# ASSESSMENT OF VARIATION BASED ON TEMPERATURE, CREEP-RECOVERY, EXTENSIBILITY TESTS, AND GLUTEN CONTENT IN HARD RED WINTER WHEAT COMPARED WITH TRADITIONAL WHEAT QUALITY TESTING

By

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iii

# TABLE OF CONTENTS

Chapter		Page
I. INTRODUCT	TION	1
Statement of Purpose of st Hypothesis Assumptions	f problems tudy s	
II. REVIEW OF	<sup>7</sup> LITERATURE	5
Gluten qualit Rheological Rheological Influence of	ty properties of gluten assessments high temperature on gluten	
III. DISCRIMIN HARD RED AT DIFFER	VATION OF VISCOELASTIC PROPERTIES OF WINTER WHEAT GLUTEN BY USING CREI CENT TEMPERATURES	F COMMERCIAL EP-RECOVERY TEST
Abstract		11
1. Introduct	tion	
2. Materials	s and methods	16
2.1 Mater 2.2 Exper 2.2.1 Gluter 2.2.2 Creep 2.2.3 Statist	rials rimental on preparation o and recovery test of gluten stical Analysis	

Chap	ter	Pa
3. R	esults and Discussions	1
3.1	Viscoelastic properties	1
3.2	Principal Component Analysis (PCA), Factor analysis	and
	Pearson correlation	2
4. Co	nclusion	2
. VAR FARI RED	IATION IN GLUTEN VISCOELASTICITY, DOUGH NOGRAPH AND BAKING PROPERTIES AMONG ( WINTER WHEAT	EXTENSIBILITY, COMMERCIAL HA
Abstr	act	3
1. In	troduction	3
2. M	laterials and methods	4
2.1	Material	4
2.2	Experimental	4
2.2.1	Gluten preparation	4
2.2.2	Dough preparation	4
2.2.3	Creep and recovery test of gluten	4
2.2.4	Extensibility of dough	4
2.2.5	Farinograph test	4
2.2.6	Baking test	4
2.2.7	Statistical analysis	4
3. R	esults and discussion	4
	Principal Component Analysis (PCA)	4
3.1	The convelation between monenties by Deguage course	lation4
3.1 3.2	The correlation between properties by Pearson corre	
3.1 3.2 <i>3.2.1</i>	The viscoelastic properties of gluten	4
3.1 3.2 3.2.1 3.2.2	The viscoelastic properties of gluten Extensibility	4
3.1 3.2 3.2.1 3.2.2 3.2.3	The viscoelastic properties of gluten Extensibility Dough mixing properties	
3.1 3.2 3.2.1 3.2.2 3.2.3 3.2.4	The correlation between properties by Pearson corre The viscoelastic properties of gluten Extensibility Dough mixing properties Baking properties	

V.	ASSESSMENT OF VARIATION IN HARD RED WINTER WHEAT FLOUR PROPERTIES FROM CREEP-RECOVERY, EXTENSIBILITY TESTS AND GLUTEN CONTENT COMPARED WITH TRADITIONAL WHEAT QUALITY TESTING			
	Abstra	ct	61	
	1. Int	roduction	63	
	2. Ma	aterials and methods	65	
	2.1	Material	65	
	2.2	Experimental	65	
	2.2.1	Gluten preparation	65	
	2.2.2	Creep and recovery test of gluten	65	
	2.2.3	Tensile test of gluten	66	
	2.2.4	Gluten index and wet gluten measurement	64	
	2.2.5	Mixograph test	65	
	2.2.6	Sodium Dodecyl Sulfate SDS Sedimentation	67	
	2.2.7	Baking test	67	
	2.2.8	Statistical analysis	67	
	<i>3</i> . Re	sults and discussion	68	
	3.1	The properties of 2008 and 2009 sample flours	68	
	3.1.1	Creep and recovery test	68	
	3.1.2	Tensile test	68	
	3.1.3	Glutomatic measurements	69	
	3.1.4	Mixograph		
	3.1.5	SDS sedimentation		
	3.1.6	Baking test	70	
	3.2	The correlations among properties of 2008 sample flours	71	
	3.3	The correlations among properties of 2009 sample flours	73	
	4. Co	nclusion	74	
VI	. REFI	ERENCES	92	
VI	I. CON	VCLUSION	97	
1/I	רידם דו		00	
۷I	II. FUI			
AF	PEND	X 1		

## LIST OF TABLES

Table

Page

# CHAPTER III. DISCRIMINATION OF VISCOELASTIC PROPERTIES OF COMMERCIAL HARD RED WINTER WHEAT GLUTEN BY USING CREEP-RECOVERY TEST AT DIFFERENT TEMPERATURES

1.	Proximate analysis of flours (means $\pm$ SD, n=2)25	
2.	Analysis of variance for viscoelastic properties of glutens treated with temperatu	re
3.	Analysis of variance for viscoelastic properties of glutens in each temperature	
4.	a) Least Squares Means of temperature x flour sample for the viscoelastic proper	ties
	of gluten using a creep and recovery test	
	b) Least Squares Means of temperature x flour sample for the viscoelastic proper	ties
	of gluten using a creep and recovery test	
5.	a Analysis of variance for %Recoverability (RCY) of glutens treated with	
	temperature by Least Squares Means	
	b) Analysis of variance for %Recoverability (RCY) of glutens treated with	
	temperature by Differences of temperature Least Squares Means Adjustment for	
	Multiple Comparison: Tukey	
6.	Pearson's correlation coefficients of the viscoelastic properties of gluten31	
7.	Explained variance (%) in Principal Component Analysis (PCA) of viscoelastic	
	properties gluten	
8.	Factor analysis of viscoelastic properties of gluten	

Table

## CHAPTER IV. VARIATION IN GLUTEN VISCOELASTICITY, DOUGH EXTENSIBILITY, FARINOGRAPH AND BAKING PROPERTIES AMONG COMMERCIAL HARD RED WINTER WHEAT

1.	Proximate analysis of flours (means ± SD, n=2)
2.	Explained variance (%) in PCA of the viscoelastic properties of gluten at 40 and
	100 Pa, extensibility of dough, farinograph, and baking characteristics
3.	Explained variance (%) in PCA of the viscoelastic properties of gluten at 100 Pa,
	extensibility of dough, loaf volume, and flour protein
4.	Explained variance (%) in PCA of selected variables after discarding redundant
	variables of Table 254
5.	Explained variance (%) in PCA of selected variables after discarding redundant
	variables of Table 355
6.	Pearson's correlation coefficients of the viscoelastic properties of gluten,
	extensibility of dough, farinograph, and baking characteristics

# CHAPTER V. ASSESSMENT OF VARIATION IN HARD RED WINTER WHEAT FLOUR PROPERTIES FROM CREEP-RECOVERY, EXTENSIBILITY TESTS AND GLUTEN CONTENT COMPARED WITH TRADITIONAL WHEAT QUALITY TESTING

1.	a) Mean values of 2008 wheat cultivars and breeder lines
	b) Mean values of 2008 wheat cultivars and breeder lines
	c) Mean values of 2008 wheat cultivars and breeder lines
2.	a) Mean values of 2009 wheat cultivars and breeder lines
	b) Mean values of 2009 wheat cultivars and breeder lines
	c) Mean values of 2009 wheat cultivars and breeder lines
3.	Explained variance (%) in PCA of 2008 wheat flours
4.	Explained variance (%) in PCA of 2009 wheat flours
5.	Explained variance (%) in PCA of selected variables after discarding redundant
	variables of 2008 wheat flours. (Table 1)
6.	Explained variance (%) in PCA of selected variables after discarding redundant
	variables of 2009 wheat flours. (Table 2)
7.	Pearson's correlation coefficients of 2008 wheat cultivars and breeder lines86
8.	Pearson's correlation coefficients of 2009 wheat cultivars and breeder lines87

## LIST OF FIGURES

#### Figure

#### Page

# CHAPTER III. DISCRIMINATION OF VISCOELASTIC PROPERTIES OF COMMERCIAL HARD RED WINTER WHEAT GLUTEN BY USING CREEP-RECOVERY TEST AT DIFFERENT TEMPERATURES

# CHAPTER IV. VARIATION IN GLUTEN VISCOELASTICITY, DOUGH EXTENSIBILITY, FARINOGRAPH AND BAKING PROPERTIES AMONG COMMERCIAL HARD RED WINTER WHEAT

- 4. Loading plot of first two principal components of selected variables after discarding redundant and low contributor variables of Table 3 based on viscoelastic using shear stress at 100 Pa and baking properties of six commercial wheat flours ....60

# Figure

1.	Loading plot of first two principal components of 2008 set of 51 samples wheat
	flours
2.	Loading plot of first two principal components of 2009 set of 51 samples wheat
	flours
3.	Loading plot of selected variables after discarding redundant variables of 2008 set
	of 51 samples wheat flours
4.	Loading plot of selected variables after discarding redundant variables of 2009 set
	of 51 samples wheat flours

#### CHAPTER I

#### INTRODUCTION

#### Statement of problem

Inconsistency of wheat end user quality traits has long been a problem for milling and baking industry. Wheat breeders can improve the variation of the overall end-use quality of cultivars through evaluation and selection. The quality of pan bread in the baking industry is mainly related to dough characteristics. Gluten protein is the major and most crucial component of dough associated directly with bread quality. Dough and gluten have a complex viscoelastic behavior. It has been long known that gluten mostly consists of glutenin which provides its elastic properties and gliadin which plays a role on its viscous behavior. Rheological assessments have been commonly applied for testing the viscoelastic properties of dough and gluten, and correlations have been found with product quality for breadmaking. Flour quality is mostly determined at room temperature except for baking. The structure of gluten protein is affected by the breadmaking process. There are, however, only a few fundamental studies on the gluten behavior using creep and recovery tests, particularly at high temperature. Heat during the breadmaking process plays a key role in denaturation of the structure of wheat gluten protein. Viscoelastic properties of gluten can be affected by temperature during processing. The conformation and the irreversible changes in the viscoelasticity of gluten have not been fully understood. Many studies have shown the alteration of the

physicochemical gluten properties when processed at temperature higher than 25°C. The viscoelastic properties of gluten are reliably characterized by rheological measurements. Therefore, the dynamic measurements and retardation tests (creep and recovery method) are good candidates to distinguish the gluten quality in wheat varieties. For practical measurements, the use of gluten is easier compared to the use of dough in rheological measurement mainly due to determining the optimum absorbance of dough which is time consuming. There are various methodologies to test wheat quality for breeding programs and for the baking industry. Empirical and rheological tests (small and large deformation) are the most common measurements used for determining and monitoring wheat quality.

# **Purpose of study**

The aims of this study were to 1) discriminate commercial hard red winter wheat viscoelastic properties of gluten associated with an effect of temperature and 2) differentiate commercial and breeder lines of hard red winter wheat properties from creep-recovery, extensibility tests and gluten content compared with traditional wheat quality testing.

#### **Objectives**

- To evaluate differentiation of the commercial hard red winter wheat flour properties using a creep-recovery test of gluten at temperatures ranging from 25 to 55°C
- 2) To discriminate the commercial hard red winter wheat flour properties by using the gluten creep-recovery test, farinograph, baking, and dough extensibility test

 To compare explanation of the variance by traditional measurements with gluten creep-recovery, gluten extensibility and glutomatic tests of two sets of winter wheat sample properties grown in 2008 and 2009

#### **Hypothesis**

Viscoelastic properties of gluten can improve the separation of differences in flour quality when analyzed at temperature higher than the 25°C. The creep-recovery of gluten using two different stresses at 40 and 100 Pa and extensibility of dough and gluten content can also improve the explanation of commercial and breeder lines flour properties and can be used as indicators of quality. This may improve the discrimination of differences in quality of flour samples.

#### Assumptions

Heat treatment affects viscoelastic properties of gluten by disrupting the disulfide bonds and changing hydrogen bonds and non-polar hydrophobic interactions. Heat increases the kinetic energy by causing vibration of the molecules and affects the forming and reforming of gluten bonds. The breakdown of secondary covalent and non-covalent bonds can affect the viscoelastic behavior of gluten. Depending on the intrinsic properties of gluten and its interactions with other components, the quality of wheat flour can be distinguished when tested with a creep and recovery test with temperature dependent experiments.

Parameters obtained from creep-recovery, extensibility and glutomatic tests can explain wheat flour properties more than traditional wheat quality testing. We assume

that testing wheat flour samples with creep-recovery, extensibility and glutomatic tests can improve a variation among hard red winter wheat samples.

#### CHAPTER II

#### LITURATURE OF REVIEW

#### **Gluten quality**

Gluten protein plays an important role in food products by altering the firmness and texture of the end product especially in bakery goods like bread, cookies, and cakes. The main components that make up gluten are gliadins, glutenins and other minor components like lipids (3.5-6.8%), minerals (0.5-0.9%), and carbohydrate (7.0-16.0%) (Song and Zheng, 2007). The quality of pan bread is usually explained based on the viscoelastic properties of dough and gluten. Strong gluten flour will have a higher elasticity and lower viscosity (Khatkar et al., 1995; Song and Zheng, 2007). There are numerous factors that will affect the gluten quality based on its solubility, extractability, structure, and physical formation. The end-use product quality is highly correlated with the genetic background in each wheat variety (Wang et al., 2004). It is well established that both the variety and environment where the wheat is grown will influence the quality of gluten. Pentosans are important fiber components in cereals. They have been related to dough-handling and baking performance (Delcour et al., 1991; Michniewicz et al., 1991; Wang, van Vliet et al., 2004). It has been proposed that pentosans affect the physical and chemical properties of gluten (Wang, van Vliet et al., 2004).

The proposed physical effects on gluten are to influence viscosity and the attraction between protein particles. The latter one most likely is due to the charged ferulic acid in pentosans (Wang, van Vliet et al., 2004). Among the proposed chemical effects are the influence of ferulic acid molecules which regulated the aggregation of gluten and the tendency of the glutenin macro-polymer (GMP) gel to aggregate (Wang, van Vliet et al., 2004). The influence of water un extractable solids (WUS) on gluten formulation revealed that wheat with WUS decreased gluten and starch yield and increased glutenin macro-polymer (GMP) gel formation (Wang et al., 2003).

Tensile test is used to evaluate the viscoelastic properties of a sample when a certain amount of stretch is applied. The strength of the gluten can be used to evaluate the gluten quality. Tschoegl et al. (1970) evaluated the strength of gluten by applying a pulling force to the sample to pull upward at a steady speed until it reached a rupture point. Based on the deformation of the sample, it was concluded that strong gluten will have a higher elastic deformation compared to weak gluten (Tschoegl et al., 1970). The quality of wheat gluten can be investigated by various approaches; however, there is still no evidence indicating which method is the most suitable measurement for each application.

#### **Rheological properties of gluten**

A number of aspects of wheat quality have been studied for several decades including gluten and dough characteristics in reference to mixing and baking functionality. Although important advances in knowledge have been made, many challenges remain to be addressed, such as an understanding of the basic mechanism of

interactions of gluten components and its unique functional properties. The composition of gluten is well established with major components being glutenin and gliadin (49.1% and 30%, respectively) and minor components being lipids (3.5-6.8%) minerals (0.5-(0.9%), and carbohydrate (7.0-16.0%) (Song and Zheng, 2007). But it is the three dimensional structure formed by gluten polymeric and monomeric proteins that has been attributed to dominate the fundamental mechanical properties and thus the degree of suitability for specific applications of different flours. The properties of gluten measured under dynamic rheology in the linear viscoelastic region have revealed differences in elasticity and viscosity of wheat with a wide range of strength (extra strong to weak) and baking potential (good and poor) (Khatkar and Schofield, 2002). Dynamic rheological properties of gluten can describe the structure formed and the relationship to processing parameters of dough, in particular G' (elastic modulus) to baking properties (Khatkar and Schofield, 2002). Examples of factors affecting the structure include the degree of crosslinking in the gluten. As the high degree of gluten crosslinking appears, it will increase the G' and decrease G" (loss modulus) (Mirsaeedghazi et al., 2008).

Glutenins and gliadins are the two major storage proteins responsible for viscosity and elasticity of dough and gluten (Song and Zheng, 2007). The ratio of gliadin/glutenin and high molecular weight/low molecular weight (HMW-GS/LMW-GS) have been proposed to explain the gluten viscoelastic properties (Popineau et al., 1994). It has been widely accepted that protein aggregation and size distribution are affected by the HMW-GS present in glutenin (Song and Zheng, 2007). Also, an increase of elastic plateau modulus of gluten network is induced by the aggregation of glutenin (Popineau, Cornec

et al., 1994). While, gliadins provide the viscosity when the hydrated gluten is formed (Wieser, 2007).

#### **Rheological assessments**

The viscoelastic properties of dough have been extensively analyzed and manipulated in order to obtain the most suitable properties for baking process. Fundamental rheological properties can be analyzed by applying a large or small deformation to viscoelastic mass gluten over time (Dobraszczyk and Morgenstern 2003). Various parameters are obtained to identify gluten properties such as stress, strain, stiffness, modulus, viscosity, hardness, and strength of gluten. However, this fundamental assessments have some challenges such as the high price of the instrument, long experimental time, skills on using the instrument, and difficulty in the interpretation of data (Dobraszczyk and Morgenstern, 2003).

Many rheological measurements commonly applied to wheat measurements are small and large deformation, shear creep and stress relaxation, large deformation extensional test, small deformation dynamic shear oscillation, and flow viscometry depending on the demanded parameters. In a creep and recovery test, a steady stress is applied to the dough or gluten and the elastic and viscous responses are obtained. Tensile test, Simon Research Extensometer, Brabender Extensigraph, Kieffer dough and gluten extensibility rig are classified as large deformation extensional experiments which are the most commonly tests (Dobraszczyk and Morgenstern, 2003). During the large deformation extensional tests, a force is applied to stretch the material and a graph of

force versus distance is obtained. However, the extension test cannot provide any rheological responses in stress, strain or viscosity.

#### Influence of high temperature on gluten

Almost all processes such as mixing, sheeting, extrusion, drying, and cooking in baking industry involve heat. The viscoelastic properties of gluten during heat treatments and its thermal stability have been studied as it relates to their potential to evaluate differences in gluten quality (Kovacs et al., 2004). The gluten thermo stability and the ratio of insoluble glutenins to total monomeric proteins (gliadins and low molecular weight-glutenin subunits) have been reported as potential indicators of flour quality evaluation (Kovacs, Fu et al., 2004). Low ratios of monomeric to insoluble glutenin decreased the thermostability of gluten and therefore affected the gluten strength (Kovacs, Fu et al., 2004). The same authors also reported that allelic variations of HMW-GS were independent of the gluten thermostability and most of the dough and/or gluten strength tests.

Conformation and molecular size of gluten protein also can be modified by heat treatment during the baking process (Hayta and Schofield, 2004). In addition, the aggregation and extractability of gluten can be altered by exposing it to high temperature. High temperature affects protein aggregation by decreasing the extractability of gluten protein (He and Hoseney, 1991; Schofield et al., 1983). In terms of the aggregation and extractability properties, in high breadmaking quality flours, there is a higher aggregation and lower extractability than those of a low breadmaking quality. Also, heat induces the development of intermolecular covalent bonds related to higher aggregation of the gluten

structure in strong gluten (Wieser, 2007). The gluten qualities from different wheat varieties associated with heat was studied by Hayta and Schofield (2004). Gluten heated at temperatures reaching 70 to 90 °C caused decrease of Sodium Dodecyl Sulfate (SDS) extractability and increase of sulphydryl (SH) and disulphide (SS) contents (Hayta and Schofield, 2004). The same authors suggested that non-covalent and covalent interaction of gluten might have been affected by heating.

#### CHAPTER III

# DISCRIMINATION OF VISCOELASTIC PROPERTIES OF COMMERCIAL HARD RED WINTER WHEAT GLUTEN BY USING CREEP-RECOVERY TEST AT DIFFERENT TEMPERATURES

#### Abstract

Gluten quality is one of the most desired characteristics in the production of pan bread in the baking industry. An effective characterization of wheat gluten during heating using rheological methodology can reveal important practical and basic properties of this important component. Six commercial flour samples (hard red winter type) and one soft red cultivar (Stephens) varying in protein content were studied. Viscoelastic properties of the isolated gluten were measured at 25, 35, 45 and 55°C using a creep and recovery test to separate the viscous flow and elastic recovery components of the gluten and were illustrated in Principal Component Analysis (PCA). The total explained variance of PCA was 88.1% which was mainly contributed by time constant of creep (TCC), %recoverability (RCY) and delta compliance (J-Jr). This suggests that SeP, RCY and TCC can be good candidates for a combined index of viscoelastic properties of gluten. J-Jr and TCC of gluten were highly correlated and were the main contributors of the first principal component explaining 64% of the variance. TCC and RCY appeared to be the main contributors in the second principal component.

Bi-plot of PCA depicted the different gluten samples according to temperature and viscoelastic parameters. The gluten at 25 and 35°C were grouped and mainly correlated with RCY; while, the gluten samples at 45 and 55°C were strongly associated to SeP. Stephens separated from the hard red winter wheat and was highly correlated to TCR when exposed to 25 and 35°C. In contrast, when Stephens was subjected to 45 and 55°C, it was highly correlated to TCC and J-Jr. Creep and recovery may effectively separate the change of viscoelastic properties as affected by temperature. Thus, it could be a potential tool for quantitative evaluation of processing quality performance of flour samples.

**Keywords:** Temperature, creep and recovery compliance, rheological properties, wheat gluten, viscoelasticity

#### 1. Introduction

Gluten protein is an important component of dough associated with bread quality (Attenburrow et al., 1990) since wheat quality is correlated to the strength of protein interactions, such as protein-protein and protein-starch interactions (Kim et al., 2004). In bread manufacturing process, heat is involved during the processing with temperature ranging from 30 to 260°C (Cuq et al., 2000). During baking, the physicochemical gluten properties were weakened (Kolpakova et al., 2007). The temperature effects on the viscoelastic properties showed that high energy was required to destabilize the hydrogen and hydrophobic interactions (Feng et al., 2010). The temperature used during baking affects the chemical bonds of all the components (hydrophobic bonds, sulfhydryl and disulfide groups), thus heat dynamically changes the viscoelastic properties of the dough and gluten (Hayta and Alpaslan, 2001). Hydrophobic interactions are formed by nonpolar side chains of amino acids and in general all proteins contain about 30 to 50% nonpolar amino acids (Scheraga et al., 1962). Scheraga et al. (1962) explained that the hydrophobic interaction increased as the temperature increased up to about 60°C and affected the stabilization of protein structure. Besides the hydrophobic interactions, covalent disulfide bonds and non-covalent hydrogen bonds are predominant bonds that destabilize the gluten protein conformation during heating (Tatham and Shewry, 1985). When exposed to temperature above  $45^{\circ}$ C, the interaction between glutenins and gliadins are weakened by decreasing  $\beta$ -sheet,  $\alpha$ -helix and hydrogen bonds (Yada, 2004). When heating the gluten from 30 to 90°C, a number of irreversible crosslinks were formed at 50°C which affected mainly the glutenin structure (Schofield, Bottomley et al., 1983).

Gliadins and glutenins, two main gluten components, are responsible for viscoelastic properties of gluten (Apichartsrangkoon, 2002). It is widely accepted that the elastic properties of gluten are mainly provided by glutenins; whilst, the viscous properties of gluten are chiefly exhibited by gliadins (Xu et al., 2007). A creep and recovery test was introduced by Bloksma (1962) applying a constant shear stress and shear strain and measuring creep and recovery compliance as a function of time (Abang Zaidel et al., 2008).One of the predominant factors of the viscoelasticity of gluten is temperature which can be analyzed by using creep recovery, stress relaxation or dynamic oscillatory measurements (Hayta and Schofield, 2005; Mirsaeedghazi, Emam-Djomeh et al., 2008; Schofield, Bottomley et al., 1983).

Dynamic oscillatory test at 0.01 to 10 Hz (frequency) revealed that heating gluten at temperature up to 90°C for 6 hours caused higher increase of G' and G'' compared to unheated and heated (30min) gluten samples (Apichartsrangkoon, 2002). It has been reported that when heating gluten from 25 to 90°C for 20 min, a decrease in free SH-groups, surface hydrophobicity and extractability of gluten was found (Stathopoulos et al., 2008). These authors also reported a decrease of tan δ (ratio of G' / G'') and a large reduction at 60°C by using a temperature sweep test (Attenburrow, Barnes et al., 1990). Creep measurement using cone and plate geometry by stressing at 50 Pa has shown that the elastic component (G') of gluten was lower in heating at 30 and 50°C compared to 70 and 90°C (Hayta and Schofield, 2005). Heating gluten beyond 90°C causes an increasing in G' and decreasing in G'' (Attenburrow, Barnes et al., 1990; Hayta and Alpaslan, 2001; Hayta and Schofield, 2004). The possible explanation was the formation of a highly crosslinked gluten structure and induction of the molecule mobility at temperature higher than 90°C (Attenburrow, Barnes et al., 1990). The comparison between good (Hereward) and poor (Riband) breadmaking quality wheat showed that Hereward had less SDS extractability and more SH-SS content than Riband cultivar after heated up to 70°C for 15 min (Hayta and Schofield, 2005). In the report of Hayta and Schofield (2005), frequency sweep test with gluten heated between 30 to 50°C revealed a decrease of elastic modulus. After heating gluten between 70 to 90°C, they found an increase of compliance in creep test (Hayta and Schofield, 2005). These authors compared the good (Hereward) and poor (Riband) wheat cultivars in creep test by increasing the temperature from 30 to 90°C and both cultivars revealed similar result in creep compliance (Hayta and Schofield, 2005).

Schofield et al. (1983) reported on after exposing winter wheat gluten to heat between 55 and 75°C, gluten was denatured and decreased its baking performance. The same study showed that gluten extractability of sulphydryl groups in SDS buffer was decreased. However, there is limited information on the effect of temperature on the viscoelastic properties of gluten from flours of different protein contents using a creep and recovery test. Therefore, the objective of this study was to evaluate differentiation of the commercial hard red winter wheat flour properties using a creep-recovery test of gluten at temperatures ranging from 25 to 55°C.

#### 2. Materials and method

#### 2.1 Materials

Six commercial hard red winter wheat flours and one commercial soft red winter wheat (Stephens) flour sample were studied.

#### 2.2 Experimental

#### 2.2.1 Gluten preparation

Wet glutens were isolated by washing 10 g of flour with 2% NaCl solution (w/v) for 10 minutes from using a Glutomatic 2200 instrument (Perten Instruments, Sweden). The wet glutens were analyzed in two replicates with coefficient of variation less than 10% within the replicates.

#### 2.2.2 Creep and recovery test of gluten

Creep and recovery tests were conducted following the method described by Yeap (2008). In brief, the gluten obtained from the Glutomatic was immediately rolled into a ball-shape and relaxed (2.5 kg top plate and 2.5 mm space between the plates) for an hour at room temperature. A 25 mm disc gluten sample was obtained by using a metal die and transferred to the lower plate of a constant stress rheometer (AR1000, TA Instruments, New Castle, DE) and re-trimmed to fit in the 25 mm parallel-plate lowered to the 2.5 mm gap. To prevent moisture loss during the test, mineral oil was applied to the edge of the gluten. The gluten sample was covered with a chamber and kept surrounded by a saturated water atmosphere. During this test a constant stress (100 Pa) was used for 100 s which deformed the gluten (viscous response) followed by a release of the stress to measure its elastic recovery. The temperature was controlled at 25, 35, 45, and 55°C in each test with a peltier plate. The analysis was performed in two replicates with coefficient of variation between replicates less than 10%. Five responses were obtained: Separation time (SeP), Delta compliance (J-Jr), % Recoverability (RCY), Time Constant Creep (TCC), and Time Constant of Recovery (TCR). Separation time (SeP) is identified when creep compliance and recovery curves diverged using semi-logarithmic plots. J-Jr was calculated by subtracting the recovery compliance from the creep compliance at 100 seconds. RCY was obtained by using the equation

$$\left(\frac{Creep \ compliance - Recovery \ compliance}{Recovery \ compliance} * 100 \right)$$
 at 100 seconds. TCC and TCR are time (s)

of the creep and recovery compliances at 63.2 percent of its final (asymptotic) value. J-Jr and TCC reflect the viscous properties of gluten. SeP, RCY, and TCR are parameters that reflect the elastic behavior of gluten. The less viscosity of gluten is expressed, the stronger gluten will be. On the other hand, the more elastic property of gluten tends to be strong gluten (Yeap, 2008).

#### 2.2.3 Statistical analysis

Analysis of variance was performed using the GLIMMIX procedure (Statistical Analysis System, SAS Institute Inc., Cary, NC). The effect of temperature and flour types (protein content) on the viscoelastic variables and the interactions were evaluated. Principal Component Analysis (PCA) using Canoco for Windows 4.5 (Biometris, Plant Research International, Wageningen, the Netherlands), factor analysis using the FACTOR procedure (Statistical Analysis System, SAS Institute Inc., Cary, NC) and Pearson correlation using the CORR procedure were also conducted.

#### 3. Results and discussions

The list of abbreviations and definitions studied is presented in Appendix 1 and Table 1. Protein, moisture, and ash content of the flour samples are reported in Table 1.

#### 3.1 Viscoelastic properties

#### Separation time (SeP)

The time at which the recovery curve separates from the creep curve is defined as SeP. A representation of the gluten viscoelastic properties using a creep and recovery procedure at 25, 35, 45 and 55°C is illustrated in Fig. 1. Significant temperature effects were observed in viscoelastic variables of all gluten samples except for A3 (P = 0.1329, Table 2). The effect of each temperature on viscoelastic properties of gluten also showed in table 3. It was observed that temperature at 25°C was not affected the viscoelastic variables of gluten (P = 0.4013, Table 3). There was a significant interaction between temperature and flour types on SeP (P < 0.05). Figure 2 shows interaction graphs between flour types and temperatures on each viscoelastic variable. The SeP values at 45°C in almost all gluten samples significantly increased except for A3 and B2 as observed in Fig. 2a (P = 0.72 and 0.99, respectively, Table 4a, Appendix 1 and Table 2). After 45°C, the SeP values in all of glutens decreased to 55°C (P < 0.05, Table 4a, Fig.2a, Appendix 1 and Table 2). SeP illustrates the gluten chain entanglements which is directly related to molecular weight (Nielsen and Landel, 1994). These authors explained that the higher molecular weight, the higher SeP will be at high temperature (Nielsen and Landel, 1994). The longer SeP, i.e., at 45°C creep and recovery compliance curves stayed superimposed, the more chain entanglement has occurred compared to 25°C (Fig. 1). For

example, A1 had the highest SeP from 35°C to 45°C (a change of 140.9%, Appendix 1 and Table 2) suggesting higher chain entanglements formed compared to the rest of the samples. The increase in SeP (elasticity) when exposed to 45 and 55°C compared to room temperature (a change of 302.6%, Appendix 1 and Table 2) could be explained in part by the unfolding of gluten structure and formation of entanglements with other gluten molecules (Lavelli et al., 1996).

#### Delta compliance (J-Jr)

J-Jr is the difference between the creep and recovery compliance at 100 s (Fig. 1). J-Jr values were significantly affected by temperatures and flour samples (P < 0.01, Table 2 and 3). There was also a significant interaction between temperature and flour samples (P < 0.01). High values of J-Jr mean low elasticity and high viscosity behavior. Stephens and A3 had more viscous and less elastic behavior compared to the rest of samples (Appendix 1 and Table 2). At temperature from 35 to 45°C, J-Jr of A2, A3 and Stephens significantly increased by 60.9, 76.1, and 42.9%, respectively (Table 4a, Appendix 1 and Table 2). It was agreed that heating above 50°C induced crosslinks in gluten resulting in an increase in G' (Schofield, Bottomley et al., 1983).

#### Recoverability (RCY)

Flour types significantly affected RCY of gluten samples (P < 0.0001, Table 5a) while there was no significant interaction between temperature and protein content. At 25 °C, the RCY was not significantly affected; however, it significantly decreased from 35 to 55°C (P < 0.05) (Table 5b, Appendix 1 and Table 2). These observations agree with literature reports of the decrease in the elastic behavior of gluten when gluten was heated at 30 to 50°C and tested by using creep test (Hayta and Schofield, 2005). However, Song Y. (2007) reported that temperature at 20 to 40°C does not alter the mechanical behavior particularly in irreversible changes in disulfide bonds of gluten. The RCY (elastic recovery of gluten) decreased with temperature and appeared to have a nonlinear response (Fig. 2c). A3 and Stephens showed a low RCY and a reduction of 20% and 23.9% elastic recovery, respectively from 25 to 55°C (Appendix 1 and Table 2).

#### *Time constant of creep (TCC)*

TCC is described as the time that it takes the gluten sample to achieve 62.3% of its equilibrium and is related to viscosity. Shorter TCC represents faster equilibrium (higher viscosity) compared to longer TCC. TCC of Stephens and A1 were not significantly affected by temperature (P = 0.7509 and 0.3963, respectively and Table 2) TCC was significantly affected by flour sample (P < 0.05) (Table 3). There was an interaction between the temperature and flour samples on TCR (P < 0.05). Gluten TCC increased at temperature of 55 °C (Appendix 1 and Table 2). This suggested high values of TCC means reach equilibrium longer time. However, all gluten samples were not significantly different when subjected to temperature at 55°C except for A2, A3 and B3 (Table 4b). TCC of all gluten samples tended to increase after exposed to 45°C (Appendix 1 and Table 2) while the tendency of the TCR decreased when the temperature increased (Appendix 1 and Table 2).

#### Time constant of recovery (TCR)

Shorter TCR means faster equilibrium (high elasticity) compared to longer equilibrium. Significant effects of temperature and flour types on TCR and significant interactions between temperature and flour protein content were observed in Table 2 and 3 (P < 0.05). The results suggested that gluten at 55°C reached equilibrium at longer times compared to temperature at 25°C (Fig. 2e). TCR of all gluten mainly decreased when exposed to 55°C (Appendix 1 and Table 2); however, only A3 was significantly different at  $55^{\circ}$ C (Table 4b). The study on the extraction of 5+10, 17+18 and triple null of glutenin subunit in SDS was reported that the glutenin amounts from all types of subunit at 70°C were lower than at 20°C and the glutenin contents were different in each subunit (Lefebvre et al., 2000). They concluded that the effect of temperature on gluten depended on the subunit composition (Lefebvre, Popineau et al., 2000). Stephens showed the greatest change with 56.9% decrease in TCR from 25 to 55°C (Appendix 1 and Table 2). The results showed that Stephens was different from others in TCC and TCR, SeP, J-Jr, and RCY (Fig. 2). Thus, the viscoelastic properties from creep-recovery test can differentiate gluten behavior from hard and soft red winter wheat.

# 3.2 Principal component analysis (PCA), factor analysis, and Pearson correlations

The five variables explaining the viscoelasticity of gluten samples were subjected to PCA, using the PRINCOMP procedure by SAS. The contribution of each variable to the explained variance of the two principal components is reported in Table 7 and Figure 3. The advantage of PCA is the visualization of the relationship between

parameters and samples (Dobraszczyk and Salmanowicz, 2008). The bi-plot of PCA illustrates the correlation of parameters. The parameters that are close to each other are closely correlated to each other; whereas, the parameters that opposite to each other are negatively associated. Besides, the parameters that are  $90^{\circ}$  to each other are independent. The most important contributors for explaining the variation are the parameters with the highest magnitude and closest to PC1. Fig. 3, PCA results indicated 88.1% of the total variation accounted for the first two principal components (Table 7). The explained variances of first and second principal components were 64.0% and 24.1%, respectively (Table 7 and Fig. 3). The first principal component (PC1) was highly correlated with J-Jr and TCC which are variables associated with viscosity properties of gluten (Fig. 3 and Table7). The association of J-Jr and TCC was supported by the Pearson correlation (r =0.91, P < 0.01, Table 6). The second principal component (PC2) was mainly associated with SeP and TCR (Table 7 and Fig. 3).A distant third major contributor to the first principal component was gluten % Recoverability whose contribution to variance to PC1 was 66% (Table 7)

Two groups of samples were separated in the PCA bi-plot (Fig. 3). First, the hard red winter flour samples were separated from the soft red winter sample (grouped on the right hand side of the plot) (Fig. 3). This suggests that their viscoelastic properties are quite different from the hard wheat samples. Second, each group of samples (hard and soft red winter) was separated into two major groups according to the temperature at which the analysis was performed (Fig. 3). Samples analyzed at 25 and 35°C were associated mainly with RCY and slightly related to TCR (Fig. 3). In contrast, the samples analyzed at 45 and 55°C were mainly associated with SeP, TCC and J-Jr. However, B2

subjected at 35°C was closely related to the sample analyzed at 45 and 55°C (Fig. 3). This suggests that B2 exposed to 35°C had properties more similar to the samples subjected to 45 and 55°C. Stephens was highly associated with TCR when analyzed at 25 and 35°C and was correlated with TCC and J-Jr when analyzed at 45 and 55°C (Fig. 3). These results suggest that at 45 and 55°C, Stephens appeared to be independent or weakly associated with SeP. SeP was negatively correlated to TCR (Fig. 3). In organic polymers, the separation time is associated with the entanglement of the polymer molecules (Heddleson et al., 1994). The Pearson correlation also showed that there was a highly (negatively) significant correlation between RCY and TCC (r = -0.87, P < 0.01 and Table 6). A negative correlation of RCY and J-Jr was also observed (r = - 0.81, P < 0.01 Table 6). Thus, as the value of J-Jr increases (more flowable gluten), the RCY decreases (lower gluten stiffness).

The factor analysis using the principal component definitions as factors supported the PCA results in that J-Jr and TCC were strongly correlated to the first principal component (Table 8). The final communality estimates for J-Jr and TCC were 0.92 and 0.97, respectively, accounting for 42.7% of the total communality (Table 8).

#### 4. Conclusion

This study of gluten from seven commercial flour samples revealed that overall, significant changes in gluten rheological properties occurred at 45°C. At 45-55°C the glutens become more flowable (increased viscosity) and less stiff (decreased elasticity). The time constant of creep and recovery assisted in the differentiation of gluten behavior. Gluten viscosity reached the equilibrium slower at 55°C than 25°C; while, gluten elastic

behavior reached the equilibrium faster at 55°C than 25°C. Two distinct groups were easily separated according to their association with the changes of their viscoelastic properties at 25 to 55°C. At 25 and 35°C, the gluten was distinctively elastic while at 45 and 35°C, the glutens were highly associated with separation time (entanglements) and highly associated with their viscous component. Gluten from soft flour was easily separated from the hard flour and their associations with the viscous and elastic component parameters were different.

Wheat type	Flour	Protein (%)	Moisture (%)	Ash (%)
Hard wheat	A1	$7.95\pm0.05$	$11.69 \pm 0.02$	$0.29 \pm 0.01$
	A2	$11.19\pm0.07$	$10.51\pm0.03$	$0.38\pm0.01$
	A3	$13.68\pm0.02$	$10.14\pm0.02$	$0.41\pm0.00$
	<b>B1</b>	$10.40\pm0.10$	$12.54\pm0.02$	$0.47\pm0.00$
	<b>B2</b>	$10.59\pm0.07$	$12.57\pm0.00$	$0.48\pm0.01$
	<b>B3</b>	$11.38\pm0.01$	$12.98\pm0.04$	$0.58\pm0.01$
Soft wheat	Stephens	$11.40 \pm 0.0$	$11.77\pm0.00$	$0.65\pm0.00$

Table 1. Proximate analysis of flours (means  $\pm$  SD, n=2).

SD = standard deviation.
					Samples	8		
		A1	A2	A3	<b>B1</b>	B2	<b>B3</b>	Stephens
Variables	Num DF <sup>a</sup>	3	3	3	3	3	3	3
, un un numero	Den DF <sup>b</sup>	24	24	24	24	24	24	4
SeP	F Value	8.96	7.49	2.06	6.91	3.48	5.41	1055.47
	<b>Pr&gt; F</b>	0.0004	0.001	0.1329	0.0016	0.0315	0.0055	< .0001
J-Jr	F Value	6.40	20.48	94.63	5.98	6.10	9.71	46.02
	Pr>F	0.0024	< .0001	<.0001	0.0034	0.0031	0.0002	0.0015
тсс	F Value	1.03	10.12	27.79	4.16	8.33	5.32	0.42
	Pr>F	0.3963	0.0002	<.0001	0.0166	0.0006	0.0059	0.7509
TCR	F Value	30.53	21.50	21.56	14.60	17.48	14.93	17.20
	<b>Pr&gt; F</b>	< .0001	< .0001	<.0001	<.0001	< .0001	< .0001	0.0095

Table 2. Analysis of variance for viscoelastic properties of glutens treated with temperature

<sup>a</sup> Numerator Degree of Freedom.

<sup>b</sup> Denominator Degree of Freedom.

			Valu	es	
Variables	Temperature(°C)	Num DF <sup>a</sup>	Den DF <sup>b</sup>	F Value	Pr>F
SeP	25	6	24	1.08	0.4013
	35	6	24	6.11	0.0005
	45	6	24	30.13	< 0.0001
	55	6	24	13.31	< 0.0001
J-Jr	25	6	19.49	28.93	< 0.0001
	35	6	19.49	31.46	< 0.0001
	45	6	19.49	91.32	< 0.0001
	55	6	19.49	144.25	< 0.0001
TCC	25	6	19.43	5.32	0.0022
	35	6	19.43	6.04	0.0011
	45	6	19.43	12.21	< 0.0001
	55	6	19.43	14.55	< 0.0001
TCR	25	6	19.52	23.19	< 0.0001
	35	6	19.52	16.91	< 0.0001
	45	6	19.52	8.86	< 0.0001
	55	6	19.52	5.31	0.0021

Table 3. Analysis of variance for viscoelastic properties of glutens in each temperature

<sup>a</sup> Numerator Degree of Freedom.

<sup>b</sup> Denominator Degree of Freedom.

				25 ai	nd 35 °C					35 a	nd 45 °C					45	and 55 °	С	
		Esti-			t			Esti-			t			Esti-			t		
Variables	Samples	mate	SE <sup>a</sup>	DF <sup>b</sup>	Value	Pr>ltl	Adj P	mate	SE <sup>a</sup>	DF <sup>b</sup>	Value	Pr>ltl	Adj P	mate	SE <sup>a</sup>	DF <sup>b</sup>	Value	Pr>ltl	Adj P
	A1	-5.56	2.681	24	-2.07	0.0491	0.1905	-8.19	2.681	24	-3.06	0.0054	0.0261	8.42	2.681	24	3.14	0.0044	0.0215
	A2	2.08	2.681	24	0.78	0.4451	0.8643	-11.58	2.681	24	-4.32	0.0002	0.0013	4.56	2.681	24	1.7	0.102	0.3455
	A3	-3.17	2.681	24	-1.18	0.2486	0.6433	-2.82	2.681	24	-1.05	0.304	0.7221	0.56	2.681	24	0.21	0.8354	0.9966
	B1	0.06	2.681	24	0.02	0.9812	1.0000	-10.59	2.681	24	-3.95	0.0006	0.0031	7.53	2.681	24	2.81	0.0097	0.045
	B2	-5.79	2.681	24	-2.16	0.0412	0.164	-0.77	2.681	24	-0.29	0.7776	0.9917	-1.54	2.681	24	-0.57	0.5715	0.939
	B3	-0.33	2.681	24	-0.12	0.9022	0.9993	-9.17	2.681	24	-3.42	0.0022	0.0113	6.70	2.681	24	2.5	0.0198	0.0858
SeP	Stephens	0.00	0.018	4	0.04	0.9671	1.0000	-0.83	0.018	4	-45.34	< 0.0001	< 0.0001	0.86	0.018	4	47.1	< 0.0001	< 0.0001
	A1	-0.18	0.09517	24	-1.87	0.0738	0.2773	-0.03	0.09517	24	-0.29	0.7719	0.9909	-0.21	0.09517	24	-2.2	0.0375	0.1625
	A2	-0.03	0.09517	24	-0.31	0.7565	0.9889	-0.28	0.09517	24	-2.96	0.0068	0.0395	-0.34	0.09517	24	-3.6	0.014	0.0108
	A3	0.13	0.09517	24	1.35	0.1889	0.5442	-0.90	0.09517	24	-9.51	< 0.0001	< 0.0001	-0.48	0.09517	24	-5.01	< 0.0001	0.0006
	B1	0.03	0.09517	24	0.32	0.7506	0.9881	-0.11	0.09517	24	-1.11	0.2782	0.6887	-0.26	0.09517	24	-2.7	0.0126	0.0664
	B2	-0.09	0.09517	24	-0.94	0.3543	0.7816	-0.19	0.09517	24	-1.96	0.062	0.2423	-0.09	0.09517	24	-0.9	0.3757	0.8036
	B3	-0.05	0.09517	24	-0.51	0.6135	0.9552	-0.13	0.09517	24	-1.33	0.197	0.5591	-0.29	0.09517	24	-3.07	0.0053	0.0319
J-Jr	Stephens	-0.50	0.3170	4	-1.58	0.1885	0.4135	-1.40	0.3170	4	-4.42	0.0115	0.0019	-1.48	0.3170	4	-4.67	0.0095	0.0012

Table 4a. Least Squares Means of temperature x flour sample for the viscoelastic properties of gluten using a creep and recovery test

<sup>a</sup> Standard Error. <sup>b</sup> Degree of freedom.

				25 ai	nd 35 °C					35 a	and 45 °C					45	and 55 °C	5	
		Esti-			t			Esti-			t			Esti-			t		
Variable	Samples	mate	SE <sup>a</sup>	DF <sup>b</sup>	Value	Pr>ltl	Adj P	mate	SE <sup>a</sup>	DF <sup>b</sup>	Value	Pr>ltl	Adj P	mate	SE <sup>a</sup>	DF <sup>b</sup>	Value	Pr>ltl	Adj P
	A1	-0.89	0.7825	24	-1.14	0.2665	0.6723	0.66	0.7825	24	0.84	0.4090	0.8346	-0.98	0.7825	24	-1.25	0.2246	0.6073
	A2	-0.59	0.7825	24	-0.76	0.4572	0.8730	-0.07	0.7825	24	-0.09	0.9285	0.9997	-3.22	0.7825	24	-4.12	0.0004	0.0036
	A3	-0.05	0.7825	24	-0.07	0.9447	0.9999	-3.19	0.7825	24	-4.08	0.0004	0.0039	-2.83	0.7825	24	-3.62	0.0014	0.0103
	<b>B</b> 1	0.19	0.7825	24	0.24	0.8138	0.9951	-0.28	0.7825	24	-0.35	0.7264	0.9843	-2.12	0.7825	24	-2.71	0.0122	0.0645
	B2	-0.02	0.7825	24	-0.03	0.9793	1.0000	-2.16	0.7825	24	-2.76	0.0110	0.0591	-1.01	0.7825	24	-1.29	0.2098	0.5821
	<b>B3</b>	-0.23	0.7825	24	-0.29	0.7748	0.9912	-0.15	0.7825	24	-0.20	0.8469	0.9973	-2.36	0.7825	24	-3.01	0.0060	0.0359
тсс	Stephens	1.27	3.1293	4	0.41	0.7058	0.9767	-1.72	3.1293	4	-0.55	0.6119	0.9454	-1.74	3.1293	4	-0.55	0.6088	0.9440
	A1	0.61	0.1993	24	3.04	0.0056	0.0336	0.63	0.1993	24	3.15	0.0043	0.0269	0.57	0.1993	24	2.84	0.0091	0.0506
	A2	-0.02	0.1993	24	-0.10	0.9188	0.9996	1.02	0.1993	24	5.13	< 0.0001	0.0004	0.22	0.1993	24	1.09	0.2865	0.7001
	A3	0.57	0.1993	24	2.84	0.0091	0.0503	0.40	0.1993	24	1.99	0.0577	0.2291	0.59	0.1993	24	2.96	0.0068	0.0397
	<b>B1</b>	0.01	0.1993	24	0.07	0.9426	0.9999	0.73	0.1993	24	3.65	0.0013	0.0096	0.33	0.1993	24	1.67	0.1069	0.3663
	B2	0.63	0.1993	24	3.16	0.0042	0.0265	0.28	0.1993	24	1.40	0.1749	0.5173	0.50	0.1993	24	2.53	0.0183	0.0905
	<b>B3</b>	0.35	0.1993	24	1.76	0.0909	0.3246	0.61	0.1993	24	3.07	0.0053	0.0318	0.22	0.1993	24	1.10	0.2831	0.6955
TCR	Stephens	0.95	0.6188	4	1.53	0.1997	0.4399	2.08	0.6188	4	3.37	0.0281	0.0173	0.89	0.6188	4	1.44	0.2229	0.4923

Table 4b. Least Squares Means of temperature x flour sample for the viscoelastic properties of gluten using a creep and recovery test

<sup>a</sup> Standard Error. <sup>b</sup> Degree of freedom.

# Table 5. Analysis of variance for %Recoverability (RCY) of glutens treated with temperature

RCY	Temperature (°C)	Estimate	Error	$\mathbf{DF}^{\mathbf{a}}$	t Value	Pr <ltl< th=""></ltl<>
	25	81.74	0.5928	27.3	137.88	< 0.0001
	35	80.08	0.5928	27.3	135.09	< 0.0001
	45	76.40	0.5928	27.3	128.88	< 0.0001
	55	69.09	0.5928	27.3	116.55	< 0.0001

a) Least Squares Means of temperature

b) Differences of temperature Least Squares Means Adjustment for Multiple Comparisons: Tukey

RCY	Temperature (°C)	Estimate	Error	$\mathbf{DF}^{\mathrm{a}}$	t Value	Pr <ltl< th=""></ltl<>
	25 and 35	1.65	0.8384	27.3	1.97	0.2224
	<b>35</b> and <b>45</b>	3.68	0.8384	27.3	4.39	0.0008
	45 and 55	7.31	0.8384	27.3	8.72	< 0.0001

<sup>a</sup> Degree of freedom.

	SeP		J-Jr		RCY	TCC	TCR
SeP		1					
J-Jr	-0.35**			1			
RCY			-0.81**		1		
TCC	-0.31*		0.91**		-0.87**	1	
TCR	-0.58**		0.51**			0.58**	1

Table 6. Pearson's correlation coefficients of the viscoelastic properties of gluten

\*Correlation is significant at  $\alpha = 0.05$  level.

\*\*Correlation is significant at  $\alpha = 0.01$  level.

Variables	PC (%)	PC1	PC2	1+2
	Axes	64.0	24.1	88.1
	SeP	27.0	51.0	78.0
	J-Jr	88.8	3.06	91.8
	RCY	65.9	27.4	93.3
	TCC	93.0	3.58	96.5
	TCR	45.0	35.7	80.7

 Table 7. Explained variance (%) in Principal Component Analysis (PCA) of viscoelastic

 properties of gluten

Variables	Factor 1	Factor 2	<b>Final Communality</b>
	3.19	1.23	4.41
SeP	-0.51	-0.73	0.79
J-Jr	0.94	-0.17	0.92
RCY	-0.81	0.52	0.93
TCC	0.96	-0.19	0.97
TCR	0.67	0.60	0.81

Table 8. Factor analysis of viscoelastic properties of gluten



Fig. 1. An example of creep and recovery behavior of wheat gluten (sample A2) at difference temperatures. The compliance of creep and recovery at 100 s represent the viscous and elastic component of gluten, respectively. Delta compliance  $(J-J_r)$  is the difference between compliance of creep and recovery at 100 s. The higher J-Jr, the more viscous the gluten. The time at which the creep and recovery components split is called separation time (SeP) and it represents the elastic component.



Fig.2. Viscoelastic properties variables of seven commercial wheat glutens as a function of temperature. Definitions of viscoelastic variables explained in Appendix 1 and Table 1.



Fig. 3. Loading plot of first two principal components based on viscoelastic properties of seven commercial wheat glutens. Definitions of viscoelastic variables explained in Table 1.

<sup>a</sup> Soft wheat.

# CHAPTER IV

# VARIATION IN GLUTEN VISCOELASTICITY, DOUGH EXTENSIBILITY, FARINOGRAPH AND BAKING PROPERTIES AMONG COMMERCIAL HARD RED WINTER WHEAT

# Abstract

Some parameters describing the quality of wheat flours can be estimated by empirical and fundamental rheological measurements. Baking performance is one of the most important tests in flour quality and a good approximation of its prediction using rheological properties of gluten and dough has been explored. Six commercial hard red winter wheat flours were analyzed. Gluten viscoelasticity, dough extensibility, dough mixing properties, baking properties, and flour protein were analyzed to evaluate their discriminatory ability of explaining the variance using principal component analysis. Creep and recovery tests were conducted using shear stresses of 40 and 100 Pa. When all the variables were included 79.1% of the variance was explained. The difference of creep and recovery compliance (J-Jr) and maximum resistance to extension (Rmax) were the largest contributors to the explained variance. Flour protein (FP), loaf volume and height were independent of the viscoelastic properties. An improvement of 5.7% of the total explained variance was obtained when using FP, LV, extensibility and viscoelastic properties at 100 Pa (83.8% total explained variance). This suggests that improved explained variance can be obtained using a creep-recovery test, extension test,

LV and FP. A similar approach needs to be validated with larger number of samples.

Keywords: Rheological properties, correlation, gluten and dough properties,

creep-recovery test, principal component analysis.

#### 1. Introduction

Gluten protein is a key component in dough providing its unique viscoelastic characteristics which are a result of the interactions of disulfide, hydrophobic, and hydrogen bonds (Wieser 2007). Studies of gluten protein and dough rheological properties suggested that dough strength and glutenin molecular sizes were highly correlated (Branlard 1985). Gupta et al. (1993) studied the effect of deficient high molecular weight -glutenin subunits (HMW-GS) and low molecular weight-glutenin subunits (LMW-GS) on dough and gluten properties. They found that the absence of Glu-1 or Glu-3 HMW-GS affected to the amount of extractable and unextractable of protein (Gupta et al., 1993).

Rheological measurements, from both empirical and fundamental methods have been widely applied to discriminate breadmaking performance in order to predict final product quality (Dobraszczyk and Morgenstern, 2003). Farinograph and extensibility tests are empirical measurements relatively simple to operate in typical laboratories and do not require highly skilled labor (Dobraszczyk and Morgenstern, 2003). However, empirical measurements are insufficient to describe fundamental properties and cannot be extrapolated to rheological parameters, e.g. stress, strain, apparent viscosity, unlike fundamental rheological test (Dobraszczyk and Morgenstern, 2003).

Traditionally, empirical measurements have been used to assess the physical properties of wheat dough on mixing properties measured by a farinograph and extensibility behavior investigated by an extensigraph. However, the obtained parameters from empirical tests are inadequate to interpret baking quality (Wang and Sun, 2002). A study on the prediction of bread quality using farinograph and extensograph concluded

that the baking quality has low relationship with the physical dough tests (Oliver and Allen, 1992). Kieffer et al. (1998) introduced an extensibility test by using smaller amount of flour sample compared to extensigraph. The examination of micro-extensibility dough test demonstrated a high correlation with bread performance in terms of loaf volume (Kieffer et al., 1998) and baking volume (Zaidel and Yusof, 2010).

Creep and recovery test has been used for studying rheological properties of wheat dough since 1930 before it was applied for measuring gluten protein (Bloksma, 1962). The gluten creep and recovery measurement with applied shear stress of 250 Pa showed a high correlation between bread volume and maximum recovery strain combined with sedimentation value, and water absorption parameter (Bockstaele et al., 2008). From another study, Wang and Sun (2002) investigated flour-water doughs with a creep and recovery technique by using dynamic mechanical analyzer (DMA). They reported a high relationship of bread loaf volume and maximum recovery strain with dough at 54% water absorption (Wang and Sun, 2002). However, more studies are needed regarding the gluten creep and recovery measurements and their correlation to breadmaking quality. The purpose of this study was to discriminate the commercial hard red winter wheat flour properties by using the gluten creep-recovery test, farinograph, baking, and dough extensibility test.

# 2. Materials and Methods

# 2.1 Materials

Six commercial hard red winter wheat flours with protein content ranging from 7.95 to13.68% were studied. The samples were identified as A1 through A3 and B1 through B3 (Table 1).

# 2.2 Experimental

#### 2.2.1 Gluten preparation

Gluten was obtained from the flour samples using a Glutomatic 2200 (Perten Instuments, Sweden). Half (0.5) ml of 2% salt solution was mixed for 60s before the isolation of gluten from 10 g flour samples with 2% NaCl solution (w/v) for 6 min.

# 2.2.2 Dough preparation

Dough was prepared following the method of Kieffer et al. (1998). Briefly, flour samples were mixed in a Farinograph to obtain a dough consistency of 600 Brabender Unit (BU) with 2% salt solution (w/v). At the consistency peak (600 BU) of the curve, the dough was retrieved, gently shaped into a roll, and transferred to the Teflon form of the Kieffer rig provided by Texture Technologies (TA.XTPlus, Texture Technologies Corp., Scarsdale, NY/Stable Micro Systems, Godalming, Surrey, UK). Mineral oil was added to avoid excessive sticking. The dough was clamped and relaxed for 40 min in a water saturated environment at room temperature.

# 2.2.3 Creep and recovery test of gluten

The creep and recovery method was followed according to Zhao (2010) and Yeap (2008). The procedure for gluten creep and recovery test was followed as described in materials and methods section of Chapter III. The test was performed at two constant shear stresses of 40 and 100 Pa for 100s. The analysis was performed in two replicates. The coefficient of variation between replicates was less than 10%.

# 2.2.4 Extensibility of dough

After the dough was rested in the Teflon form for 40 minutes inside a plastic bag with wet tissue, it was unclamped and the mold was gently opened. The dough strips were placed on the Texture Analyzer plate. A Kieffer Dough Extensibility rig was used in the Texture Analyzer TA-XT2 (TA.XTPlus, Texture Technologies Corp., Scarsdale, NY/Stable Micro Systems, Godalming, Surrey, UK). A trigger force of 1 g and 4.0 mm/s test speed were used. The dough strength (maximum resistance to extension, Rmax), dough extensibility (extensibility at maximum resistance, Emax), and work required to extend the dough to Rmax (Area) were obtained from the tracing of the curves to evaluate gluten quality. The analysis was performed on two samples and 10 subsamples with coefficient of variation between subsamples less than 10%.

# 2.2.5 Farinograph parameters

Farinograph tests were performed according to Ambardekar (2009) and approved method 54-21 (AACC International 2000). Flour samples were mixed at 63 rpm and 30°C in a Farinograph-E equipped with 10 g bowl (C.W. Brabender Instruments, Hackensack, NJ). For mixing properties of flours were obtained: 1) development time

(DT); 2) stability time (ST); 3) breakdown time (BT); and 4) water absorption adjusted to 14% protein content (WA).

#### 2.2.6 Baking test

Baking tests were performed following the methods described by Ambardekar (2009). Wheat flour samples (100 g) were baked using an optimized straight-dough procedure of approved method 10-10B (AACC International 2000). The dough was mixed in a 100-g mixer (Swanson-Working pin-type, National Mfg. Co. TMCO Inc, Lincoln, NE) and the optimum baking mixing times were obtained from various baking trials. Bread quality was identified by measuring five responses: dough proof height (PH), loaf height (LH), loaf volume (LV), oven spring (OSP), and specific volume (SV). The heights of dough proof (PH) and loaves (LH) were measured by using a digital proof height gauge (National Mfg. Co. TMCO Inc, Lincoln, NE). The loaf volume (LV) was obtained by rapeseed displacement after baked samples were removed from the oven and cooled for 10 min. The OSP was defined by subtracting proof heights from loaf height. The ratio of loaf volume to the loaf weight was obtained for specific volume (SV).

#### 2.2.7 Statistical analysis

The parameters were analyzed with Principal Component Analysis (PCA) and Pearson correlation (P < 0.01 and 0.05). Principal Component Analysis was performed using Canoco for Windows 4.5 software (Biometris, Plant Research International, Wageningen, the Netherlands). Pearson correlation was performed by the CORR procedure using Statistical Analysis System (SAS Institute Inc., Cary, NC).

#### 3. Results and discussion

The flour samples were characterized in term of protein, moisture, and ash (%), and reported in Table 1.

# 3.1 Principal component analysis

1) Loading plot of first two principal components based on viscoelastic, mixing, extensibility, and baking properties of six commercial wheat flours (Fig. 1 and Table 2)

The relationships of viscoelastic properties using constant shear stress at 40 and 100 Pa, dough extensibility and mixing properties, and baking parameters that determine the quality of flour samples were performed using principal component analysis (PCA). The bi-plot of PC1 and PC2 containing all samples and parameters is shown in Fig. 1 and Table 2. The first two principal components explained 79.1% of the total variance (Fig. 1 and Table 2). The first component (PC1) or axis 1 explained 40.9% of the total variance; while, the second component (PC2) or axis 2 explained 38.2% of the total variance (Fig. 1 and Table 2). J-Jr (40 Pa) and Emax were the two variables that individually contributed with the highest explained variance (86.7 and 86.4%, respectively) (Table 2) to the first principal component. This observation was supported by Pearson correlations with r = 0.78, P < 0.01 (Table 6). The PCA revealed a number of redundancies of the variables, i.e., vectors were too close on either principal component as well as vectors (variables) with small contribution to the explained variance. The second principal component (second axis), was positively correlated with dough water absorption and protein content (r = 0.95, P < 0.01, Fig. 1 and Table 6, 2). Emax and J-Jr at 40Pa, and flour protein and water absorption were independent (i.e., they are at about 90°) (PC1 and PC2) (Fig. 1 and Table 2), whereas dough strength (Rmax) and gluten elasticity (SeP) at 100 Pa were closely correlated (PC1) (Fig. 1 and Table 2). From the six samples analyzed, three were different and easily separated by PCA (A1, A3 and B1) while the other three were similar and appeared clustered at the center of the graph (A2, B2 and B3). B1 was highly related to PC1, A2 to PC2 and A3 was equally related to PC1 and PC2. A3 was also closely related to DT, BT, Area and ST. The results also describe the independence of viscoelastic properties with WA, FP and baking parameters (at right angles Fig. 1). The extension properties of Area and Rmax appeared to be related to baking properties FP, WA and viscoelastic properties (Fig. 1).

2) Loading plot of first two principal components based on viscoelastic using shear stress at 100 Pa, extensibility, and baking properties of six commercial wheat flours (Fig. 2 and Table 3)

The correlation between the viscoelastic properties, using creep and recovery test at 100 Pa shear stress, extensibility, LV and FP is illustrated in PCA (Fig. 2 and Table 3). The total explained variance of the two principal components was 83.8% (Fig. 2 and Table 3). The first principal component (53.3%) was mainly determined by J-Jr and Emax which contributed individually with 85.9% and 89.4% of the explained variance, respectively (Fig. 2 and Table 3). The second principal component (PC2) demonstrated a high correlation with LV and FP and explained 30.3% of the total variance (Fig. 2 and Table 3). The individual contributions of LV and FP were 97.4 and 77.7% of the explained variance (Table 3). The separation of the samples was similar to the PCA that included all the variables (Fig. 1) except that now B1 is closer to the center and to the cluster of A2, B2 and B3. However, there is an improvement of the explained variance

(5.9% improvement from 79.1 to 83.8% explained variance) (Table 2-3, Fig. 1-2). Overall, the same relationship of Fig.1 can be concluded from Fig. 2, i.e., the independence of LV and FP to the viscoelastic properties.

3) Loading plot of first two principal components of selected variables after discarding redundant and low contributors variables of Table 2 based on viscoelastic, mixing, extensibility and baking properties of six commercial wheat flours(Fig. 3 and Table 4)

The bi-plot of Fig. 1 (Table 2) showed a number of redundant variables and some variables contributing marginally to explaining the variance. Thus, in an effort to simplify the analysis these variables were removed and the results presented in Fig.3, Table 4. The total explained variance was 76.9% (Table 4 and Fig. 3) which is lower than the analysis containing all variables (79.1%, Fig. 1, Table 2). The first principal component (PC1) was mainly explained by TCC 100 Pa and J-Jr 40Pa contributing individually with 79.4% and 68.8% to the explained variance, respectively (Table 4 and Fig. 3). While the FP and LH were the main contributors to the second principal component (PC2) (Table 4 and Fig. 3). Thus, no improvement on the explained variance was obtained when the analyses were done using this approach. Figure 3 continues to support that viscoelastic properties are independent of FP, LV and LH.

4) Loading plot of first two principal components of selected variables after discarding redundant and low contributor variables of Table 3 based on viscoelastic using shear stress at 100 Pa and baking properties of six commercial wheat flours (Fig. 4 and Table 5)

After the redundant and low contributor variables were removed from Fig. 2, Table 3, the total variance explained was 83.8% (Fig. 4, Table 5). The variances identical to that obtained with the analysis of viscoelastic properties at 100 Pa, elasticity, baking properties and FP (Fig. 2 and Table 3). The individual contributions to the variance of J-Jr (100 Pa) and TCC (100 Pa) to PC1 were 81.8 and 78.7%, respectively (Table 5, Fig. 4). While, FP and LV individual contribution to the explained variance of PC2 were 94.2 and 80.3%, respectively (Table 5 and Fig. 4). Similar separation of the samples was observed in the analysis compared to the previously discussed analysis. The result suggest that similar discriminating ability of separating the characteristics of the set of samples studied can be obtained by using a creep-recovery test at 100 Pa, LV and FP. The results obtained justify the use of a larger sample size to compare the contribution to the explained variance when more genotypes or commercial samples are represented.

# 3.2 The correlations between properties by Pearson correlation

# 3.2.1 The viscoelastic properties

The results of viscoelastic properties obtained from creep and recovery tests using shear stress at 40 and 100 Pa were reported in Table 6. There was no correlation between flour protein and viscoelastic properties from both shear stresses (Table 6). This suggests that the viscoelastic properties are independent of protein content. Only J-Jr at 100 Pa showed a high correlation with DT and BT (r = 0.90 and 0.85, P < 0.01, respectively) (Table 6). This suggests that the gluten viscosity obtained at a shear stress of 100 Pa in a creep and recovery test has potential to be used for determining differences in dough properties such as development time and breaking time and thus more valuable in relating to empirical test (Table 6). It is interesting to note that ST, WA, LH, SV, and LV were not correlated to any viscoelastic parameters (Table 6). The viscoelastic properties using shear stress at 40 Pa (SeP, J-Jr, RCY and TCC) (r = -0.81, P < 0.05, r = 0.82, -0.90 and 0.77, P < 0.01, respectively, Table 6) showed a higher correlation with OSP compared to RCY, TCC and TCR from using shear stress at 100 Pa (r = 0.59, 0.62 and 0.62, respectively, P < 0.05, Table 6). Rmax correlated with all the viscoelastic parameters when 40 Pa were used while only two negative correlations were observed with 100 Pa (TCC and TCR, r = -0.66 and -0.70, P < 0.05) (Table 6).

# 3.2.2 Extensibility

There was a negative correlation between Rmax and Emax in dough (r = -0.59, P < 0.05, Table 6). This was supported by a study of gluten by using the same SMS/Kieffer rig measurement shown a high negative correlation between those two parameters (r = -0.90) (Tronsmo et al., 2003). Tronsmo et al. (2003) also indicated that adding salt solution induces ionic bonding in dough. Their results showed an increase of dough's resistance to extension as a function of salt addition. The extensibility of dough measured by Kieffer test was highly correlated to dough mixing attributes (Table 6). Highly negative correlation between Rmax and J-Jr, and Rmax and TCC at 40 Pa was observed (r = -0.87 and - 0.93, respectively, P < 0.01) (Table 6). While, extensibility (Emax) and J-Jr and TCR at 100 Pa, showed highly positive correlation (r = 0.93 and

0.82, P < 0.01) (Table 6). Only Area was highly correlated with flour protein content (r = 0.90, P < 0.01) (Table 6). Thus, as protein content decreases the Rmax were also decreases. The viscoelasticity property of gluten did not have a correlation with the extensibility of dough except for J-Jr at 100 Pa which was correlated with Area (r = 0.77, P < 0.01) (Table 6).

# 3.2.3 Dough mixing properties

Mixing properties were highly correlated with baking properties in almost all parameters except for DT which did not show any correlation. Oliver and Allen (1992) also indicated that the dough development time had low relationship with bread volume. In contrast, water absorption has been reported with low correlation with baking test (Oliver and Allen et al., 1992). There was no correlation between dough mixing properties and viscoelasticity observed at low shear stress (40 Pa); whilst, at higher shear stress (100 Pa), J-Jr and DT, BT showed a high correlation (r = 0.90, 0.85, P < 0.01) (Table 6). This suggests that using shear stress at 100 Pa used have more potential for revealing relationships with dough mixing properties than applying shear stress at 40 Pa.

#### 3.2.4 Baking properties

There was a high correlation between LH, SV, and LV with flour protein contents (r = 0.90, 0.89, and 0.90, P < 0.01) (Table 6). However, correlation between flour protein content and OSP was not found (Table 6). This means that the difference between loaf height and proof heights has no relationship with flour protein contents. No correlations were found between baking properties (loaf height (LH), specific volume (SV), and loaf volume (LV) and viscoelastic properties from creep and recovery test by using both shear

stress for 40 Pa and 100 Pa (Table 6). Rmax in extension properties has significantly high correlation with proof height (PH) in baking properties (r = 0.90, P < 0.01) (Table 6).

# 3.2.5 Flour properties

Flour protein contents showed a highly positive correlation with all dough mixing parameters (Table 6). The various flour protein contents did not reflect the change of shear stress in viscoelastic properties and are independent (Table 6). Rmax and Emax were not significantly correlated with protein content (Table 6). However, Area was significantly correlated with flour protein content (r = 0.90, P < 0.01) (Table 6). Similar correlations between the protein content and Area under the extension curve have been reported (Tronsmo et al., 2003).

# 4. Conclusion

This study revealed that using viscoelastic properties obtained with a creeprecovery test at 100 Pa, LV and FP yielded similar explained variance (83.8%) compared to using the mentioned variables plus extensibility and mixing properties. It also revealed that the viscoelastic properties obtained with a creep-recovery test are independent with FP, LV and WA. The viscoelastic parameters obtained explained the largest percent of the variance. The evidence of this study justifies the proposal to use creep-recovery test in wheat breeding programs and perhaps in milling laboratories.

Flours	Protein (%)	Moisture (%)	Ash (%)
A1	$7.95\pm0.05$	$11.69\pm0.02$	$0.29\pm0.01$
A2	$11.19\pm0.07$	$10.51\pm0.03$	$0.38\pm0.01$
A3	$13.68\pm0.02$	$10.14\pm0.02$	$0.41\pm0.00$
<b>B1</b>	$10.40\pm0.10$	$12.54\pm0.02$	$0.47\pm0.00$
<b>B2</b>	$10.59\pm0.07$	$12.57\pm0.00$	$0.48\pm0.01$
<b>B3</b>	$11.38\pm0.01$	$12.98\pm0.04$	$0.58\pm0.01$

Table 1. Proximate analysis of flours (means  $\pm$  SD, n=2).

SD = standard deviation.

Tests	Axes	PC1	PC2	1+2
	PC (%)	40.9	38.2	79.1
Creen and recovery	SeP	<i>13 1</i>	15	11 9
(100 Da)		4J.4	1.5	-++.) 75 0
$(100 \ ra)$	J-J <sub>r</sub>	01.8	14.0	75.0
	RCY	30.3	0.9	31.2
	TCR	72.1	1.4	/3.5
	TCC	73.9	0.6	74.5
Creep and recovery	SeP	60.9	6.8	67.8
(40 Pa)	J-J <sub>r</sub>	86.7	5.1	91.8
	RCY	52.2	1.7	54.0
	TCR	56.0	32.3	88.3
	TCC	34.1	23.1	57.2
Extension	Rmax	57.4	38.5	96.0
	Emax	86.4	5.6	92.0
	Area	34.9	59.6	94.5
Farinograph	WA	0.1	97.5	97.5
	DT	36.4	47.5	84.0
	ST	22.3	58.2	80.5
	BT	36.2	54.7	90.9
Baking	РН	24.3	69.9	94.2
0	LH	0.0	87.7	87.7
	SV	0.4	84.4	84.7
	OSP	64.4	6.1	70.5
	LV	0.0	89.7	89.7
Flour Protein	FP	5.5	92.6	98.2

Table 2. Explained variance (%) in PCA of the viscoelastic properties of gluten at 40 and 100 Pa, extensibility of dough, farinograph, and baking characteristics. The definitions of abbreviations are in Appendix 1, Table 1

Tests	Axes	PC1	PC2	1+2
	PC (%)	53.5	30.3	83.8
Creep and recovery	SeP	62.4	11.2	73.6
(100 Pa)	J-Jr	85.9	3.3	89.3
	RCY	39.3	0.0	39.4
	TCR	80.4	6.7	87.1
	TCC	78.2	8.2	86.4
Extension	Rmax	32.5	50.9	83.4
	Emax	89.4	0.6	90.0
	Area	49.5	46.4	95.9
Baking	LV	0.7	97.4	98.1
Flour Protein	FP	16.5	77.7	94.1

Table 3. Explained variance (%) in PCA of the viscoelastic properties of gluten at 100 Pa, extensibility of dough, loaf volume, and flour protein. The definitions of abbreviations are in Appendix 1, Table 1.

Tests	Axes	PC1	PC2	1+2	
	PC (%)	41.4	35.5	76.9	
<b>Baking properties</b>	LV	25.7	71.9	97.6	
	LH	22.6	75.6	98.2	
Creep and recovery	RCY 40 Pa	36.0	12.9	48.9	
-	J-Jr 40 Pa	68.8	6.4	75.2	
	RCY 100 Pa	39.4	23.6	63.1	
	SeP 100 Pa	50.6	0.9	51.5	
	TCC 100 Pa	79.4	11.7	91.1	
Flour protein	FP	8.4	80.8	89.2	

Table 4. Explained variance (%) in PCA of selected variables after discarding redundant variables of Table 2 The definitions of abbreviations are in Appendix 1, Table 1

Tests	Axes	PC1	PC2	1+2		
	PC (%)	50.4	33.5	83.8		
Creep and recovery	SeP	63.7	12.8	76.4		
(100 Pa)	J-Jr	81.8	2.6	84.4		
	RCY	59.2	0.8	60.0		
	TCC	78.7	10.1	88.7		
<b>Baking Properties</b>	LV	1.4	94.2	95.6		
Flour protein	FP	17.4	80.3	97.8		

Table 5. Explained variance (%) in PCA of selected variables after discarding redundant variables of Table 3. The definitions of abbreviations are in Appendix 1, Table 1

	FP	WA	DT	ST	вт	РН	LH	SV	OSP	LV	Rmax	Emax	Area	SeP 40Pa	J-Jr 40Pa	RCY 40Pa	TCC 40Pa	TCR 40Pa	SeP 100Pa	J-Jr 100Pa	RCY 100Pa	TCC 100Pa	TCR 100Pa
FP	1																						
WA	0.95**	1																					
DT	0.82**	0.71**	1																				
ST	0.84**	0.79**	0.71*	1																			
BT	0.86**	0.76**	0.99**	0.78**	1																		
РН	0.67*	0.83**				1																	
LH	0.90**	0.91**		0.79**	0.62*	0.78**	1																
SV	0.89**	0.91**		0.88**	0.63*	0.74**	0.96**	1															
OSP						-0.69*			1														
LV	0.9**	0.93**		0.82**	0.62*	0.80**	0.97**	0.99**		1													
Rmax		0.58*				0.90**			-0.77*	0.62*	1												
Emax			0.82**		0.80**				0.66*		-0.59*	1											
Area	0.90**	0.75**	0.89**	0.81**	0.93**		0.73**	0.72**		0.71*		0.76**	1										
SeP40Pa						0.59*			-0.81*		0.73**			1									
J-Jr40Pa						-0.63*			0.82**		-0.87**	0.78**		-0.89**	1								
RCY40Pa									-0.90**		0.61*	-0.58*		0.83**	-0.75	1							
TCC40Pa						-0.82*			0.77**		- 0.93**			-0.85**	0.90**	-0.67*	1						
TCR40Pa						-0.61*					-0.74**			-0.86**	0.78		0.87	1					
SeP100Pa												-0.65*							1				
J-Jr100Pa			0.90**		0.85**							0.93**	0.77**		0.58*				-0.72**	1			
RCY100Pa									0.59*						0.72**						1		
TCC100Pa									0.62*		-0.66*	0.74**		-0.58*	0.71**		0.60*		-0.72**	0.69*	-0.77**	1	
TCR100Pa									0.62*		-0.7*	0.82**							-0.82**	0.73**		0.86**	1

Table 6. Pearson's correlation coefficients of the viscoelastic properties of gluten, extensibility of dough, farinograph, and baking characteristics. The definitions of abbreviations are in Appendix 1, Table 1

\*Correlation is significant at  $\alpha = 0.05$  level.

\*\*Correlation is significant at  $\alpha = 0.01$  level.



**Total explained variance is 79.1%** 

Fig. 1. Loading plot of first two principal components based on viscoelastic, mixing, extensibility, and baking properties of six commercial wheat flours. The definitions of abbreviations are in Appendix 1, Table 1.



Fig. 2. Loading plot of first two principal components based on viscoelastic using shear stress at 100 Pa, extensibility, and baking properties of six commercial wheat flours. The definitions of abbreviations are in Appendix 1, Table 1.



Fig. 3. Loading plot of first two principal components of selected variables after discarding redundant and low contributors variables of Table 2 based on viscoelastic, mixing, extensibility and baking properties of six commercial wheat flours. The definitions of abbreviations are in Appendix 1, Table 1.



Fig. 4. Loading plot of first two principal components of selected variables after discarding redundant and low contributor variables of Table 3 based on viscoelastic using shear stress at 100 Pa and baking properties of six commercial wheat flours. The definitions of abbreviations are in Appendix 1, Table 1.

# CHAPTER V

# ASSESSMENT OF VARIATION IN HARD RED WINTER WHEAT FLOUR PROPERTIES FROM CREEP-RECOVERY, EXTENSIBILITY TESTS AND GLUTEN CONTENT COMPARED WITH TRADITIONAL WHEAT QUALITY TESTING Abstract

Large deformation rheological measurements have been proposed as potential tools to predict baking potential. Two sets of 51 hard red winter wheat flours from wheat grown in 2008 and 2009 were investigated. Gluten viscoelasticity, extensibility, wet gluten, sedimentation, flour protein and dough mixing and baking properties were analyzed. The total explained variance for the 2008 and 2009 sets was 53.2 and 49.9%, respectively, when all the variables were included. The major contributors to the first principal component were gluten strength and recoverable work in the 2008 set; while for the 2009 were gluten Separation time and %Recoverability. Flour protein, baking water absorption and loaf volume were highly associated with the second principal component in the 2008 set. In contrast, for the 2009 set, gluten work of extensibility, strength and recoverable work were highly associated with the second principal component. When the most important variables contributing to the explained variance were selected, an improvement in the explained variance was obtained for the 2008 set, 77% explained variance compared to 53.2% when all the variables were included.
A modest improvement in the total explained variance was obtained when selected variables were analyzed in the 2009 set (51.3% compared to 44.9% with all the variables). In both set of samples gluten properties (%recoverability, J-Jr, and strength) and dough mixing time explained larger percentage of variance than the baking properties and flour protein. This study also showed that loaf volume and flour protein are independent from most of the gluten viscoelastic properties and dough mixing time.

**Keywords:** Rheological properties, creep and recovery test, tensile test, baking properties, principal component analysis

## 1. Introduction

The quantitative and qualitative attributes of gluten protein account for the differences in baking performance and these are depending on wheat cultivars. Wheat gluten is made of storage proteins consisting of gliadin and glutenin which contribute to the viscosity and elasticity of the dough, respectively (Edwards et al., 2001; Khatkar et al., 1996; Taylor and Cluskey, 1962). Wheat flour quality can be determined in terms of dough properties and gluten attributes by using various measurements with the objective of predicting the breadmaking potential in wheat breeding programs. The relationship between high molecular weight-glutenin subunits (HMW-GS) and their baking quality has been studied extensively. It has been well established that subunits 5+10 have a positive correlation with strong dough and high baking characteristics (Dong et al., 1992).

Conventionally, a mixograph is defined as a low time consuming measurement of dough mixing properties requiring low amount of flour samples for the differentiation between good and poor wheat flours (Khatkar, Bell et al., 1996; Shogren and Finney, 1984). The mixing properties of wheat flours obtained with the mixograph consist of mixing time, water absorption, and mixing tolerance index. Genetics is one of the rationales of selecting methods in wheat breeding programs. The indication of wheat quality using the mixograph has been applied in hard winter wheat growing regions in the United States (Chung et al., 2001; Dong, Sears et al., 1992). The mixograph parameters have been widely used for differentiating the potential wheat in most breeding programs around the world. Besides genetics factors, the composition of HMW-GS and LMW-GS and the amount of gluten protein fraction influence the dough mixing properties (Zhang

et al., 2009). A strong correlation between the amount of gluten protein fraction (glutenin subunit composition, LMW-GS, and glutenin subunits - B3) and the dough mixing parameters have been reported(Zhang, Tang et al., 2009). Sodium Dodecyl Sulfate (SDS)-sedimentation test has also been widely applied for assessing the protein wheat quality in breeding programs (Delwiche et al., 1998; Khatkar, Bell et al., 1996). SDS-sedimentation parameters and protein content also had a positive correlation with mixing properties and baking test in hard red winter wheat (Peterson et al., 1998). However, it appears that the SDS-sedimentation test has limitation to distinguish strong or extra strong wheat quality (Wang and Kovacs, 2002). Gluten index (GI) and wet gluten (WG) was reported to describe both quantity and quality of wheat flours (Perten, 1990). Protein quality can be affected by the presence of glutenin alleles in each locus and it was reported to influence the gluten index (Tabiki et al., 2006). An effect on a double-haploid population between two wheat cultivars showed that the presence of Glu-D1 *d* or Glu-B3 *b* alleles provided a higher gluten index (Tabiki, Ikeguchi et al., 2006).

Rheological properties of gluten are significant characteristics reflecting the quality of wheat flour and perhaps end-use products. For example, extension test is one useful approach applying a large deformation to measure the gluten quality (Abang Zaidel, Chin et al., 2008). The gluten extensibility can be determined by tensile test using a texture analyzer(Abang Zaidel, Chin et al., 2008). The gluten tensile test can distinguish glutens from strong wheat that had higher extensibilities from those of weak wheat flour(Abang Zaidel, Chin et al., 2008). Creep and recovery test is a rheological measurement performed by applying a constant shear stress to gluten. Viscoelastic properties are obtained by applying small or large deformations of gluten in creep and

recovery measurements. However, evidence of direct relationship with bread volume or any other bread characteristic is missing in the literature. Breeding programs may benefit from viscoelastic parameters that can assess differences in quality. The objective of this study was to compare explanation of the variance by traditional measurements with gluten creep-recovery, gluten extensibility and glutomatic tests of two sets of winter wheat sample properties grown in 2008 and 2009.

## 2. Materials and method

## 2.1 Material

Breeder lines and cultivars of hard red wheat winter flours from two sets of 51 samples grown in 2008 and 2009 were evaluated. The samples were grown in three nurseries around Oklahoma representing slightly different environments.

## 2.2 Methods

## 2.2.1 Gluten preparation

Wet glutens were isolated by washing 10 g of flour with 2% NaCl solution (w/v) for 10 minutes from using a Glutomatic 2200 instrument (Perten Instruments, Sweden). The wet glutens were analyzed in two replicates with coefficient of variation less than 10% within the replicates.

## 2.2.2 Creep and recovery measurement of gluten

The creep and recovery method was performed as described in Chapter IV. The constant stress was applied by using shear stress at 100 Pa for this study.

## 2.2.3 Tensile test of gluten-extensibility

Washed gluten from Glutomatic was relaxed using the same method as describe in the creep and recovery test. After 60 min, the rested gluten was cut by using the bone shape cutter of 62 mm in width and 175 mm in length. The gluten tensile test was evaluated following the window-pane method of Zhao et al. (2010). The tensile test was evaluated by using the Texture Analyzer (TA.XTPlus, Texture Technologies Corp., Scarsdale, NY/Stable Micro Systems, Godalming, Surrey, UK). Briefly, the gluten samples were gently transferred to a window pane paper support. The window pane measured 10 mm width and 12.7 mm length. The gluten was well attached to the window pane by using Velcro dots on the two ends of the gluten. The gluten with window pane paper was tightened to the texture analyzer grips in vertical direction. The two sides of window pane paper were cut before the test started. The test was run in two replicates. The force (F), work of extensibility (WE), recoverable work (RE), and elasticity degree (DE) were obtained to explain the extensibility of gluten.

#### 2.2.4 Gluten index and wet gluten measurements

Isolated gluten obtained from the Glutomatic machine was immediately transferred to a special sieve and centrifuged at  $6000 \pm 5$  rpm in the Gluten index centrifuge for 1 min. The wet gluten (WG) is the weight of the entire amount of gluten. The gluten index (GI) was calculated by using the fraction of the gluten that is retained on the sieve and the gluten that passes through the sieve. The more the gluten passes through, the weaker the gluten is. The test was performed in duplicates.

## 2.2.5 Mixograph

The mixing properties were determined following the methods described by Yeap (2008). Briefly, the flour samples (10 g) were analyzed by using a Mixograph (National Manufacturing Co., Lincoln, NE) and Approved Method in 54-40A (AACC International 2000). Dough mixing quality was expressed by three parameters: corrected mixing time (CMT), mixing stability (MST), and tail width (MTW).

## 2.2.6 Sodium Dodecyl Sulfate (SDS)-Sedimentation

The gluten strength was analyzed as described by Yeap (2008). Briefly, the smallscale SDS sedimentation was determined according to Approved Method 56-61A (AACC International 2000).

## 2.2.7 Baking test

The baking properties were determined as described in Chapter IV. The loaf volume (LV), visual score (ViSc), and baking water absorption (BWA) were recorded.

#### 2.2.8 Statistical analysis

The parameters were analyzed with Principal Component Analysis (PCA) and Pearson correlation (P < 0.001 and 0.05). The software used was Canoco for Windows 4.5 (Biometris, Plant Research International, Wageningen, the Netherlands) for Principal Component Analysis and SAS (Statistical Analysis System, SAS Institute Inc., Cary, NC) for Pearson correlation using the CORR procedure.

#### 3. Results and discussion

## 3.1 The properties of 2008 and 2009 sample flours

The mean values for Flour Protein (FP), Delta compliance (J-Jr), Separation time (SeP), %recoverability (RCY), SDS sedimentation (SED), Gluten Index (GI), Wet Gluten (WG), Force (F), Work extensibility (WE), Degree of Elasticity (DE), Loaf volume (LV), Visual score (ViSc), Baking Water absorption (BWA), Corrected Mixing Time (CMT), Mixing Stability (MST) and Mixing Tail Width (MTW) from 51 samples of each 2008 and 2009 set were shown in Tables 1 and 2.

#### 3.1.1 Creep and recovery test

The viscoelastic properties of gluten were obtained from the creep and recovery test. Strong gluten samples will show a low viscosity indicated by a large J-Jr value while weak gluten samples will show low elasticity explained by SeP and RCY (Fig. 1 in Chapter III). Gluten samples from 2008 had mean (range) values of J-Jr, RCY, and SeP of 0.7 Pa<sup>-1</sup> (range 0.2-1.6 Pa<sup>-1</sup>), 80.8% (74.6-84.9%), and 4.25 s (0.3-10.2 s), respectively (Table 1a-c). The mean (range) of gluten samples from 2009 were J-Jr 0.7 Pa<sup>-1</sup> (0.2-2.5 Pa<sup>-1</sup>), RCY 80.3% (73.8-84.4%), and SeP 3.5 s (0.1-7.6 s) (Table 2a-c). Line 5312, in sample set from 2009, showed the highest J-Jr and the lowest RCY (Table 2c). Higher mean values were observed in the 2008 samples compared to the 2009 samples. This can be explained in part by differences in environmental and genetic factors.

# 3.1.2 Tensile test

The tensile test assessed the extensibility of gluten samples by evaluating F, WE, RW, and DE. WE and RW were highly correlated with F in both years which are year

2008 (r = 0.77 and 0.80, P < 0.01) (Table 7) and 2009 samples (r = 0.99 and 0.99, P < 0.01) (Table 8). Thus, strength of gluten (F) had a significant correlation with the work of extensibility and RW (elasticity) of gluten of these two sets of samples (Table 3 and 4). From the tensile test for the 2008 set the mean (range) values of F were 0.4 N (0.7-1.6 N), WE 1.7 N.cm (11.0-0.5 N.cm), RW 0.54 N.cm (3.14-0.20 N.cm), and DE 32.94 (47.27-25.69) (Table 1a-c). The mean (range) values for the 2009 set were F 0.3 N (0.1-0.7 N), WE 1.06 N.cm (0.30-2.7 N.cm), RW 0.4 N.cm (0.1-0.9 N.cm), and DE 37.7 (27.3-45.5) (Table 2a-c). In the 2008 set, line 6609 had the highest DE (Table 1a); while, line 3305 showed the lowest F, WE and RW (Table 1a). In the 2009 set, Asp had the highest values of F and WE and line 5312 showed the lowest F, WE and RW values (Table 2a).

### 3.1.3 Glutomatic measurements

Gluten Index (GI) and Wet Gluten (WG) explained the strength and quantity of gluten, respectively. No correlation was found between GI and WG from the sample set of 2008 (Table 3) while a weak but significant negative correlation was observed for the sample set of 2009 (r = -0.35, P < 0.01) (Table 4). A high correlation between WG and FP was found in the 2008 set (r = 0.76, P < 0.01) (Table 3) but no correlation was found in the 2009 set (Table 8). The mean (range) values of GI for 2008 samples were 92.9% (100-60.6%) and of WG 27.2% (33.6-21.6%) (Table 1a-c). The mean (range) values of GI for 2009 samples were 96.3% (100-67.3%), WG 28.1% (34.9-23.3%) (Table 2a-c). In the 2008 set, line 6345 had the lowest GI and line 6822w showed the lowest WG (Table 1a). Line 4315 had the highest WG and FP (Table 1b) in the 2008 set.

## 3.1.4 Mixograph

Mixing properties were obtained by using the Mixograph. In the 2008 set, mean (range) values of CMT were 4.2 s (6.5-2.7 s), MST 6.70 N.cm (12-1.20 N.cm), and MTW 16.7 (33.2-4.4) (Table 1a-c). The mean (range) values for the 2009 set were CMT 4.2 s (2.4-5.5 s), MST 6.3 N.cm (0.6-13.8 N.cm), and MTW 18.1 (10.8-33.5) (Table 2a-c). CMT and F showed a significantly high correlation only for the 2008 set (r = 0.67, P <0.01) (Table 7). Line 6528 (2008 set) had the highest CMT and lowest MST (Table 1a). Line 7820w (2009 set) showed the highest MST, lowest elasticity (SeP) and highest viscosity (J-Jr) (Table 2a).

# 3.1.5 SDS sedimentation

The mean (range) values of SDS sedimentation for the 2008 set was 7.2 (4.6-9.2) (Table 1c) while for 2009 was 6.9 (5.5-8.4) (Table 2c). The SED values showed significantly weak correlation with all parameters in both set of samples (Table 3 and 4).

# 3.1.6 Baking test

The mean (range) values for the 2008 set were LV 818.51 (723-950 N), ViSc 6.5 N.cm (4-8 N.cm), and BWA 63.1 (61.5-64.0) (Table 1c). Overall similar values were observed for the 2009 set; LV 822.6 (700-960 N), ViSc 19.3 (5-41.9 N.cm), and BWA 64.1 (63-66) (Table 2c). The baking properties had significantly but weak relationship with all parameters in both set of samples (Table 7 and 8) (maximum r = 0.48, P < 0.01).

#### 3.2 Correlations among properties of the 2008 set

Principal component analysis (PCA) revealed the relationships among the variables and samples, including the variables of viscoelastic properties, dough mixing properties, sedimentation test, gluten extensibility properties and gluten strength (Fig. 1 and Table 3). Figure 1 displays the bi-plot of the 2008 set explaining 53.2% of the total variance. The first principal component (PC1) was highly correlated with the force (F) and recoverable work of gluten (RW). PC1 explained 28.4% of the total variance (Fig. 1 and Table 3). Gluten strength was the most significant contribution of individual variance to PC1 with 79.5% of the explained variance in sample set 2008 (Fig. 1 and Table 3). The second component (PC2) showed that flour protein content (FP) and baking water absorption (BWA) were the main contributors; PC2 explained 24.8% of the total variance (Fig. 1 and Table 3).

The majority of the variables were found in the first quadrant. Mixograph stability (MST) and wet gluten (WG) were related in the second quadrant (upper right hand side, Fig. 1). MST and WG were negatively related with the degree of elasticity (DE) (Fig. 1 and Table 3). r = -0.28 and -0.45, P < 0.01, respectively Samples from N91 (6127, 6729, 6822, 6722, and Endurance) (Group 1) revealed a relation with the degree of elasticity of gluten (Fig. 1). The viscous component (J-Jr) was negatively correlated with the Force (F, maximum force in the tensile test) (Fig. 1 and Table 3) and this relationship was supported by the Pearson correlation (r = -0.58, P < 0.01 and Table 7). The lines of N92 (3305 and 3825), and N91 (6332) (group 2) were strongly correlated to J-Jr (Fig. 1 and Table 3). While lines 6629, 5312, 6345, 6814, and 5204 (group 3) were weakly related to their viscosity component (J-Jr) since they are further away (Fig. 1). Interestingly, line

6609 was close to J-Jr; even though, it had the highest F value (Fig. 1 and Table 3). This suggests that line 6609 was strong (high F) and also viscous (Fig. 1). Pearson correlation revealed no significant correlation with the flour protein content and baking performance (Table 7). In PCA, LV and FP vectors were identical and they were highly related to PC2 (Fig. 1and Table 3). LV and FP were also independent of gluten viscoelastic properties. The sedimentation showed a weak correlation with almost all the variables except for the viscoelastic properties (Table 7, Pearson correlation).

PCA revealed a number of variables with short vectors (not important contributors to the explained variance and redundant variables) (i.e., almost one on top of each other). The short vector and redundant variables were discarded and the sets were re-analyzed as illustrated in the PCA bi-plot (Fig. 3 and Table 5). The PCA with the selected variables explained 77.0% of the variance which was better when compared to the PCA containing all the variables (53.2% of the variance) (Fig. 2 and Table 3). PC1 explained 45.4% of the variance and was mainly associated with CMT and gluten strength (F) and a distant third contributor J-Jr (Fig. 3 and Table 5). This suggests that in this set (2008) CMT was highly associated to the gluten strength measured in the tensile test (Fig. 3 and Table 5). PC2 was correlated to LV and flour protein content (FP) explaining 31.6% of the variance (Fig. 3 and Table 5). Group 1 was associated with PC2; while, group 3 was associated with PC1 (Fig. 3). However, even after discarding redundant variables there were some lines which show weak correlations with the variables and PC1 and PC2 and were mainly from N91 and N93 (Group 2) (Fig. 3).

### 3.3 Correlations among properties of 2009 set

The PCA of the 2009 set was illustrated in Figure 2 and Table 4 with PC1 and PC2 explaining 44.9% of total variance (Fig. 2, Table 4). The first principal component (PC1) was associated with RCY and SeP (elastic properties of gluten). PC1 explained 25.8% of the variance (Fig. 2 and Table 4). PC2 explained 19.1% of the variance and was influenced by the gluten properties of strength (F) and WE from the tensile test (Fig. 2 and Table 4). RW, WE, and F were highly correlated and very close to each other plus they contributed almost equally to the PC1 and PC2 (Fig 2). This suggests that the three variables were redundant (Fig. 2). Gluten DE was negatively correlated to F, WE, RW (Fig. 2) which was confirmed by the Pearson correlations (r = -0.58, -0.61 and -0.46, respectively; P < 0.01) (Table 8). Flour protein content had no correlation with SED, gluten extensibility and gluten strength (Table 8). WG was closely related to PC1 but only contributed with 35.3% to the total variance (Fig. 2 and Table 4). The higher the magnitude of vector, the more explanation of the variable is. Interestingly, the 2009 set showed a correlation of the flour protein and the baking performance unlike the 2008 set (Table 7 and 8). However in PCA, the vectors for these variables were very small and contributed minimally to the explained variance (Fig. 2 and Table 4).

Almost all the samples from N91 were closely correlated to GI, FP, ViSc, BWA, and RCY except for Asp (Fig. 2) (Group 1). This suggests that lines from N93 were closely related to the gluten strength, baking performance and viscoelasticity. However, the samples from N91 were correlated to RCY which individually contributed with 67.5% of the explained variance in PC1 in contrast to WG, LV and MTW which showed individually small contributions (Fig. 2 and Table 4). N93 samples were closely

correlated to the gluten quantity (WG), baking performance (LV) and sedimentation (SED) (Fig. 2) (Group 1).

After reanalysis of PCA by removing the small vectors and the redundant variables, the PCA of the 2009 samples explained 51.3% of the total variance (Fig. 4 and Table 6) which is a modest improvement from 44.9% with all variables (Fig. 2). FP showed less explanation in the loading plot compared to analysis with all the variables (Fig. 4, Table 6 and Fig. 2, Table 4). PC1 accounted for 31.2% of the explained variance and the main contributors were form the viscoelastic properties of RCY and J-Jr (Fig. 4 and Table 6). PC2 was mainly associated with dough mixing properties of CMT and MST. PC2 explained 20% of the variance (Fig. 4 and Table 6). The viscous component (J-Jr) showed highly negative relationship with the elastic component RCY and this was supported by Pearson correlation with r = -0.98 (P < 0.01) (Table 8). This suggests that J-Jr and RCY were important contributors in explaining the variance of the two sets (Fig. 2 and 4, Table 4 and 6). The baking performance parameters LV, ViSc, and BWA were discarded since they had a limited contribution to the explained variance (Fig. 4 and Table 6). This suggests that the physical properties explained more of the variance compared to the baking properties (Fig. 4 and Table 6). Thus, the physical properties of these two sets of samples were more varied but the samples baking properties showed lower variability.

## 4. Conclusions

The parameters from viscoelastic properties (%Recoverability, Separation time, and J-Jr) and tensile test (Force, Recoverable work, and Work of extensibility) appear to be good candidates for the differentiation of physical properties in breeding programs.

After redundant variables were removed, the total explained variance was higher from the 2008 and 2009 year samples (77% and 53.1%, respectively). Overall, FP and LV were independent from viscoelastic and mixing properties. Viscoelastic and tensile parameters contributed more to the explained variance compared to FP and LV and thus would assist in the selection of new cultivars as well as in quality control of milling operations.

				Cree	ep-Reco	very		Gluto	natic		Te	nsile			Baking		N	Aixograj	oh
Sample	Nur.	Abbr.	FP (%)	J-Jr (Pa <sup>-1</sup> )	SeP	RCY (%)	SED (ml)	GI (%)	WG (%)	F (N)	WE (N.cm)	RW (N.cm)	DE	LV (cc)	ViSc (score)	BWA (%)	СМТ	MST (min)	MTW (mm)
OK Bullet	91	Blt1	10.5	0.6	5.0	80.7	5.6	100.0	28.3	0.5	1.7	0.5	30.6	870.0	7.0	62.0	5.0	6.6	18.8
Endurance	91	End1	9.1	0.4	6.3	83.9	6.1	99.3	22.7	0.5	1.7	0.5	32.6	770.0	5.0	62.0	3.3	5.7	10.8
Overley	91	Ove1	9.9	0.3	3.3	84.2	7.2	100.0	24.8	0.6	2.0	0.8	42.5	825.0	7.0	62.5	5.2	6.3	33.2
OK02522W	91	252	10.1	0.4	8.0	83.1	6.9	100.0	27.3	0.4	1.4	0.5	38.2	850.0	7.0	63.0	4.6	6.0	15.9
OK06112	91	6112	10.1	0.5	7.1	83.1	5.9	99.3	28.2	0.5	1.6	0.5	30.0	830.0	7.0	63.5	5.1	4.0	13.2
OK06127	91	6127	10.2	0.4	6.2	84.5	5.4	96.1	27.5	0.4	1.6	0.5	29.4	750.0	5.0	62.0	5.0	3.0	10.4
OK06114	91	6114	9.2	0.8	4.4	78.8	6.9	96.7	24.4	0.3	1.1	0.3	28.1	825.0	7.0	62.5	4.1	2.9	11.3
OK06232	91	6232	9.6	0.5	3.1	80.3	7.6	100.0	24.0	0.6	2.1	0.7	31.8	843.0	7.0	63.0	4.7	5.2	13.4
OK06210	91	6210	9.8	0.9	5.1	82.2	7.2	90.8	26.8	0.4	1.1	0.4	31.6	853.0	6.0	62.0	3.0	10.4	17.5
OK06332	91	6332	10.1	1.1	2.0	79.5	5.7	74.8	28.8	0.2	0.6	0.2	31.7	840.0	6.0	62.5	3.2	12.0	14.6
OK06336	91	6336	9.9	0.5	5.1	77.4	8.8	100.0	23.8	0.5	2.0	0.7	34.0	800.0	6.0	62.5	6.3	1.6	16.0
OK06345	91	6345	9.2	0.9	2.8	80.8	6.4	60.6	27.1	0.3	0.9	0.3	30.5	750.0	4.0	62.5	2.7	6.5	14.3
OK06528	91	6528	10.0	0.3	2.8	83.5	8.5	100.0	23.0	0.6	1.9	0.9	47.3	760.0	6.0	62.5	6.5	1.2	13.1
OK06609	91	6609	9.8	0.8	3.7	80.0	5.6	89.6	29.5	0.7	1.4	0.2	47.3	810.0	6.0	64.0	3.5	10.7	9.5
OK06617	91	6617	10.8	0.6	6.4	82.8	8.6	95.9	30.7	0.3	1.2	0.4	29.4	790.0	7.0	64.0	3.8	7.9	16.3
OK06618	91	6618	10.8	1.0	3.7	80.7	5.7	96.7	29.5	0.3	0.9	0.3	34.0	850.0	8.0	63.0	4.2	9.2	16.6
OK06629	91	6629	10.0	1.2	0.5	78.9	4.6	67.1	28.2	0.3	1.2	0.4	33.7	725.0	6.0	62.5	3.0	9.6	8.0
OK06729	91	6729	9.3	0.7	4.1	81.5	7.2	100.0	25.1	0.4	9.8	2.6	28.6	723.0	6.0	62.0	4.1	3.6	14.0
OK06722	91	6722	8.5	0.3	4.6	82.9	6.3	99.1	22.2	0.5	11.0	3.1	29.0	735.0	7.0	63.0	4.1	3.7	13.5
OK06743W	91	6743	9.8	0.5	3.7	82.9	4.9	83.9	27.5	0.4	1.3	0.4	28.5	765.0	6.0	62.0	3.5	9.0	7.5
OK06814W	91	6814	9.1	1.3	0.4	76.9	6.3	88.9	24.6	0.3	1.0	0.3	29.3	775.0	6.0	62.0	3.0	6.7	15.7
OK06822W	91	6822	8.9	0.4	0.4	79.7	8.0	99.8	21.6	0.5	1.9	0.6	32.9	780.0	6.0	63.0	4.2	2.9	12.4

Table 1a. Mean values of 2008 wheat cultivars and breeder lines

				Cree	p-Recov	very		Glutomatic			Ter	nsile			Baking		N	Iixograp	oh
Sample	Nur.	Abbr.	FP (%)	J-Jr (Pa <sup>-1</sup> )	SeP	RCY (%)	SED (ml)	GI (%)	WG (%)	F (N)	WE (N.cm)	RW (N.cm)	DE	LV (cc)	ViSc (score)	BWA (%)	СМТ	MST (min)	MTW (mm)
OK06848W	91	6848	9.6	0.9	3.9	80.3	8.6	100.0	25.6	0.5	1.8	0.5	26.6	800.0	6.0	61.5	3.8	5.4	16.3
Duster	92	Dst	10.1	0.7	4.7	80.8	6.4	98.8	26.1	0.3	1.1	0.4	32.4	750.0	7.0	62.5	4.3	2.8	24.2
Endurance	92	End2	10.1	0.6	2.8	82.3	5.8	99.4	26.0	0.3	0.9	0.4	37.5	775.0	7.0	63.0	3.8	7.3	17.8
Deliver	92	Del	10.9	0.3	6.9	84.8	8.7	99.6	27.2	0.6	2.3	0.7	28.8	845.0	6.0	63.5	5.6	6.4	16.2
OK Bullet	92	Blt2	11.1	0.6	5.9	74.6	6.4	99.1	29.7	0.4	1.5	0.5	35.2	845.0	7.0	64.0	4.8	7.3	15.6
Overley	92	Ove2	11.1	0.4	5.7	80.7	8.5	99.8	27.7	0.6	2.0	0.7	33.6	880.0	8.0	64.0	4.6	12.0	22.0
Fuller	92	Ful	11.1	0.2	6.7	84.5	9.2	100.0	29.2	0.6	2.0	0.6	32.9	775.0	6.0	64.0	4.9	7.1	21.8
Centerfield	92	Ctf	10.9	0.7	3.3	80.0	8.0	86.4	30.5	0.4	1.7	0.4	26.5	865.0	6.0	64.0	3.2	8.8	15.6
Guymon	92	Guy	11.1	0.7	5.8	79.0	8.8	87.8	31.1	0.5	1.8	0.5	25.7	868.0	8.0	64.0	4.1	11.6	27.2
OK00611W	92	611	11.1	0.7	3.3	80.5	8.1	98.5	31.2	0.3	0.9	0.3	34.9	883.0	8.0	64.0	3.8	10.9	20.9
OK02522W	92	2522	10.9	0.6	4.7	80.7	7.4	97.6	29.0	0.4	1.5	0.5	33.5	825.0	7.0	63.5	4.5	9.3	15.8
OK03305	92	3305	9.8	0.9	3.8	78.3	8.1	85.5	27.0	0.2	0.5	0.2	37.8	850.0	6.0	64.0	3.2	9.9	21.4
OK03522	92	3522	10.5	0.6	3.7	78.8	8.6	98.7	28.1	0.4	1.5	0.5	31.6	850.0	7.0	63.5	4.3	4.7	18.0
OK05903C	92	5903	10.2	0.6	5.1	80.0	8.3	95.9	27.4	0.3	1.0	0.3	33.8	855.0	6.0	64.0	3.6	7.1	11.7
OK04525	92	4525	11.0	1.3	0.9	78.8	8.0	70.9	31.8	0.2	0.8	0.3	31.9	850.0	8.0	64.0	3.5	10.6	28.1
OK04111	92	4111	11.1	0.7	5.5	80.6	8.1	84.3	31.4	0.4	1.4	0.4	26.6	950.0	7.0	64.0	4.6	6.6	18.0
OK04315	92	4315	11.4	1.2	2.0	79.4	8.3	73.9	33.6	0.2	0.6	0.2	37.2	950.0	6.0	64.0	3.4	10.2	14.7
OK03825- 5403-6	92	3825	11.0	0.6	3.5	82.1	6.5	65.8	30.7	0.2	0.8	0.3	34.4	760.0	6.0	63.0	5.0	9.3	4.4
OK05711W	92	5711	11.2	0.5	5.1	81.8	8.7	99.1	29.4	0.4	1.5	0.5	36.1	850.0	7.0	64.0	6.0	4.6	21.6
OK Bullet	93	Blt3	11.1	0.7	4.0	79.2	6.9	100.0	29.5	0.4	1.3	0.4	31.0	888.0	8.0	64.0	5.2	5.1	21.3
OK01420W	93	1420	9.9	0.5	7.3	81.7	7.8	100.0	29.4	0.3	0.9	0.3	37.3	780.0	7.0	63.0	3.8	2.8	16.9

Table 1b. Mean values of 2008 wheat cultivars and breeder lines

				Cree	p-Recov	very		Gluto	matic		Ten	sile			Baking		I	Mixogra	ph
Sample	Nur.	Abbr.	FP (%)	J-Jr (Pa <sup>-1</sup> )	SeP	RCY (%)	SED (ml)	GI (%)	WG (%)	F (N)	WE (N.cm)	RW (N.cm)	DE	LV (cc)	ViSc (score)	BWA (%)	СМТ	MST (min)	MTW (mm)
OK05742W	93	5742	10.7	0.5	3.6	83.5	7.2	100.0	27.8	0.4	1.4	0.4	31.4	858.0	7.0	64.0	4.5	8.3	27.1
OK06029C	93	6029	10.1	0.3	10.2	84.9	7.3	96.1	26.7	0.4	1.4	0.5	33.9	860.0	8.0	63.0	4.7	6.9	16.2
OK05128	93	5128	9.9	0.2	6.0	81.3	8.6	100.0	22.2	0.4	1.6	0.6	38.7	870.0	6.0	63.0	4.5	4.5	14.2
OK05526	93	5526	10.3	0.5	2.3	80.3	9.0	99.6	23.6	0.5	1.9	0.6	30.0	815.0	7.0	63.0	4.9	4.3	22.8
OK05312	93	5312	9.3	1.6	0.3	77.2	5.7	74.2	26.6	0.2	0.8	0.2	30.7	763.0	6.0	62.0	2.8	6.7	12.7
OK05511	93	5511	9.7	0.6	3.8	81.0	7.1	99.3	22.2	0.5	1.6	0.5	30.3	805.0	6.0	63.0	4.3	5.9	15.4
OK05204	93	5204	9.3	0.8	4.4	79.4	5.1	91.1	27.4	0.2	0.7	0.3	36.6	760.0	5.0	62.0	3.3	5.1	21.3
OK05212	93	5212	10.4	0.8	3.1	76.4	7.2	97.9	26.1	0.3	0.9	0.3	31.9	800.0	6.0	63.0	3.4	5.3	18.6
		Max <sup>a</sup>	11.4	1.6	10.2	84.9	9.2	100.0	33.6	0.7	11.0	3.1	47.3	950.0	8.0	64.0	6.5	12.0	33.2
		Min <sup>b</sup>	8.5	0.2	0.3	74.6	4.6	60.6	21.6	0.2	1.5	0.2	25.7	723.0	4.0	61.5	2.7	1.2	4.4
		Average	10.2	0.7	4.3	80.8	7.2	92.9	27.2	0.4	1.7	0.5	32.9	818.5	6.5	63.1	4.2	6.7	16.7
		<b>SD</b> <sup>c</sup>	0.7	0.3	2.0	2.3	1.2	10.6	2.8	0.1	1.8	0.5	4.6	52.9	0.9	0.8	0.9	2.8	5.5

Table 1c. Mean values of 2008 wheat cultivars and breeder lines

 $Max^a = Maximun$ 

 $Min^a = Minimum$ 

SD<sup>c</sup> = Standard deviation

				Cree	p-Recov	very		Gluto	matic		Те	nsile			Baking			Mixog	raph
Nur.	Sample	Abbr.	FP (%)	J-Jr (Pa <sup>-1</sup> )	SeP	RCY (%)	SED (ml)	GI (%)	WG (%)	F (N)	WE (N.cm)	RW (N.cm)	DE	LV (cc)	ViSc (score)	BWA (%)	СМТ	MST (min)	MTW (mm)
Duster	91	Dst1	10.1	0.9	1.3	79.0	6.2	97.7	26.3	0.3	0.9	0.3	38.0	750.0	8.0	64.0	4.2	1.9	20.4
OK Bullet	91	Blt1	10.4	0.6	5.1	80.9	5.5	98.9	27.4	0.3	1.1	0.4	35.5	790.0	8.0	63.5	4.1	5.3	23.6
Shocker	91	Shk	11.3	0.8	2.4	78.8	6.3	91.0	30.9	0.2	0.7	0.3	39.3	875.0	9.5	64.0	3.7	10.0	14.2
Aspen	91	Asp	10.0	0.4	3.3	80.7	7.2	100.0	25.2	0.7	2.7	0.9	32.7	730.0	5.5	64.5	4.6	2.2	18.4
OK Rising	91	Ris1	10.9	0.6	5.3	81.2	6.3	100.0	29.5	0.2	0.7	0.3	42.1	823.0	8.0	65.0	3.9	8.5	21.1
Centerfield	91	Ctf1	11.0	1.0	3.3	79.8	6.7	94.9	30.6	0.2	0.5	0.2	39.8	960.0	7.5	65.0	3.9	7.0	13.3
OK07S117	91	7117	10.5	0.8	3.2	77.0	7.2	100.0	28.3	0.3	0.8	0.3	39.3	755.0	7.0	64.0	3.6	4.2	21.9
OK07209	91	7209	9.9	0.5	4.2	81.2	7.5	100.0	27.7	0.4	1.3	0.4	34.4	778.0	7.5	63.0	3.6	4.7	12.4
OK07210	91	7210	9.9	0.6	2.3	79.2	6.9	98.5	24.0	0.3	0.9	0.4	38.5	700.0	5.0	63.0	4.5	3.3	23.8
OK07214	91	7214	10.5	0.4	4.1	84.3	5.6	100.0	24.6	0.4	1.4	0.5	35.6	785.0	6.0	65.5	4.1	3.8	12.1
OK07216	91	7216	10.6	0.6	2.5	80.6	6.8	100.0	26.8	0.3	1.0	0.4	36.7	800.0	7.5	65.0	5.4	1.6	15.5
OK07218	91	7218	10.3	0.4	3.8	82.7	7.1	99.6	26.4	0.3	1.0	0.4	41.6	788.0	8.0	64.0	4.5	4.7	19.3
OK07226	91	7226	10.4	0.5	4.3	81.6	6.8	100.0	25.7	0.4	1.2	0.5	38.2	850.0	8.5	63.0	4.5	5.9	11.7
OK07231	91	7231	9.9	0.5	2.5	79.7	6.9	100.0	24.3	0.2	0.7	0.3	43.1	725.0	7.0	64.0	5.4	0.6	19.3
OK07418	91	7418	9.8	0.5	1.7	81.0	7.4	99.8	23.4	0.3	1.0	0.4	38.2	768.0	6.5	63.0	4.0	4.3	26.6
OK07615	91	7615	11.1	0.4	2.3	81.2	7.2	100.0	27.8	0.4	1.4	0.5	36.4	915.0	8.0	64.0	4.8	5.0	21.7
OK07719W	91	7719	9.9	0.6	2.4	79.6	8.4	100.0	25.8	0.4	1.6	0.5	32.1	818.0	8.0	63.0	3.3	7.0	12.7
OK07729W	91	7729	9.9	0.3	2.9	79.8	7.5	100.0	23.3	0.5	1.6	0.6	37.2	783.0	8.0	63.5	5.2	2.5	23.5
OK07742W	91	7742	10.7	0.9	1.9	80.5	6.7	83.6	29.4	0.3	1.1	0.3	31.0	825.0	8.0	65.0	3.4	5.2	14.2
OK07820W	91	7820	10.2	1.5	0.1	76.6	8.1	89.5	28.8	0.2	0.5	0.2	42.2	738.0	7.0	64.0	2.6	13.8	14.6
OK07919C	91	7919	9.8	0.7	3.0	78.6	7.3	99.0	24.9	0.4	1.4	0.4	27.3	800.0	7.5	64.0	3.4	4.4	15.8
Duster	92	Dst2	10.2	0.6	3.1	80.0	6.2	98.7	26.4	0.4	1.2	0.4	35.6	785.0	8.0	65.0	4.6	0.8	29.3

Table 2a. Mean values of 2009 wheat cultivars and breeder lines

				Cree	p-Reco	very		Gluto	matic		Ter	nsile			Baking		ľ	Mixograp	ph
Sample	Nur.	Abbr.	FP (%)	J-Jr (Pa <sup>-1</sup> )	SeP	RCY (%)	SED (ml)	GI (%)	WG (%)	F (N)	WE (N.cm)	RW (N.cm)	DE	LV (cc)	ViSc (score)	BWA (%)	СМТ	MST (min)	MTW (mm)
92	OK Bullet	Blt2	10.5	0.7	3.5	78.6	5.6	97.3	29.1	0.2	0.8	0.3	38.7	820.0	8.0	64.0	4.4	3.5	16.6
92	Overley	Ove	11.0	0.3	5.2	82.8	7.2	100.0	27.9	0.7	2.5	0.8	32.8	905.0	8.0	64.5	5.4	4.2	19.9
92	OK06127	6127	11.2	0.4	5.2	83.5	5.8	99.5	28.4	0.4	1.1	0.4	39.0	725.0	6.5	64.0	4.3	9.3	11.7
92	OK06332	6332	10.4	1.1	4.2	79.6	6.7	67.2	28.4	0.2	0.7	0.2	33.5	855.0	7.5	63.0	2.4	12.7	10.8
92	OK06336	6336	10.7	0.4	4.4	81.7	7.9	99.6	27.1	0.2	0.7	0.3	37.0	855.0	8.0	66.0	5.0	3.2	16.7
92	OK06609	6609	11.4	0.9	3.9	78.7	6.1	81.5	32.5	0.7	2.7	0.9	32.8	890.0	7.5	65.0	4.1	12.0	17.6
92	OK06528	6528	11.4	0.4	2.5	81.6	7.1	99.7	28.8	0.7	2.4	0.8	32.7	918.0	7.0	65.0	5.3	8.0	33.5
92	OK06822W	6822	9.9	0.4	2.5	81.6	7.3	100.0	23.8	0.5	1.8	0.6	35.0	838.0	7.5	64.0	4.1	4.3	12.2
93	Chisholm	Chl	11.6	0.6	4.5	79.5	7.7	99.6	28.2	0.3	1.1	0.4	39.2	890.0	31.1	65.0	5.0	9.0	22.7
93	Endurance	End3	10.3	0.6	2.2	80.8	6.8	98.1	25.2	0.3	0.9	0.4	38.0	810.0	35.9	63.0	3.7	6.9	10.9
93	OK Bullet Resiln	Blt3	10.7	0.8	2.1	77.5	6.1	99.7	29.2	0.3	0.8	0.4	42.6	845.0	31.9	64.0	4.9	1.6	29.0
93	Duster	Dst3	10.5	0.7	3.8	81.8	6.2	98.5	26.7	0.2	0.7	0.3	41.6	790.0	35.3	63.0	4.7	2.2	14.2
93	Fuller	Ful	11.3	0.6	2.3	80.5	7.7	100.0	28.2	0.4	1.3	0.5	37.2	840.0	32.9	64.0	5.5	4.8	15.6
93	Jackpot	Jap	11.6	0.8	3.9	78.6	6.4	93.5	31.9	0.1	0.4	0.2	37.2	810.0	35.9	65.0	4.3	8.2	17.0
93	Centerfield	Ctf3	11.0	0.9	4.6	80.6	7.4	95.1	29.4	0.1	0.4	0.2	37.2	845.0	38.3	64.0	3.3	8.6	14.4
93	OK Rising	Ris3	12.1	0.5	6.3	83.4	6.6	100.0	32.0	0.3	0.9	0.3	39.0	895.0	36.7	65.0	5.0	9.8	16.3
93	OK 03522	3522	11.2	0.7	2.6	79.8	7.6	97.9	29.0	0.4	1.1	0.4	39.4	875.0	38.7	64.0	4.3	4.4	17.3
93	OK 03305 Pete	3305	11.0	1.0	3.7	81.6	7.4	96.4	29.6	0.2	0.6	0.3	45.5	890.0	36.5	64.0	4.1	7.6	17.2
93	OK 03825- 5403-6	3825	11.3	1.0	2.3	80.1	6.4	83.7	31.4	0.2	0.6	0.2	42.6	810.0	36.9	64.0	3.5	10.4	12.8
93	OK 04111	4111	11.1	0.9	5.1	80.4	7.4	95.0	31.0	0.2	0.7	0.3	37.9	850.0	37.8	64.0	3.5	10.8	23.9
93	OK 05526	5526	11.7	0.7	3.1	77.0	7.2	99.5	28.9	0.3	1.0	0.4	37.3	858.0	38.3	65.0	4.6	6.4	20.0

				Cree	p-Reco	very		Gluto	matic		Te	nsile			Baking		I	Mixograj	ph
Sample	Nur.	Abbr.	FP	J-Jr	SeP	RCY	SED	GI	WG	F	WE	RW	DE	LV	ViSc	BWA	CMT	MST	MTW
			(%)	( <b>Pa</b> <sup>-1</sup> )		(%)	(ml)	(%)	(%)	(N)	(N.cm)	(N.cm)		(cc)	(score)	(%)		(min)	(mm)
					_														
93	OK 05312	5312	9.8	2.5	0.2	73.8	7.0	79.2	27.8	0.1	0.3	0.1	40.8	750.0	36.2	63.0	2.6	8.1	13.5
93	OK 05511	5511	10.4	0.4	7.6	84.4	7.2	97.7	27.1	0.4	1.4	0.5	34.4	850.0	34.8	63.0	3.5	6.8	17.1
93	OK 05204	5204	10.6	0.6	3.9	82.1	7.6	99.4	26.9	0.3	1.0	0.4	37.6	808.0	34.3	64.0	4.2	3.8	15.8
93	OK 05212	5212	11.3	1.0	3.8	78.2	7.4	96.5	31.2	0.2	0.6	0.2	39.5	800.0	34.5	64.0	2.8	9.5	15.9
93	OK 05711W	5711	10.8	0.2	4.0	82.2	7.2	99.4	26.2	0.3	1.0	0.4	38.7	770.0	38.6	64.0	4.6	8.2	23.3
93	OK 06029C	6029	11.4	0.6	6.1	82.2	7.2	97.1	31.1	0.2	0.7	0.3	38.4	870.0	33.5	64.0	3.9	10.7	12.9
93	OK 06617	6617	12.6	0.8	2.4	79.6	7.0	93.5	34.9	0.2	0.7	0.3	38.9	825.0	38.3	66.0	4.3	7.8	19.4
93	OK 06618	6618	12.1	0.8	5.0	80.4	5.7	96.4	33.1	0.2	0.6	0.2	41.5	925.0	41.9	65.0	4.3	11.0	31.1
		Min <sup>a</sup>	9.8	0.2	0.1	73.8	5.5	67.2	23.3	0.1	0.3	0.1	27.3	700.0	5.0	63.0	2.4	0.6	10.8
		Max <sup>b</sup>	12.6	2.5	7.6	84.4	8.4	100.0	34.9	0.7	2.7	0.9	45.5	960.0	41.9	66.0	5.5	13.8	33.5
		Mean	10.7	0.7	3.5	80.3	6.9	96.3	28.1	0.3	1.1	0.4	37.7	822.6	19.3	64.1	4.2	6.3	18.1
		<b>SD</b> <sup>c</sup>	0.7	0.4	1.4	2.0	0.7	6.5	2.6	0.2	0.6	0.2	3.5	58.3	14.3	0.8	0.7	3.3	5.4

Table 2c. Mean values of 2009 wheat cultivars and breeder lines

 $Max^a = Maximun$ 

Min<sup>a</sup> = Minimum

SD<sup>c</sup> = Standard deviation

	Axes	PC1	PC2	1+2
Variables	PC (%)	28.4	24.8	53.2
Viscoelastic	J-Jr	46.0	0.0	46.0
	RCY	19.0	0.1	19.1
	SeP	24.6	4.1	28.7
Mixograph	CMT	58.1	4.8	62.9
	MST	29.7	27.0	56.7
	MTW	5.4	24.5	30.0
Sedimentation	SED	19.0	24.5	43.5
Tensile test	F	79.5	0.0	79.6
	WE	42.2	5.7	47.9
	RW	67.1	6.3	73.4
	DE	10.3	21.1	31.4
Daling much orting	<b>T X</b> 7	0.2	60.1	60.2
baking properties		0.2	00.1 41.7	00.5 47.1
	VISC	5.4	41.7	47.1
	BWA	0.1	07.0	0/./
Gluten index	GI	55.1	0.0	55.1
	WG	20.7	54.1	74.8
Protein Content	FP	0.0	80.2	80.2

Table 3. Explained variance (%) in PCA of 2008 wheat flours. Abbreviations defined in Appendix 1 and Table 1.

Variables	Axes	PC1	PC2	1+2
variables	PC (%)	25.8	19.1	44.9
Viscoelastic	J-Jr	61.3	31.1	92.4
	RCY	67.5	26.0	93.4
	SeP	68.0	25.5	93.5
Mizograph	OMT	1 /	6 1	7 4
wiixograpii		1.4	0.1	/.4 14.6
	MST	0.0	14.0	14.0
	MTW	0.1	0.0	0.1
Sedimentation	SED	10.5	22.3	32.8
Tensile test	F	40.9	53.9	94.8
	WE	39.3	55.6	94.8
	RW	36.7	53.2	89.9
	DE	22.1	22.2	44.3
<b>Baking properties</b>	LV	2.7	0.1	2.9
	ViSc	16.2	5.5	21.6
	BWA	15.0	8.1	23.2
	CI.	0.6	07	0.2
Gluten index	GI	8.0	0.7	9.2
	WG	35.3	0.0	35.3
Protein Content	FP	12.8	0.2	13.0

Table 4. Explained variance (%) in PCA of 2009 wheat flours (Table 2). Abbreviations defined in Appendix 1 and Table 1.

Variables	Axes	PC1	PC2	1+2
variables	PC (%)	45.4	31.6	77.0
Viscoelastic	J-Jr	54.2	28.3	82.5
Mixograph	CMT	76.0	0.5	76.4
Tensile test	F	66.3	13.3	79.6
Baking properties	LV	10.6	69.0	79.6
Protein Content	FP	20.0	62.4	82.5

Table 5. Explained variance (%) in PCA of selected variables after discarding redundant variables of 2008 wheat flours (Table 1). Abbreviations defined in Appendix 1 and Table 1.

<b>X</b> 7. <b>1</b> 1.	Axes	PC1	PC2	1+2
variables	PC (%)	31.2	20.1	51.3
Viscoelastic	RCY	83.2	5.8	89.0
	J-Jr	80.0	7.8	87.8
Miyograph	СМТ	17	20.8	41.6
Mixograph		1.7	59.8	41.0
	MIS 1	1.5	66.5	68.0
Tensile test	$\mathbf{F}$	13.8	26.5	40.3
Gluten index	GI	15.6	2.8	18.4
	WG	46.4	6.0	52.4
Protein Content	FP	7.6	5.3	12.9

Table 6. Explained variance (%) in PCA of selected variables after discarding redundant variables of 2009 wheat flours (Table 2). Abbreviations defined in Appendix 1 and Table 1.

	FP	LV	ViSc	BWA	CMT	MST	MTW	SED	J-Jr	RCY	SeP	F	WE	RW	DE	GI	WG
FP	1																
LV	0.62**	1															
ViSc	0.49**	0.49**	1														
BWA	0.68**	0.59**	0.47**	1													
CMT	0.33**		0.30**		1												
MST	0.45**	0.39**	0.21**	0.38**	-0.45**	1											
MTW	0.35**	0.35**	0.48**	0.31**			1										
SED	0.37**	0.42**	0.23*	0.49**	0.36**		0.39**	1									
J-Jr					-0.50**	0.33**			1								
RCY					0.27**				-0.40**	1							
SeP					0.35**				-0.43**	0.61**	1						
F					0.67**	-0.35**		0.37**	-0.58**	0.30**	0.32**	1					
WE			0.22*		0.50**		0.27**	0.43**	-0.37**			0.77**	1				
RW	-0.20*				0.49**	-0.42**		0.24*	-0.47**	0.25**	0.20*	0.80**	0.51**	1			
DE	-0.36**			-0.27**		-0.28**							-0.27**	0.64**	1		
GI			0.30**		0.49**	-0.45**	0.23*	0.27**	-0.38**	0.25**	0.42**	0.56**	0.30**	0.51**	0.32**	1	
WG	0.76**	0.45**	0.30**	0.54**		0.60**			0.20*			-0.39**		-0.53**	-0.45**		1

Table 7. Pearson's correlation coefficients of 2008 wheat cultivars and breeder lines. Abbreviations defined in Appendix 1 and Table 1.

\*Correlation is significant at  $\alpha = 0.05$  level.

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\*\*Correlation is significant at  $\alpha = 0.01$  level.

	FP	LV	ViSc	BWA	CMT	MST	MTW	J-Jr	RCY	SeP	SED	F	WE	RW	DE	GI	WG
FP	1																
LV	0.30**	1															
ViSc	0.27**	0.32**	1														
BWA	0.59**	0.36**	0.29**	1													
CMT				0.27**	1												
MST	0.36**	0.36**			-0.37**	1											
MTW			0.23*	0.24*	0.23*		1										
J-Jr		0.23*	-0.26**	-0.33**		0.22*		1									
RCY	0.25*		0.30**	0.41**				-0.99**	1								
SeP	-0.25*		-0.30**	-0.40**				0.98**	-0.99**	1							
SED		0.27**						0.53**	-0.43**	0.43**	1						
F												1					
WE												0.99**	1				
RW												0.99**	0.98**	1			
DE												-0.58**	-0.61**	-0.46**	1		
GI		-0.26**			-0.24*		-0.23*					0.23*		0.25*	0.06*	1	
WG			-0.23*	-0.21*				0.39**	-0.42**	0.42**		-0.30*	-0.27**	-0.28**		-0.35**	1

Table 8. Pearson's correlation coefficients of 2009 wheat cultivars and breeder lines. Abbreviations defined in Appendix 1 and Table 1.

\*Correlation is significant at  $\alpha = 0.05$  level.

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\*\*Correlation is significant at  $\alpha = 0.01$  level.



Total explained variance is 53.2%

Fig.1. Loading plot of first two principal components of 2008 set of 51 samples wheat flours. Abbreviations defined in Appendix 1 and Table 1.



Fig. 2. Loading plot of first two principal components of 2009 set of 51 samples wheat flours. Abbreviations defined in Appendix 1 and Table 1.



**Total explained variance is 53.2%** 

Fig. 3. Loading plot of first two principal components of selected variables after discarding redundant variables of 2008 set of 51 samples wheat flours. Abbreviations defined in Appendix 1 and Table 1.



Total explained variance is 51.3%

Fig. 4. Loading plot of first two principal components of selected variables after discarding redundant variables of 2009 set of 51 samples wheat flours. Abbreviations defined in Appendix 1 and Table 1.

## CHAPTER VI

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## CHAPTER VII

#### CONCLUSIONS

The objectives of this study were to discriminate variation of hard red winter wheat properties by various techniques. These techniques included 1) changing viscoelastic properties of gluten at various temperatures ranging from 25 to 55°C, 2) evaluating hard red winter wheat properties grown in 2008 and 2009 by using creeprecovery test of gluten at different shear stresses of 40 and 100 Pa, dough extensibility, farinograph and baking test, and 3) determination of two sets of hard red winter wheat properties by using gluten creep-recovery, extensibility and glutomatic tests compared with traditional measurements.

Hard and soft red winter wheat gluten samples were differentiated after they were exposed to temperature at 25 to 55°C by measuring with creep-recovery test. The hard red winter wheat of gluten was discriminated differently with soft red winter wheat gluten. Hard and soft red winter wheat glutens were separated according to temperatures. Glutens when exposed to temperature at 25 and 35°C were grouped together; while, temperature at 45 and 55°C clustered the gluten into another group. At 45 and 55°C, glutens were associated with the Separation time which illustrated an entanglement. The %recoverability was correlated with the gluten at 25 and 35°C.
The relationship among the creep-recovery rheological properties of gluten traditional empirical assessments of flour samples and baking performance was shown. The explanation of variance of all variables was 79.1%. The main contributors to the explained variance were delta compliance (J-Jr) and maximum resistance to extension (Rmax). The parameters obtained from creep-recovery test using 100 Pa, LV and FP explained the same percent variance when the parameters from extension test (Rmax, Emax and Area) take into account. However, the viscoelastic variables were independent from flour protein (FP) and loaf volume (LV).

The correlations between the viscoelastic properties and the other properties related to baking performance were obtained by evaluating the two set flour samples from year 2008 and 2009. The principal component analysis (PCA) were performed in each year and concluded that the main contributors were from creep and recovery test which are %Recoverability (RCY), Separation time (SeP), and Delta compliance (J-Jr), and tensile test which are Force (F), Recoverable work (RW), and Work extensibility (WE). The total variance improved after remove the shorter vectors and low contributors in both 2008 and 2009 year sample set. The creep-recovery and tensile test appear to be useful for breeding program.

## CHAPTER VIII

#### FUTURE RESEARCH

- According to the results from chapter III, the creep and recovery test at high temperature (25 to 55°C) showed a good differentiation on the viscoelasticity of gluten samples. It can be suggested that higher temperatures (65 to 95°C) are used to investigate the gluten quality in depth.
- The comparison between shear stress at 40 Pa and 100 Pa in creep and recovery test showed that 100 Pa was more correlated with the dough mixing properties than 40 Pa. This suggests that further study may be investigated the creep and recovery test with higher shear stress to optimize shear stress.
- Seven and six of commercial flour samples were analyzed in chapter III and IV. I suggested increasing the number of samples in order to validate the findings using small set of samples.
- Based on the environmental and genetic background factors, the more data from different crop years and varied genetic pool should be analyzed (chapter V) in order to formulate a better prediction.

APPENDIX 1

Tests	Abbr.	Units	Parameters			
	FP	%	Flour Protein			
Baking	LV	cm <sup>3</sup>	Volumes of baked loaf measured at 10 min			
	LH	mm	Height of baked loaves			
	PH	mm	Height of loaves after proofing			
	OSP	mm	Increase in height of loaves in the oven during baking			
	SV	cm <sup>3</sup> /g	Specific volume of baked loaves			
	ViSc	score	Visual Score			
	BWA	%	Baking Water Absorption			
Mixograph	CMT		Corrected Mixing Time			
	MST	min	Mixograph Stability			
	MTW	mm	Mixograph Tail Width			
Farinograph	WA	%	Water Absorption			
	DT	min	Development Time			
	ST	min	Stability Time			
	BT	min	Breakdown Time			
SDS						
Sedimentation	SED	ml	Sodium Dodecyl Sulfate (SDS) sedimentation volume			
Creep-recovery	J-Jr	Pa <sup>-1</sup>	Delta Compliance			
	RCY	%	% Elastic Recovery			
	Sep		Separation time			
	TCC	S	Time Constant Creep			
	TCR	S	Time Constant Recovery			
Extension	Rmax	Ν	Maximum resistance to extension			
	Emax	mm	Extensibility at maximum resistance			
	Area	N/mm	Work required to extend the dough to Rmax			
Glutomatic	GI	%	Gluten Index			
	WG	%	Wet Gluten			
Tensile test	F	Ν	Force			
	WE	N.cm	Work of Extensibility			
	RW	N.cm	Recoverable Work			
	DE		Degree of Elasticity			

Table 1. List of addreviations for parameters us
--

Samples	Temperature	SeP	J-Jr	RCY	TCC	TCR
	(°C)	<b>(s)</b>	( <b>Pa</b> <sup>-1</sup> )	(%)	<b>(s)</b>	<b>(s)</b>
A1	25	1.24	0.55	82.83	6.43	3.25
	35	5.67	0.73	78.56	7.41	2.37
	45	13.66	0.76	79.95	6.63	1.80
	55	9.36	0.97	73.20	7.76	1.23
A2	25	1.93	0.43	83.19	5.40	2.53
	35	1.60	0.46	81.49	6.14	2.57
	45	11.40	0.74	78.77	6.22	1.57
	55	6.80	1.11	70.15	9.36	1.35
A3	25	0.03	2.48	81.74	6.39	3.22
	35	0.04	1.38	71.79	6.47	2.66
	45	4.44	2.43	64.39	9.71	1.81
	55	5.36	2.93	57.79	12.43	1.48
<b>B1</b>	25	2.24	0.32	85.30	4.03	1.98
	35	2.04	0.29	85.96	3.92	1.98
	45	11.20	0.40	83.04	4.05	1.23
	55	5.16	0.65	73.11	6.26	0.90
<b>B2</b>	25	2.75	0.28	82.65	5.32	2.32
	35	8.95	0.37	81.42	5.28	1.69
	45	9.56	0.56	75.00	7.49	1.43
	55	11.00	0.65	71.11	8.72	0.92
<b>B3</b>	25	2.24	0.33	83.95	4.99	2.33
	35	2.65	0.38	81.46	5.09	1.99
	45	11.20	0.51	80.32	5.28	1.38
	55	4.24	0.80	71.43	7.66	1.16
Stephens	25	0.05	2.59	72.61	15.17	6.92
	35	0.05	3.12	70.81	14.04	5.91
	45	0.83	4.46	64.92	15.05	3.86
	55	0.40	5.95	58.09	16.99	2.98
	Average	4.86	1.31	75.89	7.85	2.31
	SD	4.28	1.41	7.87	3.63	1.38
	Min.	0.03	0.28	57.79	3.92	0.90
	Max.	13.66	5.95	85.96	16.99	6.92

 Table 2. Mean values of viscoelastic properties of gluten samples in each temperature in Chapter III



Fig. 1. The schematic of tensile test on gluten from initial to final state. The calculation of final to initial length ratio is shown below.

The initial length of gluten = 5-1.3-1.3 = 2.4 cm

The length of gluten from initial to final = 5+6.35 = 11.35 cm

The length of gluten after gluten was stretched = 11.35-1.3-1.3 = 8.75 cm

Therefore, the ratio of the length of gluten after gluten was stretched and the initial length

of gluten = 8.75/2.4 = 3.65 times

## VITA

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## Thesis: ASSESSMENT OF VARIATION BASED ON TEMPERATURE, CREEP-RECOVERY, EXTENSIBILITY TESTS, AND GLUTEN CONTENT IN HARD RED WINTER WHEAT COMPARED WITH TRADITIONAL WHEAT QUALITY TESTING

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# Title of Study: ASSESSMENT OF VARIATION BASED ON TEMPERATURE, CREEP-RECOVERY, EXTENSIBILITY TESTS, AND GLUTEN CONTENT IN HARD RED WINTER WHEAT COMPARED WITH TRADITIONAL WHEAT QUALITY TESTING

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**Scope and Method of Study:** The overall purpose of this study was to explore the potential improvement of differentiation of the properties of wheat samples by the use of methodology not used at the present time in breeding programs and in quality control protocols in the milling and baking industry. Gluten analyzed by a creep-recovery method at 25, 35, 45 and 55°C was used in six hard red wheat and one soft red winter wheat samples. Creep-recovery plus extensibility of gluten and gluten content were compared with mixing, baking and protein sedimentation parameters to assess the improvement in explained variance using Principal Component Analysis (PCA). Two stresses (40 and 100 Pa) were used to determine potential improvement in the ability to separate gluten properties. Commercial (6) and breeder hard red winter flour samples (102) were analyzed with creep recovery at 100 Pa (25°C) along with the extensibility, gluten content and traditional wheat breeder quality tests.

Findings and Conclusions: PCA demonstrated that the viscoelastic properties of hard and soft red winter wheat flours are separated into two distinct groups with gluten analyzed 45 and 55°C associated with separation time (SeP) suggesting the presence of more entanglements compared to those at 25 and 35°C. At 25 and 35°C, the gluten samples were associated with %Recoverability (RCY) suggesting more elastic behavior (stiffer glutens) compared to glutens at 45 and 55°C. Creep-recovery tests at 100 Pa were able to separate better the viscoelastic behavior of gluten compared to 40 Pa. The main contributors to the highest percent of explained variance (83.8%) were creep recovery parameters obtained at 100 Pa (first principal component), loaf volume (LV) and flour protein (FP) (associated with the second principal component). When the traditional methods and the three suggested methods (creep-recovery, extensibility test, and gluten content) were analyzed together, the PCA for the breeder samples sets for 2008 and 2009 had similar total explained variance. The parameters with the highest contribution to the variance were selected for a new PCA and an improvement of the total explained variance was demonstrated (77.0% for 2008 and 51.3% for 2009). In summary, creeprecovery and tensile test parameters improved the discrimination of wheat samples and they were independent of baking properties.

ADVISER'S APPROVAL: Dr. Patricia Rayas Duarte