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Development of Gluten Extensibility Measurement Using Tensile Test

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ABSTRACT

Gluten is a viscoelastic mass obtained from washing wheat flour dough. A simple set-up of tensile test was built to determine gluten extensibility, which is one of the most common measurements used in determining the quality of gluten. The main problem encountered in performing gluten and dough extensibility test is holding of the sample so that it breaks within the sample and not at the jaws that hold the sample. In this research, gluten strips of about 5.0 ± 0.5 g were clamped to the set-up which was attached to Instron 5566 series and then extended at the centre by a hook at crosshead speed of 300 mm min^{-1}. Extensibility parameters such as original gluten length, gluten length at fracture, measured force, actual force acting on the gluten strips, strain, strain rate and stress were obtained using the formulas derived from the results of measurements. The performance of gluten extensibility between strong and weak flour dough were compared. The results of the study showed that gluten obtained from strong flour has greater extensibility compared to weak flour.

Keywords: Extensibility, gluten, tensile test

NOMENCLATURE

A_o	original cross-sectional area of gluten	(mm^2)
A_t	fiknal cross-sectional area of gluten	(mm^2)
d	distance (gap) between the two clips	(mm)
F_m	measured force	(N)
F_a	actual force	(N)
l_o	gluten original length	(mm)
l_t	gluten final length at fracture	(mm)
V_o	original volume of gluten	(mm^3)
V_t	final volume of gluten	(mm^3)
y_o	gluten original position	(mm)
y_t	final hook displacement at gluten fracture	(mm)
α	angle of deformation	(^{o})
ε_H	Hencky strain	(dimensionless)
$\dot{\varepsilon}$	strain rate	(S^{-1})
α	stress	(N mm^{-2})

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INTRODUCTION

A cohesive, viscoelastic dough is obtained when water is mixed with wheat flour. Gluten is a cross-link of protein network developed during mixing of flour-water dough. Water is responsible for hydrating the protein fibrils in wheat flour and start the interactions between the proteins cross links with the disulphide bonds (Faubion and Hoseney, 1989). At the early stage of mixing, gluten fibrils are formed as the water is in contact with flour particles. As the mixing proceeds, more protein becomes hydrated and the glutenins tend to align because of the shear and stretching forces imposed. At this stage, gluten networks are more developed by the cross-linking of protein with disulphide bonds. At optimum dough development, the interactions between the polymers cross-links are becoming stronger which leads to an increase in dough strength, maximum resistance to extension and restoring force after deformation (Letang *et al.*, 1999). When the dough is mixed longer past its optimum development, the cross-links begin to break due to the breaking of disulphide bonds. The glutenins become depolymerised and the dough is overmixed. The presence of smaller chains in the dough makes the dough stickier (Letang *et al.*, 1999).

By washing the dough under running water, the starch is removed and the remaining viscoelastic mass obtained is gluten. Nowadays, the uses of gluten in industry have been intensely applied in various food and non-food industries. Day *et al.* (2006) reported that due to the unique cohesive properties of gluten it has become a commercial material in food industry such as in bakery, breakfast cereals, noodles, sausages and also meat substitutes. Its application has been expanding to other sectors such as pet food, aquaculture feed, natural adhesives and also as biodegradable films.

Rheological properties of gluten are always being connected to the quality of its end product: textural attributes, shape and expansion (Amemiya and Menjivar, 1992; Tronsmo *et al.*, 2003; Anderssen *et al.*, 2004). The rheological properties of gluten and dough were studied in terms of small and large deformation measurements (Amemiya and Menjivar, 1992; Janssen *et al.*, 1996; Uthayakumaran *et al.*, 2002; Tronsmo *et al.*, 2003). Small deformation is a fundamental rheological measurement that involves dynamic oscillation shear measurement. However, Tronsmo *et al.* (2003) found that at small strains, the result of small deformation could not be used as a correlation to the gluten quality as compared to large deformation measurements. Large deformation is more suitable to test the gluten quality used as food product since it can be related to its eating quality. A material experiences a large deformation when the stress exceeds the yield value. The commonly adapted method for large deformation test of dough and gluten is extension. Various instruments are available to perform the extension of dough and gluten such as the extensograph, texture analyser and also Instron. In this test, the sample is clamped at two ends and pulled or extended by a hook at the centre of the sample at a constant speed. Large deformation is applied to the sample until it is fractured and the material is unable to regain the original shape. In the past, many works were done regarding extensibility of gluten and dough using attachment on the Universal Testing Machine such as texture analyser and Instron (Kieffer *et al.*, 1998; Tronsmo *et al.*, 2003; Dunnewind *et al.*, 2004; Sliwinski *et al.*, 2004a; Sliwinski *et al.*, 2004b). Tronsmo *et al.* (2003) performed a uniaxial extension on dough and gluten using the Kieffer dough and gluten extensibility rig for the TA.TX2i texture analyser to test the rheological properties. They used six different wheat flours to study the difference in the breadmaking performance and determined the maximum resistance to extension and total extensibility.

The main problem encountered in performing gluten and dough extensibility test is to hold the sample so that it breaks within the sample and not at the jaws that hold the sample. Thus this research focused on a new tensile test set-up which was built to measure the extensibility of gluten. This new set-up was attached to Instron (5566 series, Instron Corporation, USA). Gluten extensibility was determined by studying the rheological properties of gluten of two types of flour; Diamond N and SP-3.

MATERIALS AND METHODS

Sample Preparation

Two types of flour, Diamond N (12.33% protein) and SP-3 (8.81% protein), were used in this study and referred to as strong and weak flour, respectively. Dough was prepared by mixing 200 g of flour with water (63.4% for strong flour; 59.5% for weak flour) in a mixer (5K5SS, KitchenAid, Belgium) for 8 minutes. Treated drinking water was used to avoid any effect or reaction from other types of minerals on protein in the flour during flour-water mixing. The dough was left to stand in water for 1 hour at room temperature to rest (AACC. 1976). The rested dough was washed under running tap water at a flow rate of 2.5 to 2.8 ml s^{-1} to remove starch until gluten was obtained. At the end of the washing, 1 to 2 drops of water from the gluten was squeezed into a container containing clear water (AACC. 1976). Starch was absent in gluten if cloudiness does not appear. The gluten, dried between dry cloths, was shaped into a ball shape and pressed to a thickness of 10 mm (*Fig. 1*) with the palm. Then, a paper clip with 10 mm gap (*Fig. 2(a)*) was used to press onto the gluten to print 10 mm width strips (*Fig. 1*) as a guide for cutting using a paper cutter (*Fig. 2(b)*). Finally, the strips were cut to 70 mm length. The 10 mm × 10 mm × 70 mm gluten strips of approximately 5.5 ± 0.5 g were immersed in tap water at room temperature and left for 30 minutes to rest (Chen *et al.*, 1998; Chiang *et al.*, 2006).

Extensibility Set-up

The rested gluten strips were then clamped at two ends using plastic clips arranged at 40 mm distance nailed to a 15.2 cm × 21.6 cm wooden platform cut according to the size of the Instron base platform. The wood was held tightly to the Instron platform using a

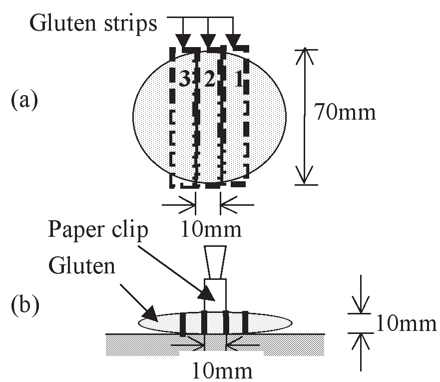

Fig. 1: Gluten imprint using paper clip (a) top (b) cross-sectional view

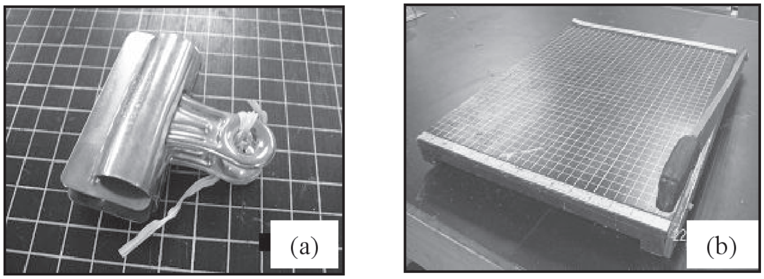

Fig 2: (a) Paper clip used to print 10 mm width of the gluten strips and (b) paper cutter used for gluten cutting

G-clamp. The tensile test started as the gluten was pulled up by the hook at a speed of 300 mm min^{-1} and stopped when the gluten fractured. The tensile test set-up (*Fig. 3*) consists of a hook bent into a V-shaped using a metal rod of 3.2 mm diameter and fitted to the Instron (5566 series, Instron Corporation, USA). The clip was set 10 mm above the wood plane for easy opening of the clamps when placing the gluten strips. *Fig. 4* shows the schematic diagram of a tensile test set-up at top and side views. To ensure that the gluten does not bend during placement on the set-up, the hook was levelled with the lower part of the plastic clips as shown in *Fig. 4(b)*.

The measured force (F_m) was exerted on the gluten at a vertical axis as shown in *Fig. 5*. Extensibility parameters: the original length of gluten (l_o), the final length of gluten at fracture (l_t) and actual force (F_a), and rheological parameters: strain (o_H), strain rate and stress (ε_H), were determined.

(i) Derivation of Extensibility Parameters

Equation [1] was used to determine the original length of gluten (l_o) before extension. d was 40 mm in this study. The final length of gluten at fracture (l_t) was calculated using equation [2]:

$$l_o = 2\sqrt{\left(d/2\right)^2 + \left(y_o\right)^2} \qquad (1)$$

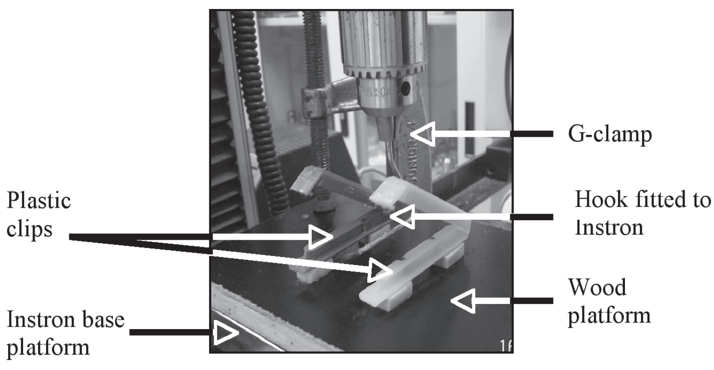

Fig. 3: Tensile test set-up for gluten extensibility on Instron (5566 series, Instron Corporation, USA)

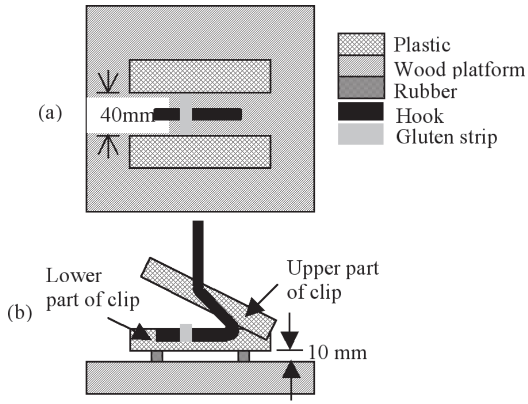

Fig. 4: Tensile test set-up diagram from(a) top and (b) side view

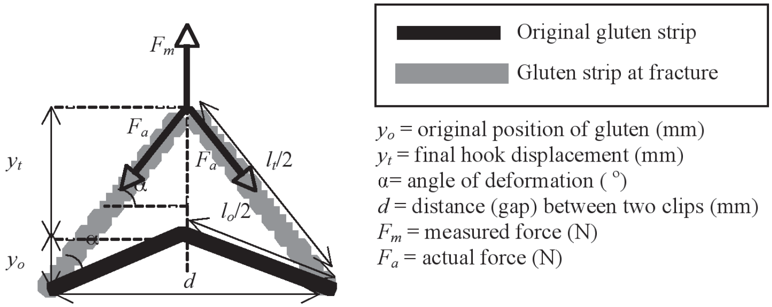

Fig. 5: Schematic diagram of forces acting on gluten and the length of gluten during tensile test [10]

$$l_t = 2\sqrt{(d/2)^2 + (y_o + y_t)^2} \tag{2}$$

Assuming that the hook passes exactly through the centre of the gap, the measured force (F_m) was divided equally over both stretched gluten at each side of the hook (Kieffer *et al.* 1998). Thus, the actual force (F_a) that acted upon the stretched gluten was determined using equation [4] while equation [3] shows the expression of the angle of deformation (α) in terms of the measured and actual force acting upon the gluten.

$$\sin\alpha = \frac{F_m/2}{F_a} = \frac{y_t + y_o}{l_t/2} \tag{3}$$

$$F_a = \frac{F_m l_t}{4(y_t + y_o)} \tag{4}$$

(ii) Derivation of Rheological Parameters

The extension parameters obtained earlier were used to determine the rheology parameters such as strain, strain rate and stress. The Hencky strain (ε_H) acting on gluten was calculated using equation [5] and the strain rate was calculated by a derivative of Hencky strain $(\dot{\varepsilon})$ with time as shown in equation [6]:

$$\varepsilon_H = \ln\left(\frac{\sqrt{(\mathrm{d}/2)^2 + (y_o + y_t)^2}}{\sqrt{(d/2)^2 + (y_o)^2}}\right) \tag{5}$$

$$\dot{\varepsilon} = \frac{d\varepsilon_H}{dt} = \frac{dl}{l_t dt} = \frac{1}{l_t} \cdot \frac{2(y_t + y_o)}{\sqrt{9^2 + (y_t + y_o)^2}} \cdot \frac{dy_t}{dt} = \frac{4v(y_t + y_o)}{l_t^2} \tag{6}$$

where v is the speed of hook (mm min^{-1}). The final cross-sectional area of gluten strip can be calculated by assuming the volume of gluten was constant throughout the test (Muller *et al.*, 1961; Sliwinski *et al.*, 2004a) as shown in equation (7).

$$\begin{aligned} V_o &= V_t \\ A_o l_o &= A_t l_t \\ A_t &= \frac{A_o l_o}{l_t} \end{aligned} \tag{7}$$

where V_o is the original volume of gluten (mm^3), V_t is the final volume of gluten (mm^3), A_o is the original cross-sectional area of gluten (mm^2) and A_t is the final cross-sectional area of gluten (mm^2). From equation (8), the stress (σ) acting on the gluten was calculated by dividing the actual force (F_a) with the final cross-sectional area of gluten strip (A_t).

$$\sigma = \frac{F_a}{A_t} \tag{8}$$

Data Analysis

The experiments were conducted using three replications. The mean value and standard deviation of three replications were calculated using Microsoft Excel. Data from the force-extension graph obtained from Instron was used to calculate the extensibility parameters. Curves of strain-hook extension, strain rate-hook extension and stress-strain were obtained to study the performance of the tensile test set-up.

RESULTS AND DISCUSSION

Figs. 6(a) to *6(d)* illustrate the tensile test for gluten extensibility from the beginning until the fracture of gluten. The gluten strip bent slightly upward at the hook as it was clamped (*Fig. 6 (a)*). This explains the original hook position (y_o) in equation (1) which is to prevent bending of the gluten sample. Previous studies by Uthayakumaran *et al.* (2002) and Dunnewind *et al.* (2004) reported that precaution has to be taken to prevent sagging during clamping of the test sample. *Fig. 6(b)* shows the gluten being pulled upward as the hook was moving at a crosshead speed 300 mm min^{-1}. Studies on the effect of various speeds on the extension of dough and gluten piece have been done (Dunnewind *et al.*, 2004; Sliwinski *et al.*, 2004a; Sliwinski *et al.*, 2004b) and the results showed that the deformation at fracture increased with increasing speed. *Fig. 6(c)* shows that as the hook was displaced further upward the gluten strip became thinner at point 2 and 4 before it fractured (*Fig. 6(d)*) at its maximum extensibility. In this set-up, the gluten test piece did not fracture at the clamping area.

Fig. 7(a) shows the typical force-extension curve for gluten from strong and weak flour mixed for 8 minutes. For both flours, an increase of force was observed with increasing hook displacement and decreased after reaching a peak. A similar trend was reported for gluten and dough in uniaxial extension tests (Dunnewind *et al.*, 2004; Sliwinski *et al.*, 2004a; Sliwinski et al., 2004b). Generally these curves resemble the curves from extensograph measurements. From these curves, the force needed to extend the gluten increased during tensile deformation and reached a maximum before gluten ruptured and then decreased after rupture. It was observed that gluten from strong flour was more extensible than weak flour as indicated by the higher measured and actual force, hook displacement, final length at fracture, stress, strain and strain rate (Table 1).

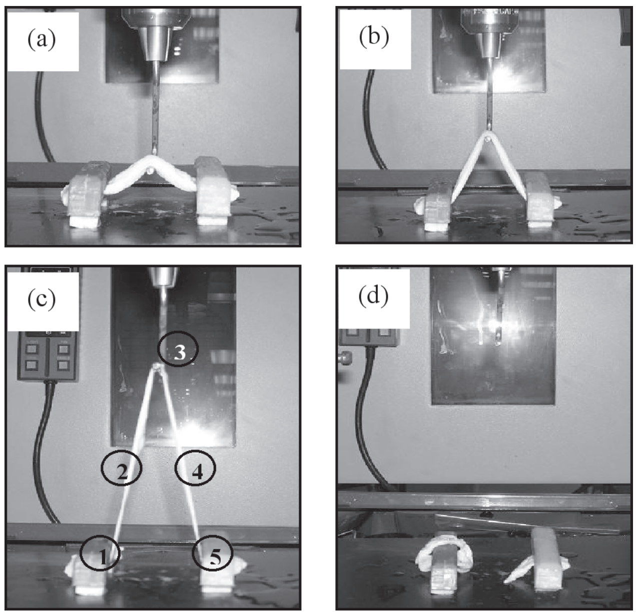

Fig. 6: Tensile test showing gluten extensibility at various stages: (a) gluten clamped at clips (b) gluten pulled upward by hook (c) gluten became thinner (d) gluten fractured

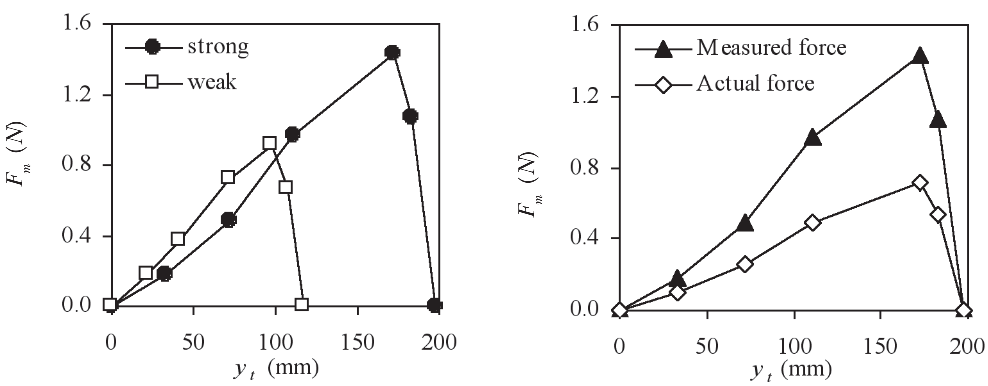

Fig. 7: (a) Measured force-hook displacement curve for gluten from strong and weak flour (b) Measured and actual force versus hook displacement for gluten from strong flour

Higher force and extensibility of strong flour gluten suggests that strong flour has stronger gluten network and the extensibility was influenced by the protein content of the flour (C'uric' *et al.*, 2001). *Fig. 7(b)* shows the curves of measured and actual force against hook extension for gluten from strong flour. It was found that the measured force was double the actual force acting on the gluten (Dunnewind *et al.*, 2004).

Figs. 8(a) and *(b)* show the strain and strain rate versus hook displacement curves for strong and weak flour mixed for 8 minutes. From these curves, strain increased and strain rate increased and reached a maximum then decreased as the hook displaced upward. These curves gave similar patterns as the extensograph and the Kieffer rig (Dunnewind *et al.*, 2004). Strain increased as the gluten extended upward and reached a maximum at gluten fracture. It was observed that strain rate for weak flour gluten was higher than for strong flour at the beginning of the extension. As the hook expanded more, the strain rate of both flours was slightly the same.

In *Fig. 9*, the stress-strain curves determined in the extension are shown for gluten from strong and weak flour mixed for 8 minutes. Both flours show an increase in stress with increasing strain and reached a peak at fracture of a sample. In the stress-strain

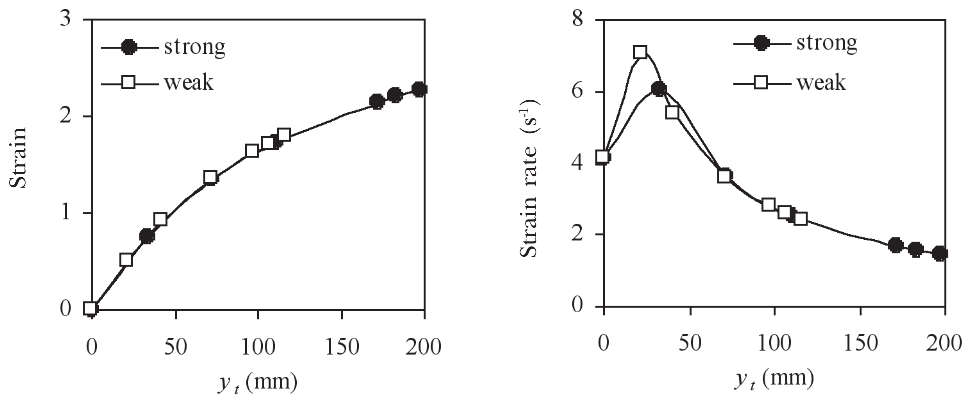

Fig. 8: Curves of (a) Hencky strain (b) Strain rate versus hook extension for gluten from strong and weak flour

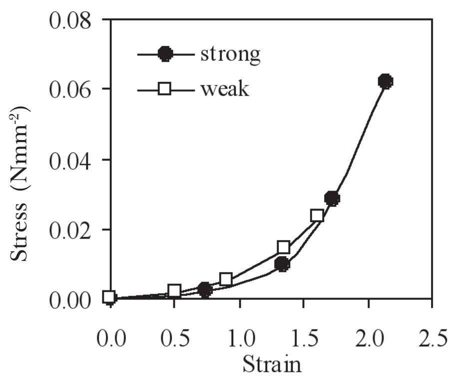

Fig. 9: Stress-strain curve for gluten from strong and weak flour. The point of fracture is indicated with an arrow

TABLE 1
Extensibility parameters for gluten of strong and weak flour mixed for 8 minutes

Flour	y_t(mm)	F_m(N)	l_t(mm)	F_a(N)	ε_H	$\dot{\varepsilon}$ (s^{-1})	σ(N mm^{-2})
Strong	0 ± 0.0	0 ± 0.00	41.8 ± 0.0	0 ± 0.00	0 ± 0.00	4.13 ± 0.00	0 ± 0.0000
	33.3 ± 0.0	0.18 ± 0.01	88.3 ± 0.0	0.10 ± 0.01	0.75 ± 0.00	6.06 ± 0.00	0.0021 ± 0.0001
	72.2 ± 5.6	0.49 ± 0.02	161.5 ± 10.8	0.25 ± 0.01	1.35 ± 0.06	3.62 ± 0.21	0.0098 ± 0.0009
	111.1 ± 11.1	0.97 ± 0.04	237.7 ± 21.9	0.49 ± 0.02	1.73 ± 0.09	2.53 ± 0.21	0.0282 ± 0.0033
*	**172.2 ± 5.6**	**1.43 ± 0.15**	**325.6 ± 11.0**	**0.72 ± 0.08**	**2.14 ± 0.03**	**1.66 ± 0.05**	**0.0618 ± 0.0060**
	183.3 ± 4.8	1.07 ± 0.24	369.7 ± 9.6	0.54 ± 0.12	2.21 ± 0.03	1.57 ± 0.04	0.0489 ± 0.0101
	197.2 ± 2.8	0 ± 0.00	397.4 ± 5.5	0 ± 0.00	2.28 ± 0.01	1.46 ± 0.02	0 ± 0.0000
Weak	0 ± 0.0	0 ± 0.00	41.8 ± 0.0	0 ± 0.00	0 ± 0.00	4.13 ± 0.00	0 ± 0.0000
	22.2 ± 2.8	0.18 ± 0.06	69.3 ± 4.4	0.11 ± 0.04	0.50 ± 0.07	7.04 ± 0.20	0.0018 ± 0.0007
	41.7 ± 0.0	0.37 ± 0.03	103.4 ± 0.0	0.20 ± 0.02	0.91 ± 0.00	5.35 ± 0.00	0.0049 ± 0.0004
	72.2 ± 2.8	0.72 ± 0.06	161.5 ± 5.4	0.37 ± 0.03	1.35 ± 0.03	3.61 ± 0.12	0.0143 ± 0.0010
*	**97.2 ± 2.8**	**0.91 ± 0.07**	**210.3 ± 5.5**	**0.46 ± 0.04**	**1.62 ± 0.03**	**2.80 ± 0.07**	**0.0234 ± 0.0023**
	106.9 ± 1.4	0.66 ± 0.06	229.4 ± 2.7	0.34 ± 0.03	1.70 ± 0.01	2.58 ± 0.03	0.0185 ± 0.0018
	116.7 ± 4.8	0 ± 0.00	248.6 ± 9.5	0 ± 0.00	1.78 ± 0.04	2.39 ± 0.09	0 ± 0.0000

Bold and * – values for gluten at fracture
± standard deviation of mean of three replications

curves, the point of fracture of the gluten sample is indicated. The fracture stress and strain determined for gluten mixed for 8 minutes is shown in Table 1. The gluten from weak flour showed lower value for fracture stress compared to the strong flour due to the low stress-level, low strain hardening and the small fracture strain. These results are in agreement with previous observations (Sliwinski *et al.*, 2004a; Sliwinski *et al.*, 2004b).

CONCLUSIONS

Determining the extensibility of gluten using the tensile test set-up was successful in terms of providing the extensibility measurements. The extensibility parameters of gluten from strong flour gave higher values than for weak flour in terms of the length at fracture, measured and actual force, strain and also stress.

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