48%) (Grzybowski and Donnelly 1979; Mestres et al 1988; Leroux et al 1995).

The GS samples were encoded as G0, G5, G8, G11, G14, and G17, where G represents gluten and the numbers show the percentage of gluten (starch db) in the mixture. A Waring blender with a modified mixing propeller was used to blend the dry ingredients.

Preparation of Undeveloped Dough

The method of Campos et al (1996) was used with some modifications to prepare undeveloped GS dough. Powdered ice was prepared in the presence of solid carbon dioxide (dry ice) in a walk-in cooler at +2°C. Large pieces of solid CO₂ were first pulverized with a Waring blender to generate a cold medium inside the blender. Ice flakes were then added and broken into smaller particles. Dry ice and ice particles were sieved to a particle size range of ≈700 μm. The powder mixture was kept at

–25°C undisturbed overnight, allowing sublimation of solid CO₂ while keeping the ice particles intact. The blending of starch, gluten and ice particles was performed inside a walk-in freezer (–25°C). Materials were carefully weighed, placed in the Waring blender, and distributed uniformly by mixing at a reduced speed. The resulting GS and ice mixture was placed in a closed container and kept frozen at –18°C. The frozen gluten-starch-ice mixture was kept in a moisture-tight container at 35°C for 60 min before each shear treatment. This holding period allowed the ice particles to melt, resulting in hydration of ingredients producing an homogeneous undeveloped dough.

Shear Cell

The simple shearing process was performed using a newly developed device in our laboratory. This new shearing device (shear cell) was based on a cone and plate rheometer concept. It was designed and sized at pilot-scale. The maximum gap size between cone and plate was small compared with the diameter of the cone. A small angle between cone and plate resulted in a constant shear rate. The contact surface of both cone and plate (moving bottom cone) was roughened (serrated) to avoid possible slippage of material during shear processing. The motor used in the testing device was able to provide shear stresses up to 50 kPa. A schematic configuration of the shear cell is shown in Fig. 1.

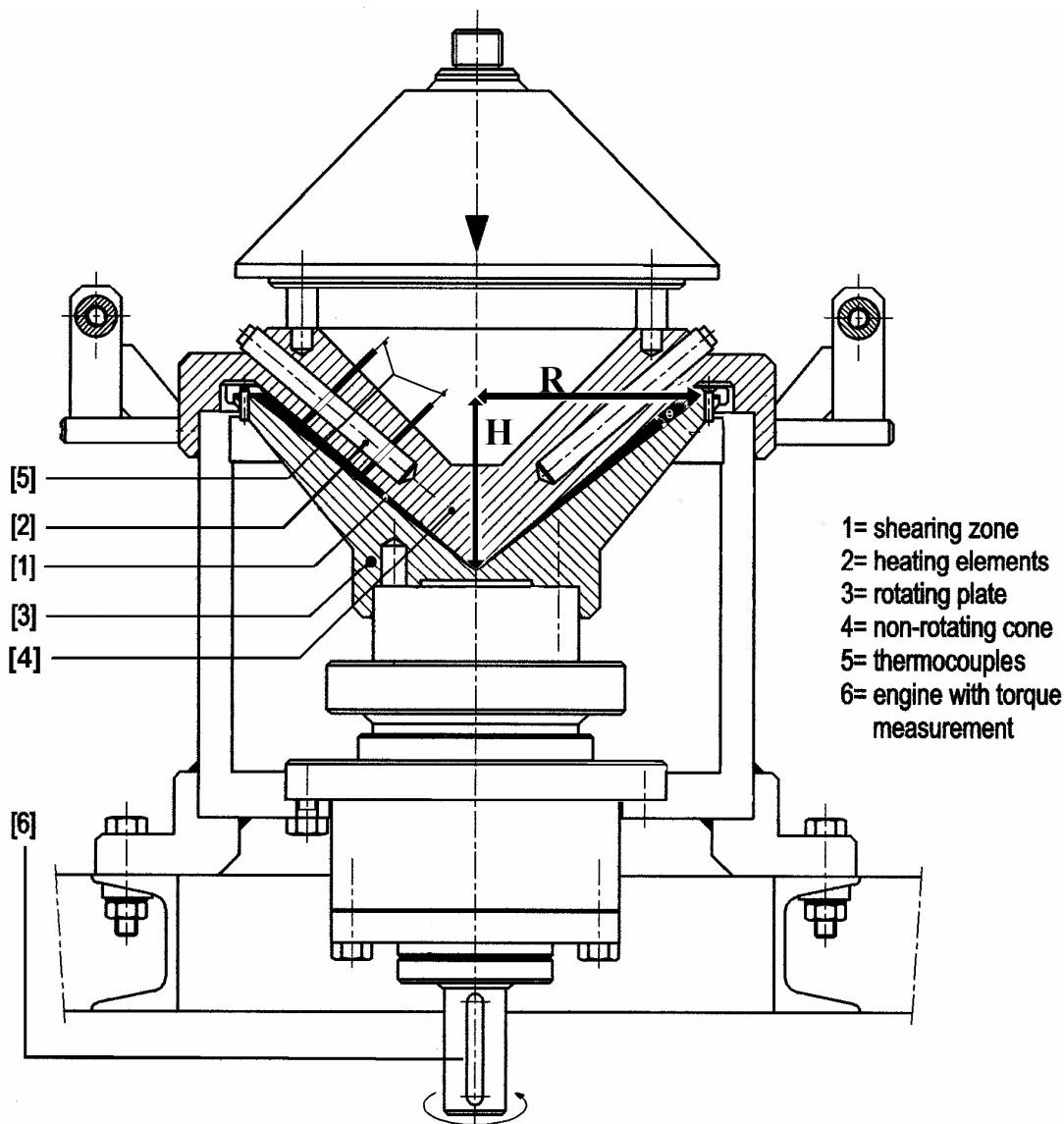


Fig. 1. Shearing device. Cone angle = 100°, angle between cone and plate (shearing zone) $\theta = 2.5^\circ$, $R = 0.108$ m, $H = 0.082$ m.

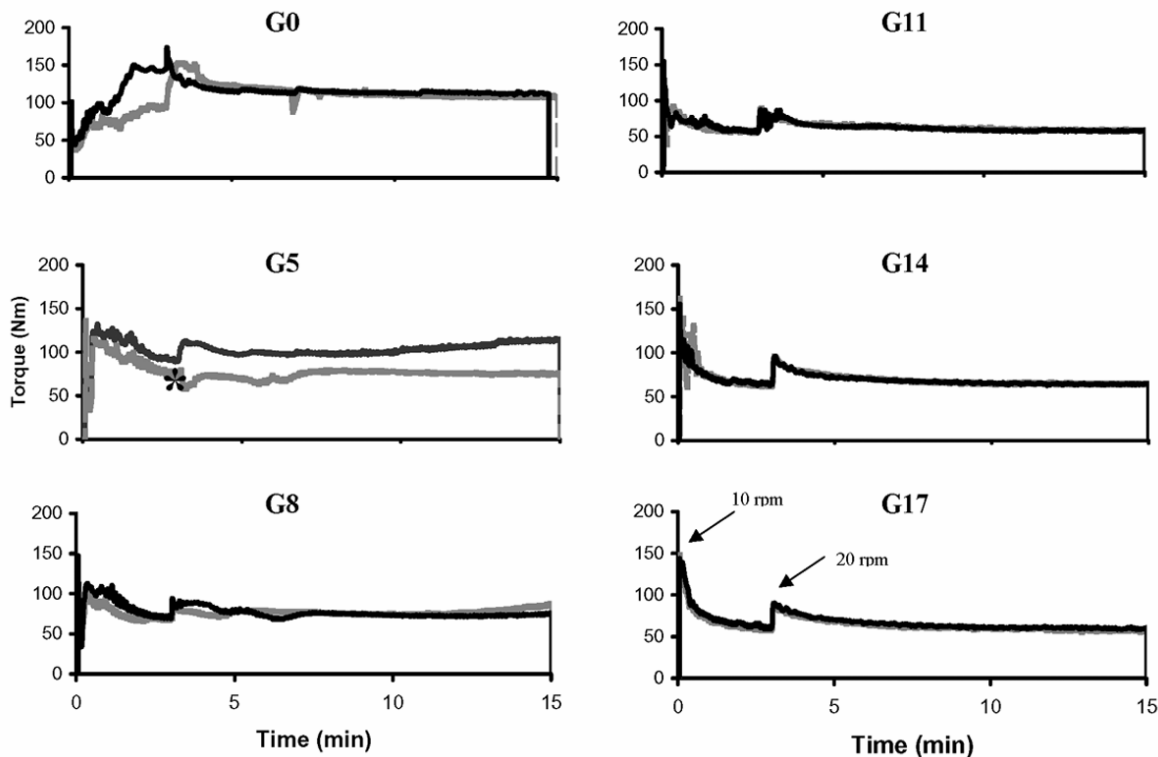


Fig. 2. Torque-time profiles of shear processing of undeveloped gluten-starch (GS) samples (G0–G17). Two curves correspond to two replicates indicating reproducible shear processing of samples (except for sample G0). G5-star line implies a sample that was not produced properly and therefore discarded. Two curves in G17 are fully overlapped. Arrows in G17 indicate two different rotational speeds used.

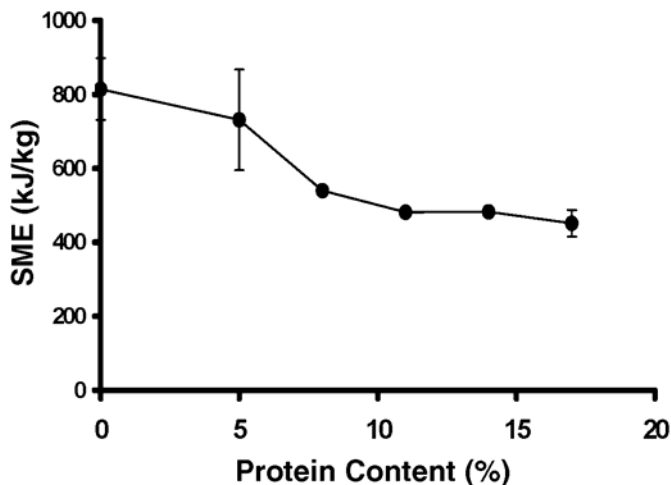


Fig. 3. Effect of gluten amount on specific mechanical energy (SME) during shearing process. Error bars show 95% confidence interval.

The shear rate ($\dot{\gamma}$ in sec^{-1}), shear stress (τ in Pa), and mechanical energy input (SME in kJ/kg) applied to the material during processing were calculated:

$$\dot{\gamma} = \frac{\omega}{\theta} \quad (1)$$

$$\tau = \frac{3M}{2\pi R^3} \longrightarrow \tau = CM \quad (2)$$

$$\text{SME} = \frac{\int_{t=0}^{t_f} \omega \cdot M(t) \cdot dt}{m} \quad (3)$$

In these equations, ω is the rotor speed (sec^{-1}), θ is the angle between cone and plate (shearing zone in Fig. 1, in degrees), M is the torque (Nm), R is the projected radius of cone and m is the mass of material in the shear cell (kg). Equations 1 and 2 are based on the cone and plate rheometer (Macosko 1994). C is a constant (m^{-3}) in Equation 2 and is equal to 379 considering the projected radius of the cone ($R = 0.108 \text{ m}$).

Shearing Process

For all samples, constant amounts of undeveloped GS dough (215 g) were placed in the shear cell. The filling procedure was a critical step in the shearing process, thus extra care was taken to evenly fill the cell. After filling the shearing zone ([1] in Fig. 1) with undeveloped dough, the cone and plate cell was closed and a vertical pressure was applied and kept constant during the shear experiments.

Undeveloped dough was rested for 30 min to allow relaxation of stresses that result from the filling and pressurized closure of system. The system was heated to 40°C by electrical heating elements inserted in the cone [2] in Fig. 2. Shear was then applied to the unprocessed material between the rotating plate [3] and the nonrotating cone [4]. Thermocouples [5] monitored the sample temperature during resting and subsequent shearing process for each experiment.

A constant shearing time of 900 sec was applied for all samples at shear rates of 24–48 sec^{-1} . The lower limit was set by the slowest possible rotation of the motor (10 rpm). This rate was kept for 180 sec to avoid slippage. It was then increased to 20 rpm, corresponding to a shear rate of 48 sec^{-1} , until the end of the shear run. All shear treatments were done at least in duplicate.

After shearing the GS samples, a visual observation of a homogenous dough indicated the absence of slip during the run. If slippage had occurred, then a product with only a smooth surface would have been the result due to the lack of shearing action in the interior of the dough piece. Preliminary experiments showed that slippage yielded a product with a smooth surface. Under the

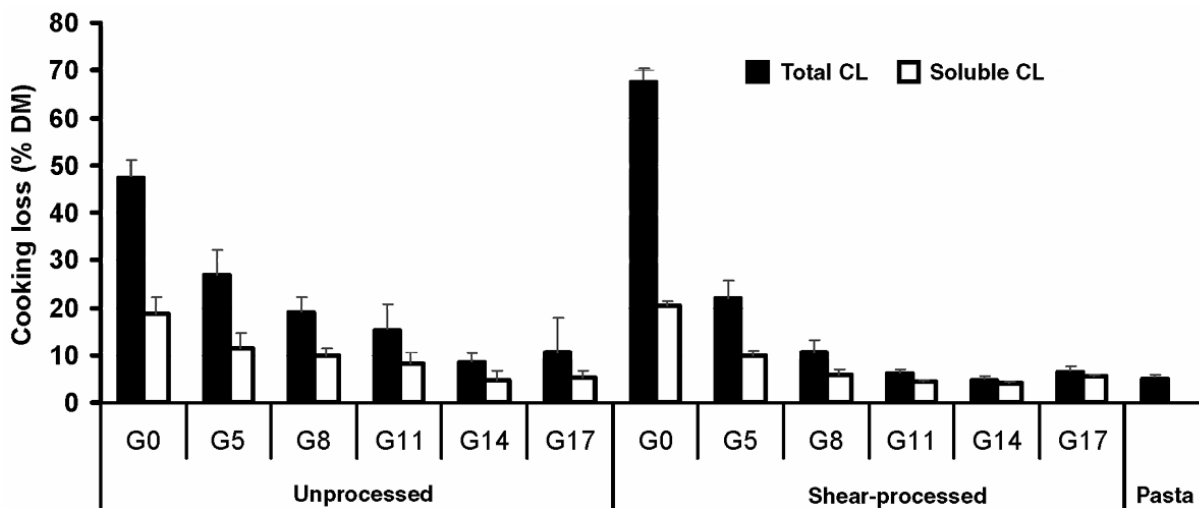


Fig. 4. Effect of shear processing and different gluten concentration on total and soluble cooking loss of cooked product. Error bars show 95% confidence interval.

process conditions reported in this study, no slippage was observed. This was apparent from the presence of lines in the dough samples originated by the grooves in the cone and plate geometry.

Sampling of Product for Cooking Experiments

As a consequence of the shear cell design, the thickness of the dough was 1.5–4.5 mm. We therefore selected material from the middle part of the plate, with an average thickness of 3 mm for sampling. Ring-shaped samples (concentric circles) were taken using a suitable sampling device. The diameter of outer and inner circles was 15.75 and 12.25 mm, respectively, which gave a wall thickness of 3.5 mm for ring-shaped samples. The resting of undeveloped dough inside the pressurized cell at a temperature of $38 \pm 2^\circ\text{C}$ for 30 min resulted in a complete hydration of the material. The resulting dough before the start of the shear run is called unprocessed dough. For comparison, sampling was done once before (unprocessed dough) and once after the shearing process for each sample. Ring-shaped samples were taken and immediately transferred to a freezer at -18°C . After freezing, the samples were freeze-dried overnight to an average value of 8% moisture. The dried samples were then stored in sealed plastic bags at ambient temperature until further analysis.

Characterization of Product

Pasta cooking quality is highly influenced by the protein network and starch gelatinization (Resmini and Pagani 1983). Water uptake (swelling) and starch gelatinization during cooking of pasta are considered to be dependent on the quality of the surrounding protein network (Grzybowski and Donnelly 1979). Cooking loss (CL), swelling index (SI), and cooked firmness were the quality parameters used in this study to assess the quality of sheared GS products. Both unprocessed and shear-processed samples were subjected to cooking. In addition, and as a matter of comparison, the quality of cooked GS samples was compared with that of commercial spaghetti.

Cooking Test

The cooking procedure of Mestres et al (1988) was used with some modifications. Distilled water (100 mL) was heated to boiling under reflux in a 300-mL beaker. Freeze-dried ring-shaped samples (≈ 5 g) were added to the boiling water. The optimum cooking time was determined using a crushing test. A piece of sample was removed from the cooking water at various time intervals and squeezed between a pair of plexiglas plates. The optimum cooking time was reached when the white central core of the cooked sample had just disappeared. Cooking was then

continued 1 min more than the optimum cooking time defined as a normal cooking time. The sample was drained for 3 min in a Büchner funnel and weighed (W_1 , g). The cooked product was dried in an oven at $130 \pm 1^\circ\text{C}$ to a constant weight (W_2 , g). The cooking and rinsing water was centrifuged ($9,000 \times g$) for 10 min. Then dry matter content of the supernatant (W_3 , g) was determined. Total cooking loss (CL), which includes solid loss (dry matter content of sediment) and soluble loss (dry matter content of supernatant) during cooking, was calculated with Equation 4. Soluble CL was also calculated with Equation 5 (dry matter [DM] concentration of crude samples and initial weight [IW] of samples before cooking). Swelling index after cooking was calculated by Equation 6.

$$\text{Total cooking loss (TCL \%)} = \frac{IW \times DM - W_2}{IW \times DM} \times 100 \quad (4)$$

$$\text{Soluble loss (SL \%)} = \frac{W_3}{IW \times DM} \times 100 \quad (5)$$

$$\text{Swelling index (SI \%)} = \frac{W_1 - W_2}{W_2} \times 100 \quad (6)$$

Cooked Firmness

A texture analyzer (TA-XT2, Stable Micro Systems, Surrey, UK) equipped with a 5-kg weight beam, was used to measure firmness of the cooked samples according to Approved Method 16-50 (AACC 2000) modified for the TA application. The width of cooked samples before each measurement was determined carefully and the contact area between the sample and the edge of the tooth (0.11 cm) was calculated. Cooked samples or spaghetti strands were placed under the plastic tooth in a specially designed sample holder to avoid any unwanted movement of the sample during the cutting test. Samples were deformed (cut) at a 90° angle using a special plexiglas tooth (Walsh 1971) with a test speed of 0.17 mm/sec. Penetration distances of 2.5 and 1.5 mm for GS and spaghetti samples were selected, respectively. These distances were $\approx 65\%$ of total sample height. Seven replicates were performed for each sample. Generally, the maximum force (F_{\max}) or maximum stress and the energy required to cut the sample (Walsh and Gilles 1971) are often used as a measure of pasta firmness. Due to differences in the geometry and contact area of the cooked GS samples and commercial spaghetti, a cutting stress measured at a penetration 65% of the sample thickness was used in this study (maximum stress, σ_{\max} in N/m^2 , Pa). This maximum stress was taken as a measure of the firmness of the cooked samples.

Characterization of Dough Microstructure by CSLM

Dough slices with a thickness of ≈ 1 mm and dimensions of $\approx 0.5 \times 1$ cm were obtained from unprocessed as well as shear processed dough. Unprocessed dough was taken from the shear cell just before starting the shear run. Specimens of shear processed samples were taken from defrosted frozen dough (at 4°C for 24 hr). A double-staining technique (combination of FITC [1% w/v] and Rhodamine B [0.1% v/w]) was used. This method allowed simultaneous observation of protein and starch under CSLM. The stained slices were stored for 1–2 hr at ambient temperature before observation. The microstructure of dough was observed using CSLM (Leica TCS SP) equipped with an inverted microscope (model Leica DM IRBE) used in the single photon mode with an Ar/Kr visible light laser according to the procedure described by Van de Velde et al (2003).

RESULTS AND DISCUSSION

Shear Cell Experiments

All shearing experiments were done in the pilot-scale shear cell. Typical changes in torque values during shearing of GS blends with different GS ratio are shown in Fig. 2. In GS doughs, the torque curves were characterized by two main peaks (arrows in G17) which are related to the two different rotational speeds applied during the tests and by a plateau after 5 min of shearing. In the shear processing of one of the G5 duplicate samples, an error accrued in the filling step of the cell and therefore only that sample (G5*) was discarded.

In general, torque measurements (Fig. 2) revealed good reproducibility of the shear treatments, especially for samples with high protein contents. This would indicate the potential of this device to apply well-controlled, simple shear deformations to materials. The effect of protein content on dissipation of the mechanical energy through the GS mixtures during shear processing was studied. Figure 3 shows changes in specific mechanical energy versus gluten content.

Increasing protein content in the GS mixtures led to a decrease in the torque (Fig. 2) and consequently in SME values (Fig. 3). It has been hypothesized that in a developed dough, starch granules are dispersed in a continuous gluten phase. In this hypothesis, the gluten plays the role of a lubricant for the starch granules in the dough (Eliasson and Larsson 1993). This effect might explain the role of protein in reducing torque (and calculated SME) values. According to Watanabe et al (2002), a lower starch-to-gluten ratio in GS doughs leads to the formation of doughs (through mixing) with a rubber-like structure of low storage modulus (G') values.

This conclusion is also supported by the rheological behavior of gluten-supplemented flour (Bhattacharya 1993) and of wheat flour dough diluted with starch (Larsson et al 2000). However, Fig. 3 shows that SME values leveled off at protein contents $>11\%$. This result could be related to the fact that the additional lubrication effect of the protein will diminish at higher protein contents, as a result of which, the stress and SME values measured will not decrease any more.

Product Quality Assessment

Cooking loss, swelling index, and firmness were used to determine the quality of the cooked shear-processed pasta-like samples. We used these parameters to see the effect of simple shear processing on quality parameters of pasta-like products and compare it with a reference pasta product.

Cooking Loss

Figure 4 shows the cooking loss (CL) values for undeveloped and shear-developed samples. The shear-processed GS samples had lower CL values compared with unprocessed samples, indicating that the cooking quality of these products improved. It is well-documented that the cooking quality of pasta products is influenced by the formation of protein network surrounding gelatinized starch granules in cooked pasta (Grzybowski and Donnelly 1979; Resmini and Pagani 1983; Guler et al 2002). This could imply that the shearing process develops a gluten network, thereby reducing CL. Both total and soluble CL values decreased in shear-processed GS samples compared with the unprocessed samples. For the starch sample, the total CL of the sheared G0 was higher than that of the unprocessed sample. This might be related to shear-induced fragmentation of granular starch, which occurs at ambient temperatures at moderate shear rates (Vergnes et al 1987; Barron et al 2001).

Figure 4 shows that increasing protein content in shear-processed GS samples leads to a decrease in CL, indicating that the cooking quality of sheared samples is related to the amount of protein in the mixture. However, CL data as a function of protein content appeared to level off for gluten content $>11\%$. These results agree with numerous investigations indicating that the protein content contributes to the cooking quality of pasta products (Dexter and Matsuo 1978; Grzybowski and Donnelly 1979; Fortini 1988; Feillet and Dexter 1996; Guler et al 2002).

The reduction of CL with gluten amount was also seen in unprocessed GS samples. This may be related to the covering effect of gluten protein on starch granules. A more discontinuous protein network was observed in unprocessed GS mixtures (CSLM

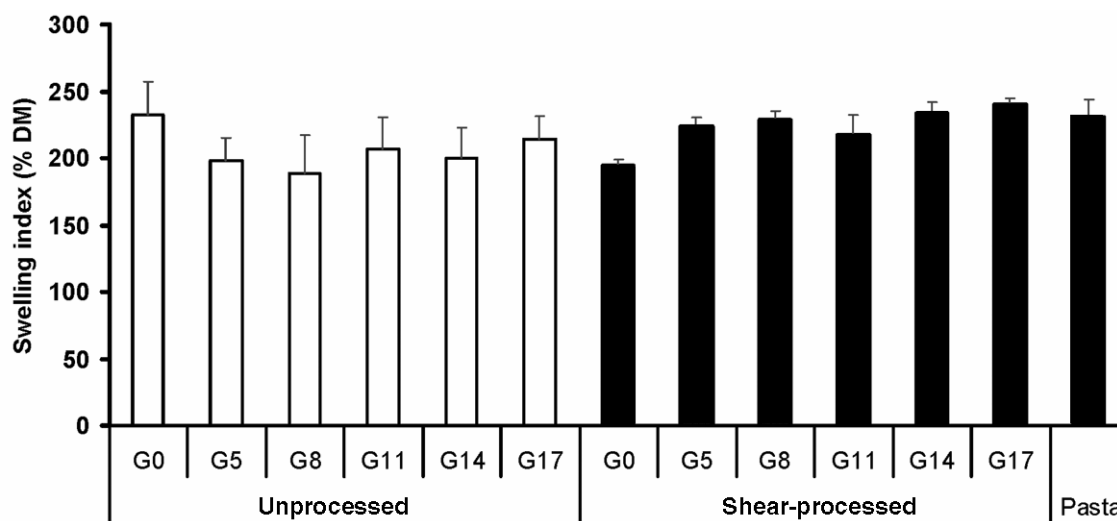


Fig. 5. Effect of shear processing and gluten amount on swelling index of cooked samples. Error bars show 95% confidence intervals.

results). According to Resmini and Pagani (1983), the cooking of a weak or discontinuous protein matrix results in a loose protein structure. It is likely that increasing gluten content could provide better covering effect to the unprocessed GS samples.

Swelling Index

The effect of shear treatment and protein concentration on the swelling index (SI) of the cooked samples is illustrated in Fig. 5. SI values of cooked processed samples were slightly higher than those of the unprocessed samples. This effect parallels the results of CL and would indicate the effectiveness of the applied shearing regime in the structure formation of GS mixtures. There is a universal agreement that protein content is the primary factor influencing pasta quality (as measured by CL, SI, and cooked firmness) and that gluten quality is an important secondary factor (Feillet and Dexter 1996). It has also been reported that swelling of pasta during cooking is dependent on the quality of the surrounding protein network (Grzybowski and Donnelly 1979). There is an incremental trend for SI values as a function of gluten amount, especially at a protein content of >14%. However, the differences are not significant ($\alpha = 0.05$). This agrees with the findings of earlier studies that concluded protein content does not affect SI of pasta products (Grzybowski and Donnelly 1979; Fardet et al 1999). The SI values of all shear-processed samples were in the range of commercial spaghetti.

Cooked Firmness

Cooked firmnesses of both unprocessed and shear-processed GS samples were determined using a texture analyzer. For practical reasons (difference in the geometry and size of GS samples with spaghetti), stress-strain data were extracted from force-deformation curves obtained by this instrument. Figure 6 shows a stress-strain curve for the G8 sample (unprocessed and shear-processed) compared to spaghetti. A similar trend was observed (results not shown) for the other GS samples. According to Fig. 6, the maximum cutting stresses of shear-processed samples (G8) are lower than those of unprocessed samples, implying that the mechanical behavior of the product changed as a result of simple shear processing. The spaghetti sample is slightly firmer than shear-processed GS samples. We are aware of the fact that, in principle, a direct comparison is not possible between freeze-dried samples and extruded durum pasta that has been dried at higher temperatures. However, we were interested whether the mechanical behavior of the sheared product differed largely from the behavior of spaghetti. The mechanical behavior of GS samples and spaghetti was quite comparable.

Maximum stress (σ_{max}) for unprocessed and shear processed samples are shown in Fig. 7. The maximum cutting stresses of all shear-processed samples, except G14, were smaller than those of the unprocessed samples. Apparently, the shearing process led to a formation of a less firm structure. Cooking results (reduced CL)

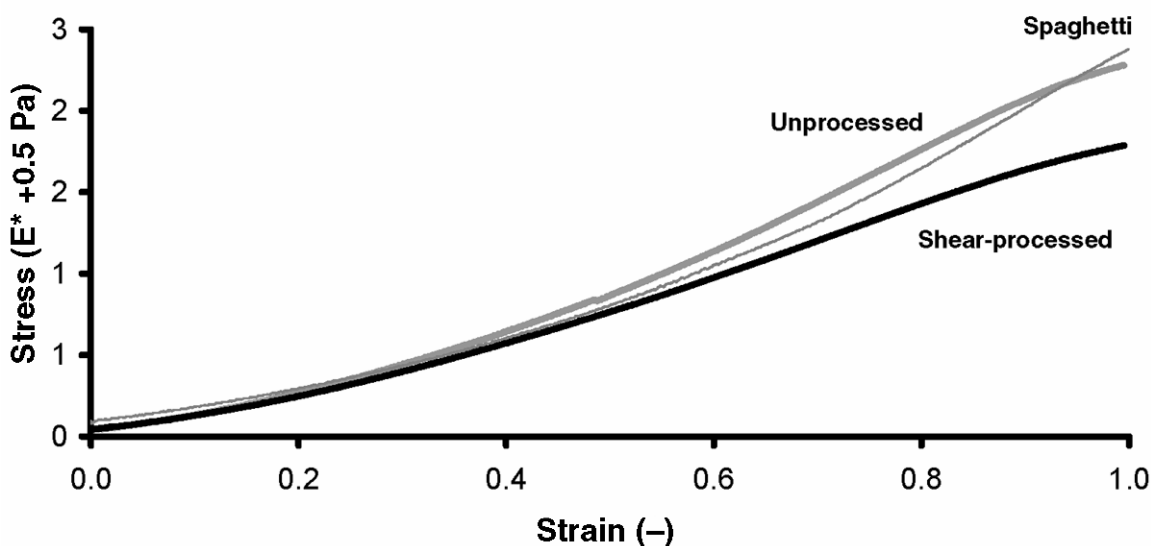


Fig. 6. Typical stress-strain behavior of cooked G8 sample (unprocessed vs. processed) compared to spaghetti. Mean of seven measurements.

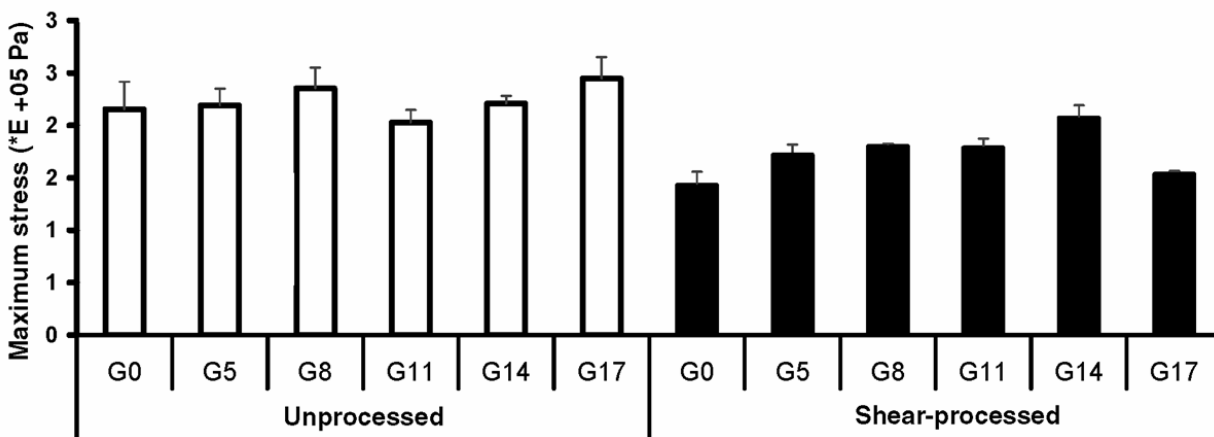


Fig. 7. Effect of shearing and gluten content on maximum cutting stress of cooked gluten-starch (GS) samples. Error bars show 95% confidence interval.

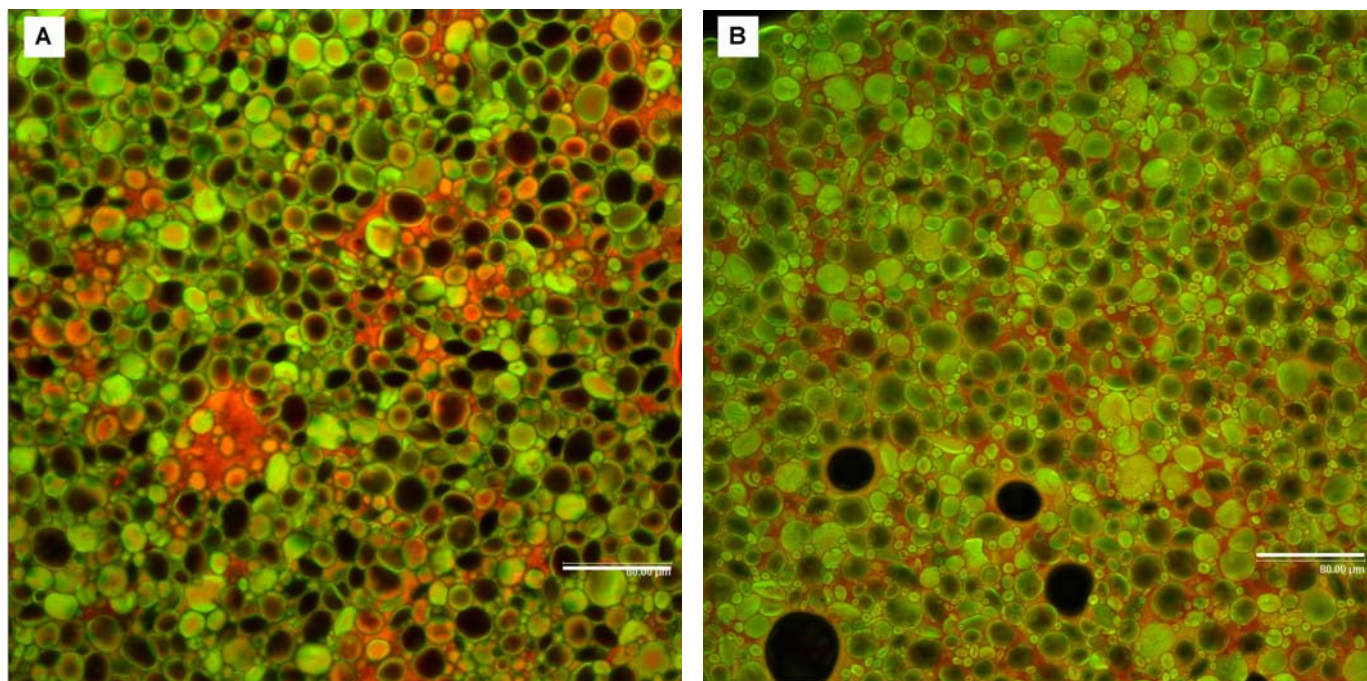


Fig. 8. Micrograph of unprocessed (A) and shear-processed (B) gluten-starch (GS) dough. Scale bar 80 μm .

indirectly indicated the effect of simple shear in the formation of a protein phase in the product, which prevented the loss of starchy material during cooking. A less firm structure formed in the shear-processed samples might be explained by the lubrication effect of protein phase among starch granules in the dough (Eliasson and Larsson 1993) as discussed above. However, there is a reduction in σ_{max} after G14 sample. This is the point that the CL values reached a plateau region, indicating ineffectiveness of further protein content increase in product behavior (possible change in the structure) after adding a given amount of protein.

Study of Dough Microstructure by CSLM Technique

Aside from performing cooking experiments, the CSLM technique was used to visualize at a microscopic level the structure differences caused by the simple shear processing. The microstructure of unprocessed and shear-processed GS dough was studied using CSLM. Figure 8A and B show the microstructure of unprocessed and shear processed G11 dough, respectively. In Fig. 8A, gluten proteins (in red) are in lumps and have not been distributed among the starch granules (in green) yet. Simple shearing resulted in a homogenous distribution of the proteins (Fig. 8B). Simple shear has resulted in the distribution of the gluten material throughout the sample. The starch particles are nicely dispersed in the gluten phase. The results of the CSLM are in line with the results of the cooking experiments (CL, SI, and cooked firmness), which also suggested a good dispersion of starch particle in the gluten phase. It can be concluded that simple shear is able to form a protein network.

CONCLUSIONS

A new method was introduced to study the effect of simple shearing processing on the cooking properties of pasta-like products obtained of wheat gluten-starch (GS). The results of shearing experiments demonstrate the value of this method to study the effect of simple shearing on dough development and properties under process conditions relevant to industrial processing. The results of cooking experiments and stress-strain behavior of cooked products reveal the effect of both shear processing and

protein content on cooking quality. The reduced CL values for shear-processed samples could be explained by the formation of a continuous protein phase in the sheared dough. This was confirmed by CSLM observation of dough microstructure.

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