DELAYED HARVEST AFFECTS DURUM WHEAT QUALITY

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MASTER OF SCIENCE

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ABSTRACT

Harvest can be delayed for many reasons. This research was conducted to determine the effect of delayed harvest on grain and semolina quality of durum wheat (*Triticum turgidum* L. ssp. *durum* [Desf.] Husn.). Twelve durum cultivars were planted in eight-row plots with four replicates at three environments. Two rows were harvested at four harvest times that were spaced about one week apart. The trend for all cultivars was for an increase in percent of large kernels, kernel brightness, 1000-kernel weight, and semolina gluten index with delayed harvest, while grain yield, test weight, kernel vitreousness, falling number, grain yellow pigment, and semolina yellowness and wet gluten content generally decreased. At all environments 'Carpio' tended to have high yields and 'Strongfield' low yields. 'Carpio' and 'Joppa' had the highest yellow pigment content and very strong gluten. In conclusion, grain and semolina quality generally declined with delayed harvest but varied with cultivar.

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INTRODUCTION

Harvest can be delayed because of inclement weather, breakdown of harvest machinery, too many acres to harvest and/or other crops needing to be harvested. Delayed harvest prolongs the exposure of the grain to the environment. Abiotic (e.g. moisture, temperature, and wind) and biotic (eg. birds, disease and insects) stressors can cause decline in agronomic and end-use qualities. Wind can cause lodging which is the bending of the stem from a vertical to a more horizontal position. Lodging promotes disease infection due to the dampness related to being near the soil surface. Lodged plants are difficult to harvest. Wind can contribute to shattering of grain from the spike, which would reduce grain yield. Moisture can cause decline in grain quality. Moisture can cause bleaching/weathering of grain and premature germination of grain in the spike. Durum wheat (*Triticum turgidum* L. ssp. *durum* [Desf.] Husn.) is susceptible to weathering. Weather-damage in wheat kernels can be caused by heavy morning dew or rainfall that occurs after the grain in the spike has dried enough for harvest. Under warm/wet cycles, saprophytic fungi have the ideal conditions to colonize wheat heads. In general, Fusarium head blight or scab (caused by *Fusarium graminearum* Schwabe) and black point (caused by *Alternanthera* spp.) could result in low test weights and poor grain quality (Paul and Lindsey, 2014). Damage to wheat kernels becomes more pronounced with delayed harvest.

In North Dakota, durum is typically harvested in August and September. The relative humidity is generally low in August. In September, it is not unusual for dew to form on plant surfaces at night making the plant including the spike to remain wet through mid-morning. Water from dew is reabsorbed by the grain causing the bran layer to swell. As the seed dries, the bran layer does not contract resulting in an increase in seed size and in the seed appearing to be weathered or bleached. Once dry, the bran layer becomes wrinkled and friable (Clarke et al.,

1986; Bason et al., 1995; Debbouz et al., 1995), which makes bran removal during roller milling difficult and often results in high speck count and ash content in semolina (Debbouz et al., 1995). The increase in seed size without an increase in weight results in a reduction in test weight, which is measured as kg/hL.

Loss of grain quality as a consequence of delayed harvest is due to decreases in test weight (Pool et al., 1958; Gan et al., 2000), 1000-kernel weight, grain hardness (Johnson, 1959), and vitreousness. Test weight is the most common quality criteria associated with delayed harvest. Test weight is determined by packing efficiency, which varies with cultivar and seed weight and density, which depends primarily on the environment (Yamazaki and Briggle, 1969). High test weight is related to high semolina or flour extraction (Matsuo and Dexter, 1980). Test weight can be reduced due to the increase in kernel size, due to expansion of bran layer, without an increase in weight. Test weight is a grading factor and when test weight falls below a critical values results in decreased grade and results in economic penalties to the growers.

Bleaching corresponds to seed coat discoloration. Initially, bleaching tends to cause a lightening of the seed coat appearance but as bleaching progresses, the seed takes on brown dull weathered appearance (Debbouz et al., 1995). Bleached kernels are not considered a grading factor in durum. However, prolonged exposure of kernels to damp conditions can result in moisture moving into the endosperm causing it to fracture and appear non-vitreous. These kernels are often classified as non-vitreous during the grading process. Durum grade has three subclasses that are determined by percentage vitreous kernels: Hard Amber Durum (>75%), Amber Durum (74-60%) and Durum (<60%). Bleached kernels have been associated with several undesirable quality factors, such as low test weight, semolina extraction, and falling number (Debbouz et al., 1995).

Falling number refers to the time it takes a plunger to drop a prescribed distance through a starch gel. Low falling number is associated with pre-harvest germination of grain in the spike, which is triggered by damp conditions before harvest. α-Amylase activity increases during germination. α-Amylase activity also can come from microorganisms found on or in the grain, which release enzymes that degrade starch, protein, and lipids. Another enzymatic change associated with bleaching, but not extensively studied, are the activities of polyphenol oxidase (PPO) and other oxidative enzymes, which are largely affected by environment (Baik et al., 1994). Polyphenol oxidase (PPO) is involved in brown pigment formation that diminishes the appearance of semolina and pasta (Demeke, 2001; Verlotta, 2010).

Durum wheat genotypes are known to differ in their susceptibility to abiotic and biotic stresses (Mohammadi et al., 2015). Grain quality differs among cultivars mainly because cultivars have different rates to reabsorb water, drying rate, rate of sugars use, and embryo dormancy, besides of spike structure and plant structure. Sandhu et al. (2009) reported that durum cultivars commonly grown in the Northern Great Plains, USA differed in their tolerance to kernel bleaching. They reported that the cultivar Ben was particularly susceptible to bleaching and loss of vitreousness. Anecdotal evidence indicates that the cultivar Grenora is more susceptible to bleaching than is Divide. Reduction in durum grain quality is a concern for durum producers since grain price is strongly related to grain quality.

Specific Objective

The current research was carried out to determine the response of cultivars to the effect of delayed harvest on grain and semolina quality of durum wheat.

LITERATURE REVIEW

Background

Durum wheat is a tetraploid species 2n=4x=28 belonging to the Poaceae family. The inflorescence is a spike (Figure 1). The spike has a rachis with spikelets. Each spikelet consists of two glumes with two to five florets. Each floret is a perfect flower enclosed by two structures the lemma and palea. Every floret is able to produce one seed (caryopsis) (Bozzini, 1998). The seed size and weight depends on the cultivar; however, the average size is ~8 mm in length and 3-5 mm wide and weigh 35-40 mg (2016 regional crop survey for the weight). The kernel can be oval, spherical to long, and narrow to flatten in shape with ~ 38 mg of weight (Mattern, 1991; Delcour and Hoseney, 2010). The durum kernel has a translucent amber color with high density, vitreous appearance, being the hardest wheat kernel.

Figure 1. Durum wheat spike morphology.

Grain Development

Five main phases of wheat seed development have been described: fertilization (0 days post-anthesis, DPA), endosperm formation (1–5 DPA), cellularization, and early grain-filling (6–13 DPA), maximum grain filling (14–24 DPA), and desiccation (25–38 DPA) (Simmonds and O'Brien, 1981; Yu et al., 2016).

Pollination and Fertilization

Pollination is the deposition of the pollen onto the stigma. Wheat is considered to be a self-pollinated crop, although there is 1 to 2% cross-fertilization (Sleeper and Poehlman, 2006). It is assumed that the anthers shed the pollen onto the stigma of the same floret (Evers and Millart, 2001).

Pollen is formed in the anthers. To capture pollen, stigmas use adhesive interactions to retain pollen grains. When the pollen is on the stigma, a pollen tube develops and two pollen grains (sperm) move down into the style and enter the ovary specifically to the embryo sac. Once in the embryo sac, one of the sperm (1N) fuses with the egg nuclei (1N) forming the zygote or embryo (2N). The second sperm fuses with two polar nuclei (1N each) forming the endosperm (3N) (Evers and Millart, 2001). The entire process is called double fertilization. The pericarp is maternal tissue that develops from integuments that surround the ovary and represents the majority of the bran that is removed during roller milling.

The endosperm develops through repeated divisions of the primary endosperm nucleus (Olsen, 2004). Next, the endosperm undergoes cellularization, where cell walls are formed starting from the micropylar area (Evers and Millart, 2001). Cellularization is complete in about four days after pollination.

A stress during pollination and fertilization might cause a reduction on the number of kernels per spike that means a decline on yield. Stress during cellularization can cause a reduction in the number of cells in the endosperm and subsequently affect kernel size (Pignocchi et al., 2009).

Grain Filling and Physiological Maturity

After fertilization, grain filling occurs and continues until physiological maturity. Throughout grain filling, the grain moisture declines from about 65 to 35% which is the typical moisture content of kernel at physiological maturity (Sofield et al., 1977). During grain filling, the kernel undergoes six developmental stages: watery stage, milk stage, soft dough stage, hard dough stage, kernel hard stage, and harvest ripe (Anderson et al., 1985).

There are three physiological processes involved in determining the grain weight from grain filling to maturity: grain dry matter accumulation, desiccation, and grain morphological expansion, all of them occur simultaneously (Xie et al., 2015). Grain dry matter accumulation is a consequence of the deposition of the starch (around 70% of mature grain weight), protein accumulation (~13% of mature grain weight), and minerals, vitamins and fibers (Shewry, 2009). Almost all carbohydrate assimilates come from photosynthesis and storage photosynthates (Schnyder, 1993). When grain water accumulation is reached, there is a rapid loss of moisture during desiccation (Lizana et al., 2010). Water is used to transport photo-assimilates and other nutrients into developing grains and for metabolic synthesis of storage products. A strong association between maximum grain water content and final grain weight has been found in wheat (Lizana et al., 2010; Hasan et al., 2011).

The morphological changes are associated with grain length, width, height, and volume. Maximum grain length is reached ~15 days DPA. Maximum grain width, height, and volume

occur by ~28 DPA (Lizana et al., 2010; Hasan et al., 2011), which corresponds to the period of endosperm cell enlargement (Briarty et al., 1979). Li et al. (2012) observed that at 3 DPA, the pericarp was the major site of starch deposited. At 6 DPA the number and size of starch granules in the endosperm increased.

Physiological maturity occurs at the end of the hard dough stage (Sofield et al 1977). At physiological maturity, the nutrients stop moving into the kernel (Schnyder and Baum, 1992; Calderini et al., 2000). The loss of water from the kernel causes a size reduction, which is consequence of the connective protein network of the endosperm, which contracts during desiccation eliminating open spaces resulting in a vitreous kernel (Anjum and Walker, 1991). After physiological maturity, the kernel continues to desiccate until 13 to 12% moisture when it is ready to harvest. In the northern areas of US, the moisture content of harvested grain can range from 12 to 16%. At 16% moisture content, the grain must be dried to 13% before storage. During desiccation, the grain loses it's green color and the dimensions of grain decrease due to contraction of bran layer and protein found in the endosperm (Lizana et al., 2010).

Kernel maturity varies within a spike. Kernels from the middle of a spike are larger and mature before kernels found in the distal portion of the spike. Distal grains have fewer endosperm cells (Gao et al., 1992) and a slower rate and shorter duration of grain filling, with lower grain water content, which results in smaller grain dimensions (Lizana et al., 2010). The determination of physiological maturity could be critical under some circumstances, due to the increased risks of yield penalties during desiccation, from abiotic (temperature and moisture) and biotic stresses (disease and insect) (Calderini et al., 2000). The duration of the grain filling and grain desiccation stages are influenced by the temperature, for example cool air temperature promotes long period of grain fill and desiccation and large heavy grain (Gebeyehou et al.,

1982). Warm temperatures promote shorter periods of grain fill and lower grain weight (Royo et al., 2014). An increase of one Celsius degree from 17 to 24°C during grain filling can reduce the yield four percent and reduce weight of the grain; even if there is available water from irrigation (Al-Karaki, 2012). High temperatures up to 30°C during grain filling mainly affect the deposition of starch (Hoseney, 2003). High temperatures have also been reported to increase the gliadin content and subsequently increase the gliadin:glutenin ratio, which results in diminished gluten/dough properties and soft pasta/noodles products (Blumenthal et al., 1993).

Major Developmental Structures of the Kernel

The major parts of durum kernel are endosperm, bran and the germ. Most of the kernel consists of endosperm that is hard, yellow, and starchy. The endosperm is surrounded by bran. Germ is exposed to the environment and is located at the apex of the seed and close to the base to the floret.

The germ is composed of a single cotyledon (the scutellum), embryonic axis (coleoptile, primary root, and secondary roots), and epiblast (Delcour and Hoseney, 2010) The germ/embryo is the smallest part of the kernel (2-3 %) and is rich in lipids (8-13%), proteins (25%) and sugars (18%) along with a small content of ash (5%) and vitamins (B and E). The germ does not contain starch (Mattern, 1991).

The bran is composed of the seed coat, pericarp, and aleurone layer, representing 14.5% of kernel dry weight. The bran is rich in complex non-starch polysaccharides (dietary fiber), essential fatty acids, protein, minerals, and vitamins. The pericarp surrounds the entire seed and is composed of an inner and outer part. It corresponds to 5% of the kernel dry weight with 6% protein, 2% ash, 20% cellulose, and 0.5% fat. Pericarp is important to the protection of the seed. Seed coat has the pigment layer. Pericarp and seed coat are composed of dead empty cells.

Aleurone layer, even though is often associated with the bran, is actually a component of the endosperm (non-starchy part). It is rich in proteins with high enzymatic activity, lipids, and ash. The protein and enzymes play a vital role in the germination process. The germ, all the components of bran and most of the aleurone layer are removed during milling (Šramková et al., 2009; Delcour and Hoseney, 2010).

The starchy endosperm represents the 70% of the dry kernel weight (Mattern, 1991). The endosperm formation starts during grain filling. Wheat endosperm contains three types of starch granules: A-type (> 10 μm), B-type (5–10 μm), and C-type (< 5 μm). The endosperm contains mainly starch; however, it also contains 13 to 15% protein that consists of glutenins and gliadins and small fractions of albumins and globulins (Hurkman and Tanaka, 2007). The endosperm also contains small portions of fats (1.5 %), ash (0.5 %), and dietary fiber (1.5 %) (Belderok et al., 2000).

Kernel Storage Material

Starch

Starch constitutes 60 to 75% of the dry weight of the wheat kernel (Hoseney, 1986; Šramková et al., 2009) and is the main source of energy for the developing embryo. Starch is found as a granule. There are three types of starch granules, A-type, B-type, and C-type. However, A- type and B-type are the most important, because they represent nearly 100% of the starch in mature wheat grains. Starch granules are classified according to the shape and size: A-type, the lenticular and large (> 10 μm), which are formed within the first 15 days after pollination, and B- type, the spherical small granules (5-10 μm), formed after first 15 days up to 30 days after pollination (Wilson et al., 2006; Yu et al., 2016). The small granules represent 88% of the total number of granules (Belderok et al 2000).

Starch is composed of two glucose polymers: amylose and amylopectin. Amylose consists of ~1,500 glucose molecules with a molecular weight of ~250,000 (Collao and Corke, 2003; Šramková et al., 2009; Serna-Saldivar, 2010). Amylose is primarily a linear polymer of α-1,4 linkages of α-D- glucopyranosyl units; however, it does have some branches of α-1,6 linkages. The axial position of α-1,4 linkages results in a right-handed spiral or helical shape. The interior of the helix has hydrogen atoms associated with glucose, making it lipophilic, while the exterior has hydroxyl groups, making it hydrophilic. Amylose represents ~25% of the starch granule (Delcour and Hoseney, 2010). Amylopectin is highly branched, with a linear chain of α-1,4-linkages and α-1,6 branches (Myers et al., 2000). It has a molecular weight of 10^8. Durum wheat starch granules typically contain 73 to 74% amylopectin (Leloup et al., 1991).

Protein

The protein content of wheat kernels may vary between 10 to 16%, depending on cultivar, environmental conditions and production practices during growth. Protein is important for nutrition as well as functionality. There are four types of proteins classified according to their solubility based on Osborne fractionation: albumins, globulins, prolamins, and glutelins. Albumins are soluble in water; globulins are insoluble in pure water but soluble in dilute low concentration of NaCl solutions. Prolamins are soluble in 70% ethanol, and glutelins are soluble in acid and base solutions (Delcour and Hoseney, 2010).

Albumin and globulins are important nutritional proteins. They have very good amino acid balance, such as, lysine, tryptophan, and methionine (Žilić et al., 2011). They constitute 25% of the total grain protein. They are concentrated in the aleurone cells, bran and germ (Delcour and Hoseney, 2010). These non-gluten forming proteins can be found in small percentage in the endosperm, having an important role in cellular metabolism, development, and

response to environment (Hurkman and Tanaka, 2007). Albumins are the smallest wheat proteins, followed by globulins. Albumins and globulins accumulate in the kernel up to 20 DPA, after which their content remains at constant level (Panozzo et al., 2001).

Gliadins (prolamins) and glutenins (glutelins) are storage proteins. Storage proteins are located in membrane-bound spherical bodies, which are derived from endoplasmic reticulum and Golgi apparatus (Kim et al., 1988). Storage proteins accumulate from 6 DPA to physiological maturity (Panozzo et al., 2001). It has been reported that gliadins accumulate earlier than glutenins (Stone and Nicolas, 1996; Panozzo et al., 2001).

The key products to form gluten are gliadins and glutenin subunits. They represent about 75% of the total protein content. They are responsible for the viscoelastic properties of the dough (Sissons et al., 2007). Gliadins are monomeric globular proteins with intramolecular disulfide bonds responsible for dough plasticity (D'Edigio, 2001). Glutenins are polymeric proteins with intermolecular disulfide bonds that form large complex that are responsible for-the strong elastic properties of dough; they can be low or high molecular weight (HMW-GS and LMW-GS) (D'Edigio, 2001). Storage protein in wheat grain contains at least 100 different protein subunits (D'Edigio, 2001).

The basic components that form gluten are water, gliadins and glutenins, and energy. The quality of pasta products are directly related to gluten formation. Gluten can be affected by variation in gliadin/glutenin ratio, which affect the viscoelastic properties of the dough (Sissons, 2008). The ratio of monomeric/polymeric proteins are mainly controlled by genotype; however, it can be modified by the environmental conditions during growing season (D'Edigio, 2001; Alternbach et al., 2003). Panozzo et al. (2001) identified changes in the protein composition in grain samples exposed above 30°C; dough made from this protein often rapidly breaks down.

Effect of Environment During Harvest on Grain Quality

Durum requires special environmental conditions to grow properly. It is best adapted to a semi-arid climate, with hot, long days and cool nights as found in the Mediterranean regions. Dry conditions during harvest are important for good grain quality. For a good yield, the optimum temperatures during grain fill are between 18 to 24°C. Temperatures between 28 to 32°C for short periods can cause 20% or more yield losses (Stone and Nicholas, 1994). North Dakota fulfills the climate requirements to grow durum wheat.

In North Dakota, durum is sown between mid-April and May and harvested between August and September. The total period to grow durum is from 90 to 120 days. As mentioned above, high temperatures during grain filling have been identified as one of the major source of variation of dough properties (Panozzo et al., 2001). However, the growth environment does not only affect the protein and starch deposition, but it also can affect grain quality parameters, such as, test weight, grain moisture, kernel size, kernel weight, and kernel vitreousness, (Edwards et al., 1989).

Delayed Harvest

Harvest can be delayed because of inclement weather, breakdown of harvest machinery, too many acres to harvest and/or to other crops needing to be harvested. Delayed harvest due to inclement weather can result in loss of grain quality and grade. The damage associated to delayed harvest can be due to abiotic (wind, rainfall) and biotic stressors (birds, diseases, insects). Adverse weather before harvest can delay harvest and affect the grain yield, and grain and semolina quality. The main consequences of yield reduction are due to hail damage, lodging and shattering (Ferrer et al., 2006). Both lodging and shattering can be consequence of high winds before harvest. Lodging has been reported to be the most limiting factor preventing

increased wheat yields (Rajkumara, 2008). Lodging ten days after heading can cause yield losses up to 40% (Kelbert et al., 2004). The main consequences of lodging are rain, strong winds, and hail especially during the last period of the crop growth (Rawson and Macpherson, 2000).

Reduction in grain quality is mainly associated with low test weight, lack of vitreousness (translucency of the kernel), and pre-harvest germination of grain in the spike. Pre-harvest germination of grain results in increased enzymatic activity, and decreased ratio of protein to starch (Edwards et al., 1989). Also, Pool et al. (1958) and Johnson (1959) reported a reduction in the grain hardness and kernel weight. Gan et al. (2000) showed that delayed harvest in spring wheat reduced test weight up to 50 kg m^{-3}. Damp conditions associated with heavy morning dew might not cause sprouting, but can cause bleaching of the seed coat (Czanecki and Evans 1986).

Biotic stressors, such as, birds or saprophytic fungi, can be also consequence of quality losses during delayed harvest. Birds eat wheat kernels resulting in yield reduction. Fusarium Head Blight (FHB) or black point are some examples of seed diseases. Fusarium Head Blight caused by *Fusarium graminearum* Schwabe is a problematic disease on durum wheat. The damage of this fungus is dependable of the growth stage and weather at that moment (Stack et al., 2002). When the fungus colonized the spikes a black mold is formed on the spike surfaces, usually that not affect grain quality. However, the problem occurs when the pathogen starts to infect the grain, which results in lower test weight and loss of grain quality. In durum wheat, the pathogen-produces a toxin called deoxynivalenol (DON) which ~50% is retained in semolina after milling and has an adverse effect on pasta color (Schisler et al., 2002). The presence of DON has to be less than 1 ppm in food ingredients (Paul and Lindsey, 1994). Kernel black point is caused by different pathogens some of them are *Alternanthera* spp. and *Helminthosporium* spp. Blackpoint pathogens are associated with a dark brown discoloration, and *Fusarium* spp. are

associated with a reddish color. Durum is susceptible to black point infection and serious infections can result in a discoloration throughout the endosperm causing loss of semolina color and an increase of semolina specks (Dexter and Matsuo, 1982). Under dry/wet cycles these fungi have an ideal environment to colonize the spikes even when grain moisture has dropped to the level needed for harvest. The consequence of all these are anomalies in the appearance of the kernels and end use pasta products.

Grain Quality Factors Affected by Delayed Harvest

Test Weight

One of the oldest criteria to classify the quality of the grain is test weight, which is the weight per unit volume (Pushman and Bingham, 1975). It indicates the weight of grain to fill a volume under standard conditions of packing, and is a function of density and packing characteristics of the grain (Hlynka and Bushuk, 1959). A high test weight generally is associated with high semolina extraction during milling and high grain protein content (Watson et al., 1977). Loss in test weight is associated with delayed harvest due to changes in the shape of the grain and seed coat roughening during wet and dry periods which reduces grain packaging efficiency, and decreases density and the mass of the kernels (Czarnecki and Evans, 1986). Low test weight results in a decrease in the US grade scale and subsequently a reduction in value.

Kernel Vitreousness

Vitreous kernels have a glassy, translucent appearance. They have no air spaces between the starch and protein matrix, which allows light to pass directly through the kernel. Vitreousness is associated with grain hardness and with high levels of protein content (Oury et al., 2015). It is affected by genotype, environment, and their interaction (Phillips and Niernberger 1976). Choice milling durum represents the highest quality durum and grades US No. 1 and has at least 90%

vitreous kernel content and a minimum of 13% protein. Sub-classification of durum wheat is based on kernel vitreousness. Subclasses are Hard Amber Durum (HAD) which has ≥ 75% vitreous kernels; Amber Durum (AD) 60 to 74% vitreous kernels; and Durum <60% vitreous kernels.

Vitreous kernels are associated with fracturing of endosperm during milling which results in a coarse semolina granulation, high extraction yield, and enhanced yellow color (Matsuo and Dexter, 1980; Neethirajan et al., 2006). Non-vitreous kernels have less protein content, are opaque, starchy, and softer because there is less adhesion between starch granules and protein which results in air spaces in the endosperm that prevents the penetration of light. This kind of grain decreases considerably the yield of semolina and increases the amount of flour produced during milling. Flour is a considered a by-product of durum milling (Dowell, 2000).

The most common method used to determine vitreous kernels is performed through visual analysis by an inspector. Visual inspection is subjective and can result in misclassification of grain (Dowell, 2000; Neethirajan et al., 2006). An inspector might not observe a small chalky spot, or a shadowy spot which will cause a kernel to be incorrectly classified as vitreous. Non-vitreous kernels have a negative economic impact to the producer, decreasing the final price and in extreme cases, can result in rejection.

Bleaching

Bleaching is a change in the color of the seed coat associated with loss of vitreousness (Lovell, 2013). Bleaching is the result of changes in the reflective properties of the kernel caused by cracks in the seed coat structure under periods of wet/dry conditions before harvest (Bason et al., 1995). As moisture increases, fissures form in the endosperm changing the appearance of the kernel from translucent amber color to a chalky white spot which can cause a kernel to be

classified as non-vitreous (Dowell, 2000; Neethirajan et al., 2006). As the moisture in the bran decreases, the bran layer does not contract causing the bran to become quite friable and becomes difficult to remove during milling and often results in bran contamination in the semolina. Bleached kernels do not have elevated enzymatic activity because pre-harvest germination has not been activated.

Pre-harvest Sprouting

Pre-harvest sprouting is defined as premature germination of kernels in the spike. Pre-harvest sprouting results from a break in dormancy of seeds under wet conditions that can occur during delayed harvest, but also it is associated with warm environmental conditions and cultural practices such as swathing (Gelin et al., 2006). Some of the consequences related to pre-harvest sprouting are decreased grain weight (Groos et al., 2002) and increased hydrolytic enzymes α-amylase, protease, and lipase that degrade starch, proteins, and lipids, respectively (Kruger, 1976; Debbouz et al., 1995). However, the degree of response depends on the level of germination and the susceptibility of the cultivar (Skerrit and Heywood, 2000).

The mechanism typically used to determine pre-harvest sprouting is the falling number test (AACC International Approved Method 56-81.03). In the falling number test, starch gelatinizes in boiling water and forms a gel. The time for a plunger to pass through the gel is recorded in seconds. If α-amylase is present, it breaks down the gel and the plunger drops quickly through the degraded gel. Falling number is basically an indirect measure the α-amylase activity registered in seconds, a high α-amylose activity means starch damage and a low falling number.

Environmental Activation of Oxidative Enzymes

As mentioned above, hydrolytic enzymes can be activated by damp conditions at harvest time. Besides hydrolytic enzymes, there are other enzymes, which can be affected by the environment and cause a decrease in end-use product quality. These enzymes include polyphenol oxidase (PPO) and lipoxygenase (LOX).

Polyphenol Oxidase

Polyphenol oxidases (PPOs) are ubiquitous Cu-containing enzymes that are found in many different plants, including durum wheat. PPOs catalyze the hydroxylation of *o*-monophenols to *o*-diphenols and the oxidation of *o*-dihydroxyphenols to *o*-quinones (van Gelder et al., 1997). The products of this oxidation react with some thiols, amines and phenolic acids as ferulic acid and sinapic acid, which play an important role as substrates, forming melanins that are colored products responsible of brown discoloration. The activities of these enzymes varies according to pH and are optimum in plants with a pH range between 4.5-8.0 (Kavrayan and Aydemir, 2001). Different wheat cultivars present different levels of PPO activity (Demeke et al., 2001).

In immature seeds, most PPO activity occurs in the endosperm, whereas when the kernel ripens, PPO activity is mostly located in the embryo and outer layer of the kernel bran, with decreasing activity in the endosperm (Marsh and Gilliard, 1986). During the milling process, PPOs are removed almost totally. Although, when a portion of PPOs remain, they can cause serious problems in the color and quality of semolina pasta products (Rani et al., 2001; Demeke, 2001; Verlotta, 2010). PPO can cause the formation of brown pigment that can mask the yellow appearance in pasta products. These oxidative enzymes could be related with delayed harvest anomalies, because their activity is largely affected by environment (Baik et al., 1994).

Lipoxygenase

Lipoxygenase is a group of enzymes located mainly in the embryo, followed by bran and endosperm (Rani et al., 2001). They are also known as lipoxidase and carotene oxidase. They catalyze the oxidation of polyunsaturated fatty acids containing a *cis*-*cis*-1, 4-pentadiene moieties (Troccoli et al., 2000). These enzymes have a role in plant development and senescence, and pest resistance (Hildebrand, 1989). Lipoxygenase activity in durum wheat depends on genotype and on the environment (Troccoli et al., 2000).

Yellow Pigment Content

The bright yellow pasta color is highly demanded by buyers and durum wheat industry (Troccoli et al., 2000). The yellow color in durum endosperm is caused primarily by carotenoids. People consider bright yellow color of semolina as an indicator of high nutritional value. Yellow pigments in durum are affected by cultivar characteristics (Borrelli et al., 2003) and environmental conditions at the different stages of plant development (McCaig et al., 2006). Taghouti et al. (2010) indicated that variation in yellow pigment content was due more to genotype effect than to the environment.

Lipoxygenase has a role in durum wheat yellow color. Lipoxygenase indirectly causes the oxidation of α-tocopherol and carotenoid pigments, resulting in the bleaching of semolina (Sissons, 2008). Lipoxygenase oxidizes unsaturated free fatty acids to form hydroperoxides through a free radical mechanism. Free radicals are quenched by carotenoid pigments which results in loss of color. Yemenicioglu and Ercan (1999) reported that LOX activity can be inactivated during processing by temperatures above 65°C.

MATERIALS AND METHODS

Plant Material

Experiments were established at the ND Agricultural Research Stations located near Langdon, ND in 2014 and near Prosper, ND in 2014 and 2015. The soil type at Prosper was a Parilla loam and the soil type at Langdon was a Svea-Barnes loam. Soil was fertilized based on soil tests with nitrogen for a 4.4 MT/ha yield goal at Prosper in 2014 and 2015. At Langdon 2014, the soil fertilization base was N-P-K-S-Cl: 85-39-30-20-25 plus soybean N 33.6 kg/ha.

Twelve cultivars were sown at 80 g/plot in eight-3 m rows that were spaced 0.3 m apart. Cultivars can be classified into three groups: old cultivars including 'Ben' (Elias and Miller, 1998), 'Mountrail' (Elias and Miller, 2000) 'Lebsock' (Elias et al., 2001),'Dilse' (Elias et al., 2004a), and 'Pierce' (Elias et al., 2004b); current cultivars including 'Strongfield' (Clarke et al., 2005), Alkabo'(Elias and Manthey, 2007a), 'Divide' (Elias and Manthey, 2007b), and 'Grenora' (Elias and Manthey, 2007c); and new/recently released cultivars including 'Tioga' (Elias and Manthey, 2013), 'Carpio' (Elias et al., 2014),' and 'Joppa' (Elias and Manthey, 2016).

The establishment at Prosper was on May 27, 2014 and April 23, 2015 while at Langdon was on May 15, 2014. The herbicide, Huskie Complete (a.i. Thiencarbazone-methyl, Pyrasulfotole, and Bromoxynil), was applied on June 13, 2014 and June 6, 2015 at Prosper to control grass and broadleaf weeds. Caramba fungicide (a.i. Metconazole) was applied on July 1, 2015 to control leaf diseases. Weather data was obtained from the weather station located at the Langdon and Prosper experiment stations (NDAWN).

Two rows were harvested at each harvest time. Harvest was to begin when the grain had moisture content below 20% and subsequent harvests would be one week apart. Harvest 1 was considered as standard. As discussed in the results and discussion section, harvest times did vary

as determined by rainfall. Twelve spikes per row were collected before each harvest and used to determine moisture content. Rows were harvested by MB223 DH Mitsubishi binder. Grain was threshed and placed in a cloth bag. Grain harvested with moistures above 15% were dried in a dryer until it reached 12% moisture.

Grain Quality Analysis

Agronomic Traits/ Physical Qualities

Yield (g/plot) was determined according to the weight after cleaning the sample. Test weight (kg/hL) was determined by AACC International Approved Method 55-10.01 (AACC International, 2010). 1000-Kernel weight was determined by counting with an electronic seed counter (Seedburo Equipment Co., Chicago, IL) the total number of kernels in 10 g of cleaned grain and adjusting the weight to 1,000 kernels. Kernel size distribution was determined using the methodology described by Shuey (1960); where kernels were classified as large when remained on Tayler No 7 sieve with 2.92 mm opening (top sieve); medium when they remained on Tayler No 9 sieve with 2.24 mm opening (middle sieve); and small kernels passed directly through both sieves. Vitreous kernel content was determined by manually inspecting 15 g of intact kernels. Visual appearance of starchy and opaque kernels was classified as non-vitreous kernels (USDA, 1997). Grain color was determined by Minolta CR410 colorimeter (Konica Minolta, Ramsey, NJ) configured to measure Commission Internationale d' Eclairage (CIE) L*, a*, and b*-color values. L* measures the lightness of samples from black (0) to white (100), a* measures the greenness (-60) and redness (60), and b* measures blue (-60) to yellow (60).

Chemical Qualities

Grain was ground using a Thomas Wiley Mill (Thomas Scientific, Swedesboro, NJ) fitted with a 0.4 mm screen. Grain moisture and protein contents were determine using NIR technology

from FOSS Infratec^{TM} 1241 Grain Analyzer (FOSS Tecator, Hogonas, Sweden). Falling number was determined by AACC International Approved Method 56-81.03.

Yellow pigment content was determined by a modified AACC International Approved Method 14-50.01, where sample size was reduced from 8 to 4 g of ground whole wheat. The solvent was prepared in 5:1 ratio, which corresponds to 20 mL of water saturated n-butanol reagent (WSB) added to 4 g of ground sample. The mixture was shaken on a vortex mixer for 2 min followed by a 30 min rest; after which the samples were centrifuged (Eppendorf 5810R centrifuge, Rotor: F-34-6-38, Radius: 11.5cm) for 5 min at 18,514 *g relative centrifuge force (RCF). Absorbance of the supernatant was measured in a spectrophotometer (Beckman Coulter DU 720 General Purpose UV/Vis Spectrophotometer) at 436 nm. Measurements per extracted sample were converted to yellow pigment concentration (μg/g) using β-carotene extinction coefficient 1.6632.

Polyphenol oxidase activity was determined using the method described by Anderson and Morris (2001). Solution (1.5 mL) composed of 5 mM of L-3, 4 dihydroxyphenylalanine (L-DOPA) in 50 mM of 3-(*N*-morpholino) propanesulfonic acid (MOPS) buffer pH 6.5 was added to micro-centrifuge tube containing five undamaged seed. The tubes were placed on an orbital shaker (Glas-Col, Terre Haute, IN, USA) and were rotated for 1 h at room temperature to allow the reaction. Supernatant absorbance was measure at 475 nm (Beckman Coulter DU 720 General Purpose UV/Vis Spectrophotometer).

Semolina Quality Analysis

Grain was milled into semolina using AACC Method 26-50.01 (AACC International, 2010). Grain samples were tempered to 15% moisture 24 h before milling on a Brabender Quadrumat Jr (C.W. Brabender Instruments, Inc. South Hackensack, NJ, USA).

Ash content was determined by AACC International Approved Method (08-01.01). Semolina moisture and protein contents were determined using NIR technology from Foss Infratec 1241 Grain Analyzer. Dough properties were evaluated by mixograph (National Manufacturing, TMCO Division, Lincoln, NE) by using a modified AACC International Approved Method 54-40.01, where 10 g of semolina at 14% moisture basis was mixed for 8 min using a constant water absorption of 5.8 mL and a spring setting of 8. Wet gluten/gluten index were determined by AACC International Approved Method (38-12.02).

Semolina Dough Sheet Color

Semolina dough sheet was made using a modified method described by Fu et al. (2011). Semolina (30 g) was hydrated to 38% moisture with distilled water at 45°C and mixed for one min in KitchenAid mixer (4.3 L KitchenAid CLASSIC Stand Mixer 5K45SS) at speed 4. The dough sheet was made using a sheeting roll. First, the dough was sheeted twice in gap setting 3 mm, followed by two sheetings at 1 mm. The smooth dough sheet was transferred to a plastic bag and stored in the dark at room temperature. CIE L* and b* was measured on the dough sheet surface after 0.5 and 24 h using a Minolta CR410 colorimeter.

Experimental Design and Statistical Analysis

Field experiment was a randomized complete block design (RCBD) with four replicates in a split-plot in time arrangement, where cultivar was the main plot and harvest time was the sub plot. Each plot consisted in eight rows (two rows for each harvest time). The experiment was planted at three environments, Langdon 2014 and Prosper 2014 and 2015.

Cultivars and harvest time were considered fixed effects. To prove the homogeneity of the error mean square among dependent variables, Bartlett's Chi-square test (Steel and Torrie, 1980) and factor of 10 (Tabachnik and Fidell, 2001). Because no homogeneity of variance each

environment was analyzed individually by Statistical Analysis System (SAS) version 9.3 for Windows (SAS Institute Inc., Cary, NC, USA). All data collected was subject to analysis of variance (ANOVA) at 95 % level of confidence (F tests: $P \leq 0.05$). Means were separated by Fisher's-protected LSD at $P=0.05$. Pearson correlations were run using all the grain and semolina parameters evaluated.

RESULTS AND DISCUSSION

Environmental Conditions Before Harvest

Harvest date, daily rainfall, and maximum and minimum daily air temperatures during the harvest period for Langdon-14, Prosper-14, and Prosper-15 are presented in Figure 2. Cold winter and excess soil moisture in the spring 2014 delayed planting until mid-May and resulted in later harvest in 2014 (Aug 22 to Sep 24 at Prosper and Aug 28 to Sep 17 at Langdon) than 2015 (Aug 4 to Aug 31 at Prosper). Delayed harvest probably resulted in the lower minimum and maximum average temperatures during harvest in 2014 than 2015.

The goal was to space harvest time seven days apart; however, logistically this was not always possible due to rainfall. Differences in temperature and rainfall before harvest resulted in differences in kernel moisture content for all environments and within environment at each harvest date. At Langdon-14, the first harvest was on August 28 when the grain had 17% moisture content, the second was on September 4 when the grain had 14.5% moisture content, the third was on September 11 when the grain had 13% moisture content, and the fourth harvest was on September 17 when the grain had 13% moisture content. At Prosper-14, the first harvest was on August 22 when the grain had a 20% moisture content, the second was made on September 12 when the grain had 14% moisture, the third was made on September 19 (one week delayed) when the grain had 18% moisture content, and the fourth was made on September 26 (two week delayed) when the grain had 17% moisture content. In 2015, the first harvest at Prosper was on August 4 when the grain had 28% moisture, the second harvest was on August 11 when the grain had 16% moisture content, the third harvest was on August 24 when the grain had 14% moisture content, and the fourth harvest was on August 31 when the grain had 11% moisture content.

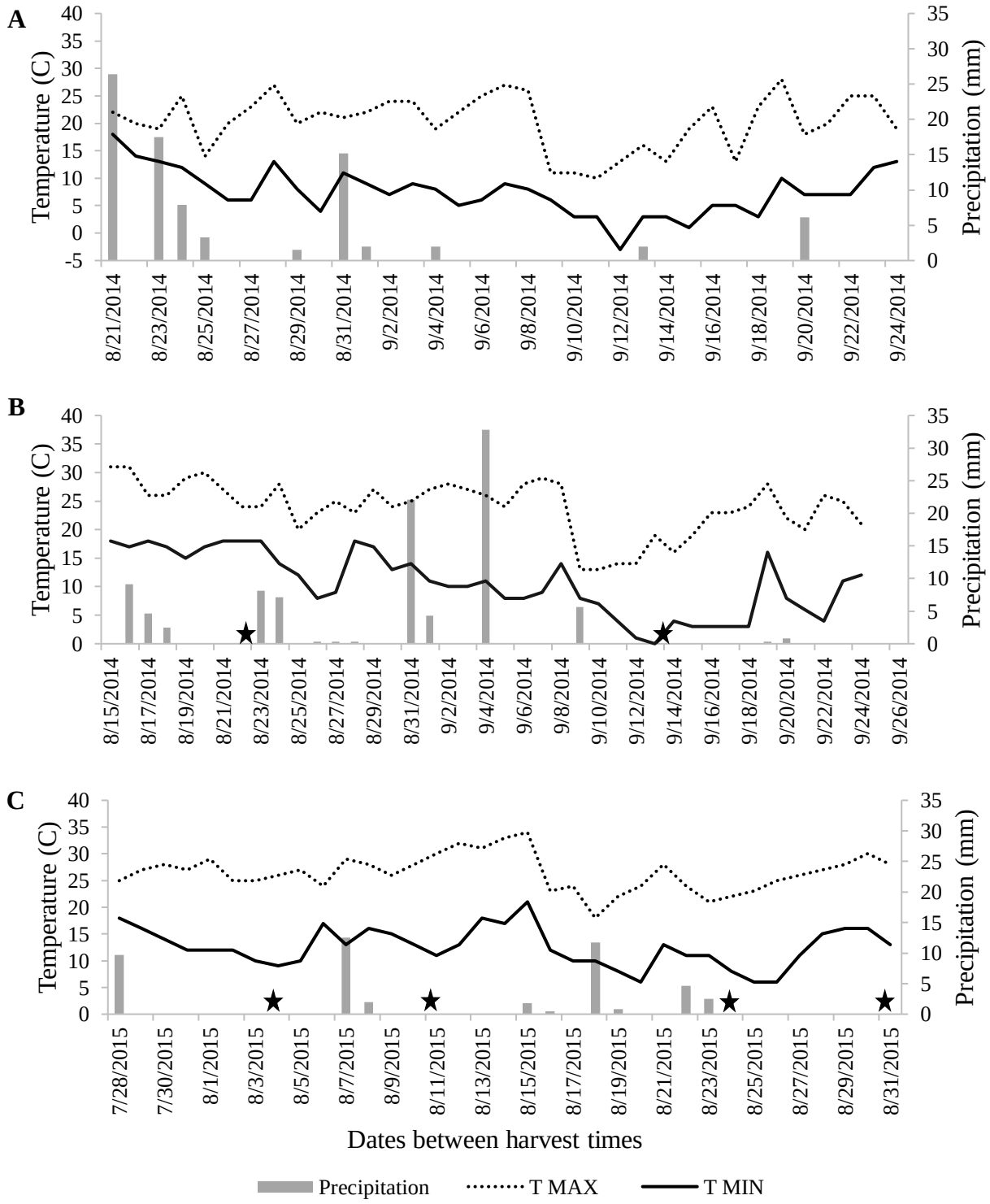

Figure 2. Harvest dates (★), total precipitation, and maximum and minimum temperature at Langdon-14 (A), Prosper-14(B), and Prosper-15(C).

Grain Quality Parameters

Yield

Cultivar x harvest time interaction was significant for yield at Prosper-14 and Prosper-15, but not at Langdon-14 (Table 1). At Prosper-14, yields were highest at the first harvest and declined at the second harvest except for Divide where yield was similar at the first, second and third harvests (Table 2). For Carpio and Grenora, yield was intermediate at the second and third harvests and least at the fourth harvest. Otherwise for the remaining cultivars, the second, third, and fourth harvest times resulted in similar yields. Joppa, Grenora, Alkabo, Carpio, and Pierce had the highest yields. Joppa, Alkabo, and Divide had the smallest decline in yield between harvest 1 and 4 (8.0, 8.2, and 8.8%, respectively). Conversely, Carpio and Grenora had the biggest yield decline (22.6 and 27.1%, respectively).

At Prosper-15, highest yields were with Tioga, Grenora, Divide and Carpio (Table 2). Dilse had the lowest yield decline between harvest 1 and 4 (8.5%), while the greatest decline was with Divide (29.9%). Yields were generally similar and did not decline between the first and second harvest at Prosper-15. Ben, Divide, Joppa, Lebsock, Mountrail, Strongfield and Tioga had significant decline in yield between the second and third harvest times. Alkabo, Carpio, Dilse, Divide, and Pierce had a decline in yield between the third and fourth harvests.

Cultivar main effect and harvest time main effect were significant for yield at Langdon - 14 (Table 1). Dilse had the highest yield (1,010 g plot) while Strongfield, Alkabo, and Ben had the lowest yields (890, 890 and 870 g/ plot, respectively) (Table 2). Yield was highest at the first harvest (average yield was 1007 g/plot). Yields of subsequent harvests were 8.0 to 12.0 % lower than that of the first harvest (data not shown). Although not statistically comparable, the average

yield loss between the first and fourth harvest was least for durum grown at Langdon-14 (9.4%), intermediate at Prosper-14 (14.1%), and greatest at Prosper-15 (18.0%).

Table 1. Analysis of variance for grain quality parameters of twelve cultivars harvested four times at Langdon-14, Prosper-14, and Prosper-15.

	Source of Variation								
	Cult†	Harv†	CxH†	Cult	Harv	CxH	Cult	Harv	CxH
Parameter	df=11	df=3	df=33	df=11	df=3	df=33	df=11	df=3	df=33
	Langdon-14			Prosper-14			Prosper-15		
Yield	***	**	ns	***	***	***	***	***	***
Test Weight	ns	*	ns	**	***	**	***	*	ns
1000-Kernel Weight	***	ns	ns	***	ns	*	***	ns	ns
Large Kernel Content	***	**	ns	***	***	***	***	***	*
CIE L* Color	***	***	ns	***	***	**	***	***	ns
Vitreous Kernel Content	ns	***	ns	ns	ns	ns	***	***	***
Falling Number	***	***	***	***	***	ns	***	*	**
Kernel Protein Content	***	ns	ns	ns	ns	ns	***	***	ns
Yellow Pigment Content	***	***	***	***	***	***	***	***	***
Polyphenol Oxidase Activity	***	ns	ns	***	ns	**	***	ns	***

†Cult = Cultivar main effect; Harv = harvest time main effect, and CxH, corresponds to their interaction.
df = degrees of freedom; ns = not significant.
*Significant at the 0.01 probability level.
** Significant at the 0.05 probability level.
*** Significant at the 0.001 probability level.

The decline in yield with delayed harvest might be a consequence of lodging. The main consequences of lodging are rain, strong winds, and hail especially at the last period of the crop growth (Rawson and Macpherson, 2000). Lodging was present at the later harvest times at all three environments, and was greatest in 2014 (personal information). Lodging in the plots could be due to the wind during June, July, August, and September (the highest wind speed average at Prosper-14 was 44.3, 36.4, 26.1, 34.0 km/h; at Langdon-14 was 41.4, 34.9, 28.9, and 36.5 km/h; at Prosper-15 was 36.7, 33.1, 29.3, and 32.2 km/h, respectively). Ferrer et al. (2006) reported that

wheat yield reduction during delayed harvest was due to hail damage, lodging, or shattering. Lodging has been reported to be the most limiting factor for high wheat yields as lodging ten days after heading can cause yield losses up to 40% (Kelbert et al., 2004).

Table 2. Means for grain yield, averaged across harvest time at Langdon-14 and as affected by cultivar x harvest time interaction at Prosper-14 and Prosper-15.

		Prosper -14				Prosper -15			
		Harvest time							
Cultivars	Langdon-14	1	2	3	4	1	2	3	4
	----------g/plot----------								
Alkabo	890	940	846	866	863	957	999	897	808
Ben	870	820	734	742	728	932	934	798	728
Carpio	950	929	838	839	719	1089	1160	1012	923
Dilse	1010	841	758	774	763	926	987	902	847
Divide	909	818	807	779	746	1044	1040	934	732
Grenora	939	954	879	885	695	1017	1048	941	893
Joppa	959	960	874	848	883	937	930	817	792
Lebsock	900	799	692	708	675	976	983	829	829
Mountrail	987	891	806	805	805	928	895	793	718
Pierce	935	921	775	806	779	943	971	908	810
Strongfield	890	830	704	695	648	862	851	694	675
Tioga	969	863	780	790	773	1047	1082	846	805
LSD (0.05)	28	----------63----------				----------92----------			

Test Weight

Harvest time x cultivar interaction was significant for Prosper-14 (Table 1), but was due to magnitude. Harvest time main effect was significant for test weight at Langdon-14, Prosper-14 and Prosper-15. At all environments, test weight was greatest at the first harvest time and declined with delay in harvest (Table 3). For Langdon-14 and Prosper-15, test weights were similar at the second, third and fourth harvests, but at Prosper-14, test weight declined between the third and fourth harvest. Overall, test weight declined 2.2 kg/hL at Langdon-14, 3.8 kg/hL at

Prosper-14, and 2.7 kg/hL at Prosper-15. Difference in test weight between US grades is 2.6 kg/hL, so the decline in test weight with delayed harvest was large enough at Prosper-14 and Prosper-15 to reduce the grade from US No. 1 to US No. 2.

Table 3. Means for test weight, averaged across cultivars at Langdon-14, Prosper-14, and Prosper-15.

Harvest	Langdon-14	Prosper-14	Prosper-15
	----------kg/hL----------		
1	81.3	79.4	79.3
2	79.1	77.0	77.5
3	79.3	77.5	76.7
4	80.0	75.6	76.6
LSD (0.05)	1.8	1.8	2.3

Cultivar main effect was significant for test weight at Prosper-14 and Prosper-15, but not at Langdon-14 (Table 1). Ben (79.0 kg/hL, US No. 1) and Carpio (79.1 kg/hL, US No.1) had the greatest test weight and Strongfield had the lowest test weight (75.5 kg/hL, US No. 3) at Prosper-14, when averaged over harvest times (Table 4). Similarly, at Prosper-15, Carpio had the greatest test weight (80.1 kg/hL, US No. 1), while Strongfield had the lowest (70.3 kg/hL, US No. 5) test weight, averaged over harvest times.

Czarnecki and Evans (1986) and McCaig et al. (2016) determined that the primary consequence of test weight reduction associated with delayed harvest was due to wet and dry cycles that caused wrinkling of the seed coat. Wrinkling of seed coat causes increased size and irregular shape of the grain. Gan et al. (2000) showed a reduction of test weight in spring wheat up to 50 kg m^{-3} related with rewetting and drying of the grain caused the roughening of the bran. They showed that rains before harvest ripening did not have bigger impacts on test weight, but increased between harvest 1 and 3. Lighter precipitation events do not have a big impact on test weight values (Swanson, l94l).

Table 4. Means for test weight, averaged across harvest time at Langdon-14, Prosper-14, and Prosper-15.

Cultivars	Langdon-14	Prosper-14	Prosper-15
	----------kg/hL----------		
Alkabo	78.0	78.1	78.4
Ben	80.7	79.0	78.7
Carpio	79.2	79.1	80.1
Dilse	80.5	78.1	76.9
Divide	80.2	78.0	78.3
Grenora	80.1	75.9	77.4
Joppa	80.0	77.6	77.6
Lebsock	81.1	78.3	79.2
Mountrail	80.6	76.3	76.6
Pierce	80.7	76.1	78.5
Strongfield	78.0	75.5	70.3
Tioga	79.4	77.1	78.0
LSD (0.05)	2.7	4.9	3.5

Thousand Kernel Weight

Cultivar x harvest interaction was significant for 1000-kernel weight at Prosper-14 (Table 1). Except for Ben and Mountrail, the 1000-kernel weights for a given cultivar were similar at all four harvest times (Table 5). 1000-Kernel weight of Ben was greater at the third and fourth than at the first or second harvest times. For Mountrail, 1000-kernel weight was greater at the third than at the second or fourth harvest times. In general, 1000-kernel weights were greatest with Alkabo, Ben, and Tioga and lowest for Strongfield, Divide, and Pierce.

Table 5. Means for 1000-kernel weight, averaged across harvest time at Langdon -14 and Prosper -15 and as affected by cultivar x harvest time interaction at Prosper -14.

		Prosper-14				
		Harvest time				
Cultivars	Langdon-14	1	2	3	4	Prosper-15
	---------- g/1000 kernels----------					
Alkabo	47.2	40.0	40.1	38.5	39.7	38.5
Ben	46.3	40.3	39.9	42.3	42.6	39.0
Carpio	43.9	38.2	39.5	40.2	40.5	40.6
Dilse	44.7	37.9	39.5	39.1	39.6	37.1
Divide	45.1	36.0	36.4	37.7	37.8	35.2
Grenora	46.6	39.7	39.5	38.6	38.5	38.6
Joppa	43.5	39.0	39.0	38.1	40.1	37.0
Lebsock	44.7	38.6	39.2	39.6	39.7	37.3
Mountrail	44.1	38.9	37.5	40.5	37.6	34.5
Pierce	41.0	37.2	36.3	37.8	38.2	33.3
Strongfield	43.5	36.0	35.5	35.9	35.4	32.3
Tioga	46.6	39.9	39.7	40.5	41.1	41.4
LSD (0.05)	1.3	----------2.3----------				1.0

For all three environments, harvest time main effect was not significant for 1000-kernel weight (Table 1). It seems reasonable that 1000-kernel weight would not be affected by harvest time since grain filling is complete by physiological maturity and any variation in weight would probably reflect fluctuations in kernel moisture with time and exposure to rain or heavy morning dew.

Cultivar main effect was significant for 1000-kernel weight at Langdon-14 and Prosper-15 (Table 1). At Langdon-14, 1000-kernel weight ranged from 41.0 g for Pierce to 47.2 g for Alkabo (Table 5). At Prosper-15, 1000-kernel weight ranged from 32.3 g for Strongfield to 40.6 g for Carpio.

Large Kernel Content

Cultivar x harvest time interaction was significant for large kernel content at Prosper-14 and Prosper-15, but not at Langdon-14 (Table 1). Large kernel content of Alkabo, Grenora, Mountrail, and Strongfield did not vary with delayed harvest at Prosper-14 (Table 6). For the remaining cultivars, large kernel content increased or tended to increase with delay in harvest. At Prosper-15, large kernel content increased between the second and third harvest for Alkabo, Dilse, and Grenora and by the fourth harvest for Carpio; otherwise, large kernel content was not affected by harvest time.

Table 6. Means for large kernel content, averaged across harvest time at Langdon-14 and as affected by harvest time x cultivar interaction at Prosper-14 and Prosper-15.

		Prosper-14				Prosper-15			
		Harvest time							
Cultivar	Langdon-14	1	2	3	4	1	2	3	4
	----------%----------								
Alkabo	79	61	68	64	65	44	51	53	59
Ben	81	62	66	68	73	51	49	52	50
Carpio	74	62	71	65	73	59	60	66	70
Dilse	71	52	59	60	63	27	29	37	34
Divide	73	34	49	49	54	44	47	44	41
Grenora	77	64	69	67	64	47	55	58	56
Joppa	59	47	60	60	60	39	35	41	39
Lebsock	73	61	67	68	70	46	48	48	49
Mountrail	65	51	56	58	57	30	25	32	34
Pierce	65	49	56	57	60	27	27	33	32
Strongfield	72	53	59	57	58	29	28	37	30
Tioga	77	63	72	73	76	64	58	62	61
LSD (0.05)	4	----------8----------				----------9----------			

Harvest time main effect and cultivar main effect were significant for large kernel content at Langdon-14 (Table 1). Large kernel content increased between the second (71%) and third

harvest time (74%, LSD=2). Large kernel content was similar at the first and second harvest times and at the third and fourth harvest times. At Langdon-14, Ben had the biggest large kernel content (81%) while Joppa had the smallest (59%) (Table 6). The increase in large kernel content between some of the harvest times could be related to high moisture in the environment. Sandhu et al. (2009) found that kernel size increased with exposure to high relative humidity. They suggested that the increased size was due to the swelling of the bran as moisture was absorbed by the kernel.

CIE L* Color

Cultivar x harvest time interaction was significant for kernel CIE L* color at Prosper-14 (Table 1). Kernel brightness of most cultivars increased between the first and second harvest times, with no further increase at the third or fourth harvest times (Table 7). Lebsock and Strongfield increased from the second and third harvest times and brightness decreased between second and third harvest times for Tioga. No additional brightness occurred between the third and fourth harvest times. Carpio had the highest kernel brightness at the first harvest. By the fourth harvest, Carpio, Divide, and Strongfield had the lowest kernel brightness.

Harvest time main effect was significant for kernel CIE L* color at Langdon-14 and Prosper-15 (Table 1). At Langdon-14, kernel brightness increased between the first (49.69) and second (50.87) harvest time but remained similar between the second, third and fourth harvest times. At Prosper-15, brightness increased incrementally between each harvest time, with L-values at first, second, third and fourth harvest times being 47.55, 48.80, 50.58, and 51.61, respectively (LSD=0.65).

Table 7. Means for CIE L* kernel color, averaged across harvest time at Langdon-14 and Prosper-15 and as affected by cultivar x harvest time interaction at Prosper-14.

		Prosper-14				
		Harvest time				
Cultivar	Langdon-14	1	2	3	4	Prosper-15
Alkabo	50.26	49.43	51.99	52.18	52.32	49.50
Ben	50.67	49.15	51.91	52.63	52.30	49.30
Carpio	50.61	48.92	50.83	51.69	50.90	48.60
Dilse	50.98	50.16	52.88	52.79	53.27	50.10
Divide	49.64	49.78	50.65	50.97	51.42	48.10
Grenora	51.49	50.15	52.96	52.42	53.15	51.20
Joppa	51.16	50.17	51.63	51.53	52.15	50.00
Lebsock	50.63	49.74	50.84	52.44	52.44	50.00
Mountrail	50.32	49.55	52.56	52.13	52.75	49.90
Pierce	50.71	50.09	52.49	52.35	53.14	50.30
Strongfield	50.67	49.43	50.45	51.64	50.36	49.70
Tioga	50.68	48.99	52.32	50.93	52.67	49.00
LSD (0.05)	0.74	----------1.24----------				1.10

L* represents CIE L*- value ranging from 0 (black) to 100 (white).

Cultivar main effect was significant for CIE L*color at Langdon-14 and Prosper-15 (Table 1). Kernel brightness was greatest with Dilse, Grenora, and Joppa and least with Divide at Langdon-14 (Table 7). At Prosper-15, kernel brightness was greatest with Grenora and Pierce and least with Carpio, Divide, and Tioga.

Grain brightness correlated negatively with test weight at Prosper-14 and Prosper-15 (r=-0.51, *P*=0.0002 and r=-0.39, *P*=0.0056, respectively). Brightness was positively correlated with large kernel content at Prosper-14 (r=0.37, *P*=0.0098). As stated above, bran absorbs moisture causing its surface to become wrinkled and irregular which would adversely affect kernel packing ability; also the bran will swell and cause a small increase in kernel size. Both irregular surface and increase in size without an increase in weight would result in decreased test weight.

Vitreous Kernel Content

Cultivar x harvest time interaction was significant for vitreous kernel content at Prosper-15 (Table 1). Vitreous kernel content decreased with delayed harvest (Table 8). Carpio, Dilse, Strongfield, and Tioga had little or no decrease in vitreousness between the first and second harvest, while Alkabo and Grenora had the greatest decline (21 and 24%, respectively). By the fourth harvest time, Alkabo and Grenora and Lebsock had the lowest vitreous kernel content (39, 47, and 47% respectively) and Dilse and Divide had the highest vitreous kernel content 78 and 76%, respectively. Vitreous kernel content had a negative correlation with kernel brightness (r=-0.70, *P*=0.0001).

Table 8. Means for vitreous kernel content as affected by cultivar x harvest time interaction at Prosper-15.

	Prosper -15			
	Harvest Time			
Cultivar	1	2	3	4
	----------%----------			
Alkabo	76	60	49	39
Ben	82	73	67	67
Carpio	76	79	71	66
Dilse	84	83	73	78
Divide	81	77	76	76
Grenora	82	62	49	47
Joppa	68	65	53	57
Lebsock	74	63	46	47
Mountrail	86	81	72	69
Pierce	80	77	59	60
Strongfield	78	80	59	64
Tioga	82	80	62	60
LSD (0.05)	----------2----------			

Harvest time main effect was significant for vitreous kernel content at Langdon-14 but not at Prosper-14 (Table 1). Cultivar main effect was not significant for vitreous kernel content at Langdon-14 or Prosper-14 (Table 1). At Langdon-14, vitreous kernel content was similar

between first (77%) and second harvest times (77%) but increased between the second (77%) and third (80%) and the third and fourth (84%, LSD=2) harvest times. Overall, kernel vitreous content was greatest at Prosper-14 (82%), intermediate at Langdon-14 (80%) and least at Prosper-15 (69%).

Falling Number (FN)

Cultivar x harvest time interaction was significant for falling number at Langdon-14 and Prosper-15 (Table 1). At Langdon-14, falling number for Grenora and Strongfield declined between the first and second harvest times (Table 9). The falling number for the remaining cultivars did not change significantly with harvest time. This could be related with the rainfall events before and between harvest 1 and 2 at Langdon-14, which had the highest rainfall accumulation (73.9 mm); while from harvest 2 to 4, the rainfall accumulated declined (Figure 2). Lebsock and Pierce had the least amount of variation among the four harvest times. Ranking of cultivars at a given harvest time did not vary greatly. In general, Carpio and Divide tended to maintain a relatively high falling number across harvest times, while Strongfield, Alkabo, and Tioga tended to have relatively low falling number across harvest times. At Prosper-15 the cultivar x harvest time interaction was due primarily by the high falling number value for Carpio at the fourth harvest time. Otherwise falling number did not vary with harvest time for a given cultivar. There is no quality advantage for falling numbers above 400 sec.

Table 9. Mean for falling number averaged across harvest time at Prosper-14 and as affected by cultivar x harvest time interaction at Langdon-14 and Prosper-15.

		Langdon-14				Prosper-15			
		Harvest time							
Cultivar	Prosper-14	1	2	3	4	1	2	3	4
	----------sec----------								
Alkabo	298	247	206	210	200	454	435	449	439
Ben	321	290	242	257	252	481	473	470	471
Carpio	355	265	302	315	307	511	527	536	608
Dilse	320	258	247	241	250	464	488	457	478
Divide	386	300	292	306	286	494	506	524	531
Grenora	270	340	268	289	276	504	502	487	517
Joppa	315	312	278	276	270	465	444	460	442
Lebsock	301	287	271	261	293	484	488	495	497
Mountrail	326	329	286	298	292	498	525	494	520
Pierce	342	278	272	280	284	486	466	476	480
Strongfield	293	287	159	168	152	568	584	513	567
Tioga	303	242	197	209	209	487	466	462	477
LSD (0.05)	28	----------48----------				----------40----------			

Harvest time main effect and cultivar main effect were significant for Prosper-14 (Table 1). Divide and Carpio had relatively high falling numbers while Alkabo, Grenora, and Strongfield had relatively low falling numbers. Prosper-14 falling number declined between the first (377 sec) and second harvest (291 sec) but had no further change at the third (306 sec) and fourth (302 sec; LSD=16) harvests. These results could be related to the greater amount of rainfall between harvest 1 and 2 (36 mm) compared with the other harvest times (Figure 2).

Falling number at the first harvest time averaged over cultivars was 286 sec at Langdon-14, which indicates that pre-harvest sprouting had initiated in the grain. Falling number at the first harvest time at Prosper-14 averaged across cultivars was 377 which indicates that the grain was exposed to damp conditions but was still sound. Falling number at Prosper-15 averaged 491

sec at the first harvest time which indicates that the grain was sound and was not affected by damp conditions.

The increase in the kernel brightness throughout delayed harvests might be related with the presence of some bleached kernels. It also could be related with the loss of vitreousness in some of the samples, as well as with initiation of pre-harvest sprouting as indicated by decreased falling number. McCaig et al. (2016) found increases in the brightness of the grain that could be related with the bleaching and weathering of samples after rain. They also determined that there was a relationship between increased brightness and the loss of vitreousness. However, for the results reported in this thesis, the relationship between falling number and vitreous kernel content was not that clear except for Prosper-15 where vitreous kernel content decreased as brightness increased (r=-0.70, P=0.0001).

Kernel Protein Content

Cultivar x harvest time interaction and harvest time main effect were not significant for kernel protein content at Langdon-14, Prosper-14, or Prosper-15 (Table 1). Kernel protein content would not be expected to be affected by harvest time since all deposition of protein is complete at the time of first harvest. Ferrer et al. (2006) and Lloyd et al. (1999) did not find any effect of delay harvest on grain protein of soft red winter wheat. Overall, protein content was greatest at Prosper-14 (13.5%), intermediate at Langdon-14 (13.1%), and least at Prosper-15 (12.9%).

Cultivar main effect was significant at Langdon-14 and Prosper-15 (Table 1). Kernel protein content was not significantly affected by cultivar at Prosper-14. At Langdon-14, Strongfield and Ben had the highest protein content values (13.7 and 13.5%, respectively), while Mountrail had the lowest value (12.7%) (Table 10). At Prosper-15, Dilse and Strongfield had the

highest protein content (14.3 and 14.2%, respectively); while Joppa and Alkabo had the lowest protein content (12.1%).

Table 10. Means for kernel protein, averaged across harvest time at Langdon-14, Prosper-14, and Prosper-15.

Cultivars	Langdon-14	Prosper-14	Prosper-15
	----------%----------		
Alkabo	13.1	13.2	12.1
Ben	13.5	13.2	12.8
Carpio	13.3	13.4	12.8
Dilse	13.4	13.7	14.3
Divide	12.9	13.7	13.1
Grenora	13.0	13.2	12.3
Joppa	12.4	12.9	12.1
Lebsock	12.9	14.2	12.6
Mountrail	12.7	13.3	12.6
Pierce	12.8	13.4	12.6
Strongfield	13.7	14.0	14.2
Tioga	12.9	13.2	13.0
LSD (0.05)	0.3	ns	0.2

Yellow Pigment Content

Cultivar x harvest time interaction was significant for yellow pigment content at Langdon-14, Prosper-14, and Prosper-15 (Table 1). At Langdon-14, yellow pigment content was not affected by delayed harvest for Alkabo, Ben, Mountrail and Strongfield (Table 11). Yellow pigment content was similar for first and second harvest times for all cultivars except Joppa where yellow pigment content declined. Yellow pigment content declined between the second and third harvest times for Carpio, Dilse, Joppa, Lebsock, Pierce, Strongfield and Tioga. Grenora had the most uniform yellow pigment content across harvest times (7.8 to 8.7 ppm), although, the yellow pigment content was not very high.

Table 11. Means for yellow pigment content as affected by cultivar x harvest time interaction at Langdon-14, Prosper-14, and Prosper-15.

	Langdon-14				Prosper-14				Prosper-15			
	Harvest time											
Cultivar	1	2	3	4	1	2	3	4	1	2	3	4
	ppm											
Alkabo	9.1	8.8	8.6	9.0	11.9	10.0	9.1	9.8	10.7	9.9	9.3	9.2
Ben	7.0	6.9	6.5	6.7	10.2	8.1	7.5	7.7	8.3	7.8	7.5	7.7
Carpio	11.3	11.2	10.5	10.3	13.9	12.1	10.7	10.8	12.9	11.5	10.6	10.9
Dilse	9.4	9.2	8.0	8.7	10.9	9.6	9.4	9.8	11.0	9.8	9.1	9.5
Divide	8.3	8.4	7.8	8.9	11.9	10.2	9.1	9.6	9.6	8.9	8.6	9.0
Grenora	7.8	8.1	7.6	8.7	10.8	9.9	9.7	9.2	10.1	9.1	8.6	9.1
Joppa	11.2	10.6	9.8	10.3	13.3	11.0	10.2	10.5	11.2	10.6	10.2	10.7
Lebsock	7.7	7.3	6.5	7.0	9.4	8.1	9.1	8.3	8.2	7.6	7.2	7.3
Mountrail	6.4	6.1	6.0	6.3	9.2	7.5	10.7	6.9	7.9	7.3	6.6	6.9
Pierce	9.3	9.0	8.3	8.6	10.8	9.3	10.0	8.7	10.5	9.7	9.6	9.5
Strongfield	9.5	9.5	8.7	9.3	12.1	11.1	10.2	10.5	11.2	10.9	10.0	10.9
Tioga	9.6	9.6	8.4	8.8	12.9	9.5	8.5	10.9	10.4	9.6	9.1	9.3
LSD (0.05)	0.6				1.0				0.5			

At Prosper-14, all cultivars lost yellow pigment content between the first and second harvest times except for Grenora which maintained its yellow pigment content (Table 11). At Prosper-14, Carpio and Joppa had the greatest reduction of yellow pigment content between harvest 1 and 4 (22 and 21%, respectively); however, they were also the cultivars with the higher yellow pigment content at all harvest times. Similarly, at Prosper-15, all cultivars lost pigment content between the first and second harvest. At Prosper-15, Strongfield and Joppa had the lowest decline in yellow pigment content between harvest 1 and 4 (4 and 3%, respectively), also being cultivars with high yellow pigment content throughout harvest times. Even though Carpio had the greatest reduction in yellow pigment (16%), it had good yellow pigment content.

For all three environments, yellow pigment content was generally greatest with Carpio, Joppa, and Strongfield and lowest with Ben, Lebsock, and Mountrail. At Prosper-14 and Prosper-15, yellow pigment content had a negative correlation with grain brightness (r=-0.62, *P*=0.0001 and r=-0.30, *P*=0.0373). This indicates a possible relationship between bleaching (increased grain brightness) and loss of yellow pigment. No correlation existed with data from Langdon-14.

Polyphenol Oxidase (PPO)

Cultivar x harvest time interaction was significant for PPO activity at Prosper-14 and Prosper-15 (Table 1). At both environments there were no clear tendencies of cultivars throughout harvest times, since some of the cultivars had increased PPO activity, while others had decreased activity (Table 12). At Prosper-14 half of the cultivars increased PPO activity between harvest 1 and 4. Tioga and Strongfield had the greatest reduction in PPO activity, 57 and 59%, respectively, from first to the fourth harvest times. Although their PPO activities were much lower than Tioga and Strongfield, Alkabo and Ben had similar percentage decline in activity from first to fourth harvest (62 and 60%, respectively). Lebsock had the greatest increase in PPO activity (5.3 fold increase).

The ranking of cultivars varied greatly with harvest time (Table 12). At Prosper-15, ten of the twelve cultivars had decreased PPO activity between the first and fourth harvest. The reduction was almost the same for all twelve cultivars. Carpio, Strongfield, and Tioga generally had the highest PPO activity at first, second and fourth harvest times. However, Carpio and Tioga had low PPO activity at the third harvest time.

Interestingly, PPO activity increased with yellow pigment content at Langdon-14 and Prosper-15 (r=0.45, *P*=0.0013 and r=0.49, *P*=0.0005) respectively. In addition, PPO activity increased with increased falling number at Prosper15 (r=0.56, *P*=0.0001).

Table 12. Means for PPO, averaged across harvest time at Langdon-14 and as affected by cultivar x harvest time interaction at Prosper-14 and Prosper-15.

		Prosper-14				Prosper-15			
		Harvest time							
Cultivar	Langdon-14	1	2	3	4	1	2	3	4
	----------475 nm----------								
Alkabo	0.113	0.305	0.353	0.270	0.115	0.041	0.049	0.145	0.040
Ben	0.121	0.303	0.295	0.253	0.120	0.055	0.047	0.081	0.034
Carpio	0.799	0.088	0.110	0.270	0.103	0.416	0.477	0.168	0.470
Dilse	0.171	0.108	0.080	0.083	0.095	0.085	0.090	0.040	0.057
Divide	0.104	0.163	0.130	0.175	0.205	0.042	0.040	0.293	0.038
Grenora	0.156	0.303	0.135	0.170	0.435	0.041	0.164	0.125	0.037
Joppa	0.144	0.290	0.073	0.110	0.400	0.043	0.049	0.044	0.035
Lebsock	0.159	0.078	0.090	0.173	0.415	0.045	0.052	0.076	0.046
Mountrail	0.283	0.155	0.213	0.315	0.138	0.042	0.044	0.089	0.035
Pierce	0.130	0.315	0.108	0.110	0.288	0.041	0.046	0.220	0.031
Strongfield	0.704	0.715	0.103	0.095	0.295	0.255	0.169	0.042	0.235
Tioga	0.487	0.675	0.100	0.173	0.288	0.194	0.153	0.041	0.173
LSD (0.05)	0.116	----------0.246----------				----------0.108----------			

In general, delayed harvest resulted in a decline in yield, test weight, kernel vitreous content, falling number, and yellow pigment content at all three environments. Large kernel content and kernel brightness tended to increase with delayed harvest. Protein content was not affected by delayed harvest. There was no clear trend as to the effect of delayed harvest on PPO activity. Most of the effect of delayed harvest can be attributed to effects caused by moist conditions. At harvest, grain reabsorbed moisture from rainfall or from heavy dew. As grain absorbed moisture, the bran layer expanded and its surface became irregular, which resulted in increased kernel size and decrease in test weight. Fractures form as moisture moves into the

endosperm. These fractures result in the endosperm appearing to be non-vitreous. Moisture will also activate hydrolytic enzymes associated with pre-harvest germination, which will result in a reduction in falling number. Yellow pigment in durum wheat acts as an antioxidant. Conditions that cause oxidative stress will result in a reduction in yellow pigment.

Semolina Quality Parameters

Semolina Extraction

Cultivar x harvest time interaction was not significant for semolina extraction at Langdon-14, Prosper-14, or Prosper-15 (Table 13). Harvest time and cultivar main effects were significant for semolina extraction at Langdon-14 and Prosper-15 but not Prosper-14.

The effect of harvest time was variable at both Langdon-14 and Prosper-15. At Langdon-14, semolina extraction was greatest at the second harvest time (59.8%), intermediate at the fourth harvest time (59.1%) and least at the first and third harvest time (both 58.8%, LSD=0.4). At Prosper-15, semolina extraction was greatest at the fourth harvest (58.1%), intermediate at the first and third harvest (57.0 and 57.6%, respectively) and least at the second harvest time (55.0, LSD=1.0).

Semolina extraction from cultivars grown at Langdon-14 was greatest with Tioga, Joppa, Alkabo, and Ben and least with Pierce, Carpio, and Divide, while from cultivars grown at Prosper-15, semolina extraction was greatest with Joppa, Alkabo, Carpio, Lebsock, Tioga, Ben, and Grenora and least with Strongfield, Pierce, and Divide (Table 14). It is interesting that Carpio had one of the highest extraction rates when grown at Prosper-15 and one of the lowest when grown at Langdon-14.

Table 13. Analysis of variance for semolina quality parameters of twelve cultivars harvested four times at Langdon-14, Prosper-14, and Prosper-15.

	Source of Variation								
	Cult[†]	Harv[†]	CxH[†]	Cult	Harv	CxH	Cult	Harv	CxH
Parameter	df=11	df=3	df=33	df=11	df=3	df=33	df=11	df=3	df=33
	Langdon-14			Prosper-14			Prosper-15		
Semolina Extracation	***	***	ns	ns	ns	ns	***	***	ns
Semolina CIE L*-value	ns	***	ns	ns	ns	ns	ns	ns	ns
Semolina CIE b*-value	***	***	ns	***	***	***	***	***	ns
Ash Content	***	***	ns	ns	ns	ns	***	ns	ns
Semolina Protein Content	**	ns	ns	ns	***	ns	ns	***	ns
Wet Gluten Content	***	***	***	***	**	ns	***	***	ns
Gluten Index	***	***	***	***	***	***	***	***	***
Peak time	***	***	ns	***	ns	***	***	***	ns
Peak height	ns	***	ns	**	ns	ns	***	***	ns
Peak width	ns	ns	***	ns	**	ns	***	***	ns

[†]Cult = Cultivar main effect; Harv = harvest time main effect, and C x H, corresponds to their interaction.
df = degrees of freedom; ns=not significant.
*Significant at the 0.01 probability level.
** Significant at the 0.05 probability level.
*** Significant at the 0.001 probability level.

Semolina extraction was positively correlated with 1000-kernel weight at Langdon-14 (r=0.44, *P*=0.0017) and 1000-kernel weight (r=0.41, *P*=0.0035) and large kernel content (r=0.42, *P*=0.0031) at Prosper-15. Previous works have indicated that the milling performance is related to 1000-kernel weight and large kernel size (Troccoli et al., 2000). Although not statistically comparable, the overall average semolina extraction was higher with grain harvested from Langdon-14 (59.1%) and Prosper-14 (59.3%) than grain from Prosper-15 (57.0%).

Table 14. Means for semolina yield, averaged across harvest time at Langdon-14 and Prosper-15.

Cultivars	Langdon-14	Prosper-15
	----------%----------	
Alkabo	59.7	58.3
Ben	59.6	57.5
Carpio	58.3	57.9
Dilse	59.2	56.6
Divide	58.5	55.5
Grenora	58.6	57.2
Joppa	60.1	58.8
Lebsock	59.5	57.8
Mountrail	59.4	56.7
Pierce	57.9	55.1
Strongfield	58.7	54.8
Tioga	60.2	57.6
LSD (0.05)	0.7	1.7

CIE L* Semolina Color

Cultivar x harvest time interaction and cultivar main effect were not significant for CIE L* semolina color at Langdon-14, Prosper-14, or Prosper-15 (Table 13). Harvest time main effect was significant for CIE L* semolina color at Langdon-14, but not at Prosper-14 or Prosper-15. At Langdon-14, brightness was greater at the first (L*=81.91) and fourth (L*=81.75) harvest times than at the second (L*=81.30) or third (L*=81.45, LSD=0.35) harvest times. Although not statistically comparable, brightness varied with environment with overall averages of L*=81.60 for Langdon-14, L*=79.99 for Prosper-14, and L*=77.95 for Prosper-15.

CIE b* Semolina Color

Cultivar x harvest time interaction was significant for CIE b* semolina color at Prosper-14, but not at Langdon -14 or Prosper -15 (Table 13). At Prosper-14 there was a reduction in yellow color between the first and fourth harvest times (Table 15). However, 9 of the 12 cultivars

had an increase in b* color from between the second and third harvest times and a reduction between the third and fourth harvest times. Variation in b*-values could be due to yellow pigment content and also to particle size distribution of semolina. Finer particles will appear less yellowness and have a lower b*- value (Hruskova et al., 2011).

Table 15. Means for CIE b* semolina color, averaged across harvest time at Langdon-14 and Prosper -15, and as affected by cultivar x harvest time interaction at Prosper-14.

		Prosper-14				
		Harvest Time				
Cultivar	Langdon-14	1	2	3	4	Prosper-15
Alkabo	26.31	26.83	25.58	25.27	25.09	25.40
Ben	23.73	23.83	21.54	22.73	22.35	22.70
Carpio	28.02	29.09	26.28	26.97	25.39	27.21
Dilse	26.69	26.22	25.04	25.15	25.15	26.04
Divide	25.78	27.26	25.90	26.15	25.50	24.69
Grenora	25.71	24.62	22.84	24.14	23.02	24.27
Joppa	28.22	26.98	26.25	24.97	26.20	25.88
Lebsock	23.99	23.65	22.84	20.72	21.68	21.92
Mountrail	22.66	22.28	20.94	21.13	20.83	21.91
Pierce	27.02	26.70	25.35	25.72	24.55	25.76
Strongfield	25.34	24.70	23.79	24.28	23.54	25.72
Tioga	26.02	26.54	23.82	24.36	24.02	24.35
LSD (0.05)	0.46	----------1.07----------				0.67

Harvest time main effect was significant for CIE b* semolina color for Langdon-14 and Prosper-15 (Table 13). At Langdon-14, b*-value was greater at the first harvest (26.35) than at the second (25.42), third (25.80) or fourth (25.58, LSD=0.27) harvest times. At Prosper-15, b*-value was greatest at the first harvest (25.29), intermediate at the second (25.10) and least at the third and fourth (24.16 and 23.85, LSD=0.39) harvest times.

Cultivar main effect was significant for CIE b* semolina color for Langdon-14 and Prosper-15 (Table 13). At all three environments, Mountrail, Ben, and Lebsock had the lowest

b*- values, while Carpio, and Joppa had the highest values. Yellow pigment content was correlated positively with CIE b* semolina color at Langdon-14 (r=0.883, *P*=0.0001); Prosper-14 (r=0.721, *P*=0.0001); and Prosper-15 (r=0.920, *P*=0.0001). Positive correlations of CIE b* color (yellowness) with total yellow pigment content were also found by Ramachadran et al. (2010).

Ash Content

Cultivar x harvest time interaction was not significant for ash content at Langdon-14, Prosper-14 or Prosper-15 (Table 13). Ash content, averaged over harvest time and cultivar, was greatest at Prosper-14 (0.87%), intermediate at Prosper-15 (0.81%), and least at Langdon-14 (0.72%). Harvest time main effect was significant for ash content only at Langdon-14. Ash content increased between the first (0.70%) and second (0.72%) harvest and between the second and third (0.74%) harvest. The ash content was similar at the third and fourth harvest (0.73%; LSD=0.01).

Cultivar main effect was significant for ash content at Langdon-14 and Prosper-15 (Table 13). Cultivar rankings varied greatly between the three environments. For example, ash content was similar for all cultivars at Prosper-14. Pierce and Tioga had the lowest and Grenora had the highest ash contents at Langdon-14. Alkabo, Joppa, and Pierce had the lowest ash content at Prosper-15 (Table 16). In general, Alkabo, Joppa, and Pierce ranked as low ash content cultivars at all three environments.

Table 16. Means for ash content, averaged across harvest time at Langdon-14 and Prosper-15.

Cultivars	Langdon-14	Prosper-15
	----------%----------	
Alkabo	0.71	0.74
Ben	0.73	0.82
Carpio	0.73	0.82
Dilse	0.73	0.85
Divide	0.71	0.83
Grenora	0.76	0.82
Joppa	0.72	0.78
Lebsock	0.74	0.81
Mountrail	0.73	0.83
Pierce	0.69	0.78
Strongfield	0.72	0.85
Tioga	0.70	0.81
LSD (0.05)	0.02	0.05

Semolina Protein Content

Cultivar x harvest time interaction was not significant for semolina protein content at Langdon-14, Prosper-14 or Prosper-15 (Table 13). Harvest time main effect was significant for semolina protein content at Prosper-14 and Prosper-15. Semolina protein content did not vary with harvest time at Langdon-14. At Prosper-14, semolina protein content declined between the first (12.3%) and second (11.8%) harvest times, with no significant decline at the third (11.7%) and fourth (11.6%; LSD=0.3) harvest times. At Prosper-15, semolina protein content was similar for durum harvested at the first (11.1%), second (11.1%) and third (11.1%) times but increased at the fourth harvest time (11.4%; LSD=0.1).

Cultivar main effect was significant for semolina protein content at Langdon-14 (Table 13). Cultivars did not differ in semolina protein content when grown at Prosper-14 or Prosper-15. At Langdon-14, Ben (12.0%), Dilse (12.1%), and Strongfield (12.0%) had the highest semolina

protein content, while Joppa had the lowest (11.2%, LSD=0.3). Overall, Semolina protein content, averaged over harvest time and cultivar, was greatest at Prosper-14 (11.9%), intermediate at Langdon-14 (11.7%), and least at Prosper-15 (11.2%).

Wet Gluten

Cultivar x harvest time interaction was significant for wet gluten at Langdon-14 but not at Prosper-14 or Prosper-15 (Table 13). For a given cultivar at Langdon-14, wet gluten content was similar at each harvest time, except for Lebsock, where wet gluten was lower at the fourth (30.2%) than at the first (33.5%) harvest time (Table 17). Six of the twelve cultivars tended to have increased wet gluten between the first and fourth harvest times. In general, Carpio and Joppa had the lowest wet gluten content and Ben, Dilse and Lebsock had the highest wet gluten content.

Harvest time main effect was significant for wet gluten for Prosper-14 and Prosper-15 (Table 13). At Prosper-14, wet gluten content declined between the first (31.3%) and second (30.3%) harvest time, with no further decline at the third (30.6%) or fourth (29.9%) harvest time. At Prosper-15, wet gluten increased between first (30.4%) and second (31.1%) harvest times and declined between the second and third (28.8%) harvest times. Third and fourth harvest times had similar wet gluten content (28.8%, LSD=0.7).

Cultivar main effect was significant for wet gluten at Prosper-14 and Prosper-15 (Table 13). Ranking of cultivars varied greatly at both environments (Table 17). At Prosper-14, wet gluten content was greatest with Lebsock (34.5%) and least with Joppa (27.7%). At Prosper-15, wet gluten content was greatest with Dilse (35.9%) and least with Joppa (25.3%).

Table 17. Means for wet gluten as affected by cultivar x harvest time interaction at Langdon-14 and averaged across harvest time at Prosper-14 and Prosper-15.

	Langdon-14					
	Harvest time					
Cultivar	1	2	3	4	Prosper-14	Prosper-15
	----------%----------					
Alkabo	30.0	32.3	31.8	31.9	30.3	28.1
Ben	33.3	33.7	32.0	33.7	29.7	30.1
Carpio	26.4	25.7	23.2	24.8	28.4	27.8
Dilse	32.4	32.9	34.8	33.7	33.1	35.9
Divide	28.7	29.2	27.2	28.9	29.8	30.2
Grenora	30.5	31.0	29.1	31.8	28.9	27.3
Joppa	27.3	25.8	25.4	25.1	27.7	25.3
Lebsock	33.5	32.3	32.3	30.2	34.5	29.5
Mountrail	31.2	31.8	31.5	31.9	33.1	31.1
Pierce	32.1	30.8	29.9	30.0	31.3	29.5
Strongfield	32.1	31.6	29.9	30.1	30.0	33.3
Tioga	29.3	28.6	26.7	27.1	29.3	29.2
LSD (0.05)	----------2.8----------				1.5	1.3

Wet gluten content was positively correlated with grain protein and semolina protein at Langdon-14 (r=0.44, *P*=0.0019 and r=0.57, *P*=0.0001, respectively) and at Prosper-14 (r=0.57, *P*=0.0001 and r=0.54, *P*=0.0001, respectively) but not Prosper-15. Wet gluten content is a quantitative measure of the gluten forming proteins in semolina and is important in determining mechanical strength and cooking quality (NDWC, 2014).

Gluten Index

Cultivar x harvest time interaction was significant for gluten index at Langdon-14, Prosper-14, and Prosper-15 (Table 13). At Langdon-14, gluten index increased between first and second harvest times for Ben, Dilse, Divide, Lebsock, and Pierce and between second and third harvest times for Alkabo, Mountrail, Strongfield, and Tioga (Table 18). Gluten index for Carpio,

Grenora, and Joppa were not affected by delayed harvest. At Prosper-14, first increase in gluten index relative to the first harvest occurred at the second harvest time for Ben, Grenora, Joppa, Mountrail, and Pierce; at the third harvest time for Alkabo, Carpio, and Strongfield; and at the fourth harvest time for Dilse. Gluten index of Divide did not change with harvest time. For Prosper-15, the first increase in gluten index, relative to the first harvest, occurred at the second harvest time for Genora, Lebsock, and Tioga; at the third harvest time with Alkabo, Ben, Dilse, Divide, Joppa, Mountrail, Pierce, and Strongfield. Gluten index of Divide did not change with harvest time. At each environment, the ranking of cultivars varied with harvest time. However, gluten index was generally greatest with Carpio and Joppa and lowest with Mountrail.

Table 18. Means for gluten index as affected by cultivar x harvest time interaction at Langdon-14, Prosper-14, and Prosper-15.

	Langdon-14				Prosper-14				Prosper-15			
	Harvest time											
Cultivar	1	2	3	4	1	2	3	4	1	2	3	4
	----------%----------											
Alkabo	55	55	67	71	46	51	74	67	40	47	68	66
Ben	49	59	73	70	41	54	62	77	48	55	71	75
Carpio	98	99	99	99	81	91	92	96	94	94	97	98
Dilse	43	56	76	65	36	42	47	73	43	48	61	60
Divide	77	87	95	92	80	73	86	88	74	78	91	90
Grenora	84	74	84	83	44	70	74	90	55	71	85	74
Joppa	97	98	99	99	71	87	89	96	88	92	98	98
Lebsock	43	52	59	64	41	48	40	85	34	43	58	66
Mountrail	36	38	54	52	31	47	22	39	2	4	30	37
Pierce	65	73	84	84	42	55	71	75	62	68	78	85
Strongfield	69	72	85	83	54	64	76	85	61	68	78	77
Tioga	87	88	95	97	71	68	72	86	70	85	90	92
LSD (0.05)	----------10----------				----------13----------				----------9----------			

Gluten index is an indicator of the gluten strength. Gluten strength is a very important parameter in pasta production because it is related to the balance between viscosity and elasticity of the dough (Shewry et al., 2002). Stronger gluten often results in firmer pasta (Marchylo et al., 2001). Ames et al. (2003) evaluated 10 durum cultivars found a variation from 9 to 77 GI with a protein content of 12.5 to 15.1% determined a positive relationship between pasta quality and gluten strength.

Mixogram Analysis

Peak time

Cultivar x harvest time interaction was significant for peak time at Prosper-14 but not at Langdon-14 or Prosper-15 (Table 13). At Prosper-14, peak times did not change with harvest time for Carpio, Divide, Grenora, Joppa, Lebsock, or Tioga. Peak time for Alkabo, Ben, Dilse, Mountrail, Pierce, and Strongfield increased or tended to increase between the first and second harvest times and remained steady over the third and fourth harvest times. Ranking of cultivars differed with harvest time. In general, peak time was greatest with Alkabo and Joppa and lowest with Tioga (Table 19).

Harvest time main effect and cultivar main effect and significant for peak time at Langdon-14 and Prosper-15 (Table 13). At Langdon-14 and Prosper-15, Joppa (270 and 263 s) and Carpio (255 and 244 s) had the greatest peak times and Mountrail (163 and 155 s), Lebsock (166 and 163 s), and Alkabo (170 and 164 s) had the lowest peak times. At Langdon-14, peaked time increased from first (185 s) to third (213 s, LSD=11) harvest times and remained similar between the third and fourth harvest times with both being 213 s. Similarly, at Prosper-15, peak time increased from first (165 s) to the third (236 s, LSD=15) but then decrease between the third and fourth (219 s) harvest times. Peak time was positively correlated with gluten index (r=0.780,

P=0.0001) and negatively correlated with wet gluten content (r=-0.776, *P*=0.0001) at Langdon-14 but not at Prosper-14 or Prosper-15.

Table 19. Means for peak time, averaged across harvest time at Langdon-14 and Prosper-15 and as affected by cultivar x harvest time interaction at Prosper-14.

		Prosper-14				
		Harvest time				
Cultivar	Langdon-14	1	2	3	4	Prosper-15
	----------sec----------					
Alkabo	170	147	230	224	239	164
Ben	173	168	206	233	196	193
Carpio	255	179	181	180	170	244
Dilse	176	150	204	189	184	178
Divide	196	175	175	178	178	201
Grenora	184	163	168	170	170	208
Joppa	270	241	198	224	226	263
Lebsock	166	160	178	153	181	163
Mountrail	163	133	184	189	180	155
Pierce	193	151	215	218	171	191
Strongfield	238	227	160	104	168	205
Tioga	220	170	150	148	156	205
LSD (0.05)	19	----------46----------				26

Peak height

Cultivar x harvest time interaction was not significant for peak height for Langdon-14, Prosper-14, or Prosper-15 (Table 13). Harvest time main effect was significant for peak height at Langdon-14 and Prosper-15. At Prosper-14, peak height did not vary with harvest time. At Langdon-14, peak height declined between first (6.3 cm) and second (6.1 cm) harvest times then did not change between the second and fourth (6.0 cm, LSD=0.2) harvest times. At Prosper-15, peak height declined between the first (5.5 cm) and second (5.2 cm) harvest times but increased

between the second and third (5.9 cm) harvest times. Peak height was similar at third (5.9 cm) and fourth (5.9 cm, LSD=0.2) harvest times.

Cultivar main effect was significant for peak height at Prosper-14 and Prosper-15. Cultivars did not vary in ranking at Langdon-14. At Prosper-14, peak height was lowest with Joppa (5.1) and highest with Dilse (5.9). Similarly, at Prosper-15, peak height was lowest with Joppa (5.0) and highest with Dilse (6.3) and Carpio (6.2) (Table 20).

Table 20. Means for peak height, averaged across harvest time at Langdon-14, Prosper-14, and Prosper-15.

Cultivars	Langdon-14	Prosper-14	Prosper-15
	----------cm----------		
Alkabo	6.2	5.3	5.2
Ben	6.2	5.5	5.5
Carpio	6.3	5.4	6.2
Dilse	6.3	5.9	6.3
Divide	6.0	5.4	5.9
Grenora	6.4	5.3	5.7
Joppa	6.0	5.1	5.0
Lebsock	5.9	5.5	5.3
Mountrail	6.2	5.6	5.9
Pierce	6.3	5.4	5.5
Strongfield	5.5	5.3	5.4
Tioga	5.9	5.6	5.6
LSD (0.05)	0.3	0.3	0.3

Peak width

Cultivar x harvest time interaction was significant for peak width at Langdon-14 but not at Prosper-14 or Prosper-15 (Table 13). Except for Pierce, Strongfield, and Tioga, peak width did not vary with harvest time at Langdon-14 (Table 21). For Pierce and Strongfield, peak width decreased between the first and second harvest times, with no further decline at the third or fourth harvest times. Peak width declined between the first and fourth harvest times.

Harvest time main effect was significant for peak width at Prosper-14 and Prosper-15. At Prosper 2014, harvests 1 and 3 had the greatest peak width value (2.9 cm); while harvest 4 had the lowest value (2.3 cm, LSD=0.2). Conversely, at Prosper 2015, peak width value was lowest (2.0 cm) at the first harvest time and highest (2.9 cm, LSD=0.3) at the fourth harvest time.

Table 21. Means for peak width, averaged across harvest time at Prosper-15 and as affected by cultivar x harvest time interaction at Langdon-14.

	Langdon-14				
	Harvest time				
Cultivar	1	2	3	4	Prosper-15
	----------cm----------				
Alkabo	3.33	2.73	3.35	2.80	2.13
Ben	2.80	3.00	3.55	3.30	2.19
Carpio	2.43	3.28	2.88	2.55	3.31
Dilse	2.98	3.78	3.30	2.85	2.93
Divide	2.78	3.35	3.25	2.90	2.74
Grenora	3.18	3.20	3.48	3.23	2.59
Joppa	2.83	3.78	2.55	3.05	2.29
Lebsock	2.73	3.38	2.50	2.33	2.11
Mountrail	3.70	3.90	2.70	3.98	1.84
Pierce	4.75	3.05	2.95	3.45	2.54
Strongfield	4.65	3.25	2.40	2.93	2.78
Tioga	4.08	3.38	2.95	2.58	2.91
LSD (0.05)	----------1.37----------				0.54

Cultivar main effect was significant for peak width at Prosper-15. Cultivars did not vary in peak width at Prosper-14. At Prosper-15, peak width was greatest with Carpio (3.31 cm), Dilse (2.93 cm), and Tioga (2.91 cm) and least with Mountrail (1.84 cm), Lebsock (2.11 cm), Alkabo (2.13 cm), and Ben (2.19 cm) (Table 21).

Mixograph is used to measure the dough strength of semolina (Welle and Trentesaux, 1980). Rao et al. (2001), found a relationship between variation in the mixograph curves and

dough mixing properties among different durum cultivars. They identified that durum cultivars with the strongest gluten had longer mixing times than did cultivars with weak gluten. Cultivars with weak gluten produced a more viscous but less elastic dough. The results of this research found that gluten strength, as measured by gluten index was correlated with peak time (r=0.78, *P*=0.0001) and peak width (r=0.47, *P*=0.0008) at Langdon-14. Peak height, peak time and peak width were not correlated with gluten index at Prosper-15. Peak height was negatively correlated with gluten index (r=-0.54, *P*=0.0001) at Prosper-14. Peak height is generally associated with semolina protein content and wet gluten content. Peak height was correlated with semolina protein content (r=0.44, *P*=0.0016) and wet gluten content (r=0.45, *P*=0.0013) at Prosper-14, not at Prosper-15 or Langdon-14.

In general, delayed harvest did not greatly affect semolina yield, semolina brightness, ash content, semolina protein content or peak height. Semolina b*- value (yellow color), wet gluten content, and peak width declined with delayed harvest. Interestingly, gluten index increased with delay harvest at all three environments. Debbouz et al. (1995) found that gluten strength on semolina was the only quality parameter evaluated not affected by severe weather damage.

Cultivars with the best semolina extraction were Tioga, Alkabo, and Joppa, contrarily Divide had the lowest semolina extraction. Carpio and Joppa had the best semolina b* value (yellow color), while Mountrail, Ben, and Lebsock the lowest b* value. The highest semolina protein content was obtained by Ben, Dilse, and Strongfield, while Joppa had the lowest protein content. In the case of wet gluten content, Ben, Dilse, and Lebsock had the highest wet gluten content while Carpio and Joppa the lowest content. Joppa and Carpio had the strongest gluten index, and Mountrail the weakest. For dough properties measured by mixograph, Joppa, Carpio,

Dilse had good performance, while Mountrail, Lebsock, and Alkabo had poor performance at all three environments.

According to the overall information a positive correlation between peak height and falling number was found at Langdon-14 and Prosper-14 (r=0.40, *P*=0.002, and r=0.40, *P*=0.01, respectively). While a negative correlation between falling number and kernel and semolina protein was found at Langdon-14 (r=-0.36, *P*=0.01and r=-0.30, *P*=0.03, respectively). These make sense since peak height is related with protein content, and as was mentioned above, lower falling number is related to the activation of hydrolytic enzymes that degrade starch, protein and lipids, making the dough weaker. Johansson (2002) found a negative correlation between falling number and gliadin and glutenin contents, and alpha amylase content in winter wheat.

Peak width was correlated with gluten index at Langdon-14 (r=0.47, *P*=0.0008). Peak width was associated with dough strength; a wide peak width means stronger gluten. Clarke et al. (2000) found a correlation between SDS sedimentation, mixograph and gluten index as an indicator of gluten strength.

Millers consider parameters, such as test weight, thousand kernel weight, and large kernel content, as indicators of semolina yield. No correlation between semolina yield and test weight were found. However, semolina extraction was positively correlated with 1000-kernel weight at Langdon-14 (r=0.31, *P*= 0.03), at Prosper-14 (r=0.44, *P*= 0.002), and at Prosper-15 (r=0.41, *P*= 0.004). While semolina extraction was positively correlated with large kernel content at Prosper-14 (r=0.30, *P*=0.04), and at Prosper-15 (r=0.42, *P*= 0.003). Other researchers have reported positive correlations between these parameters as well (Dexter et al., 1987; Halverson and Zeleny, 1988).

A negative correlation between ash and semolina brightness was found at Prosper-14 (r= -0.39, *P*=0.006) and Prosper-15 (r=-0.38, *P*=0.007) and negative correlated with yellow pigment content at Langdon-14. These results make sense since high ash content is related with reduced color of semolina (Cubbada, 1988). Oliver et al. (1992) found a high negative correlation between brightness (L*- value) and ash content in flour, but they did not found any correlation with b*- value.

A positive correlation between vitreousness and wet gluten was found at Langdon-14 (r=0.44, *P*=0.0019) and Prosper-15 (r=0.55, *P*=<0.0001), which probably relates to the relationship between protein content and vitreousness. El-Khayat et al. (2006) found a correlation between vitreous kernel and kernel protein and wet gluten in Syrian durum genotypes.

Semolina Dough Sheet Color

Dough Sheet CIE L*- Value at 0.5 h

Cultivar x harvest time interaction was not significant for dough sheet CIE L*- value at 0.5 h for Langdon-14, Prosper-14, or Prosper-15 (Table 22). Although not statistically comparable, dough sheet CIE L*-values at 0.5 h, averaged over cultivar and harvest time, were greatest for Prosper-15 (74.75), intermediate for Langdon-14 (74.21), and least for Prosper-14 (72.81).

Table 22. Analysis of variance for dough sheet color of twelve cultivars harvested four times at Langdon-14, Prosper-14, and Prosper-15.

	Source of Variation								
	Cult†	Harv†	CxH†	Cult	Harv	CxH	Cult	Harv	CxH
Parameter	df=11	df=3	df=33	df=11	df=3	df=33	df=11	df=3	df=33
	Langdon-14			Prosper-14			Prosper-15		
CIE L*- Value at 0.5 h	***	***	ns	***	**	ns	***	ns	ns
CIE L*- Value Difference	***	***	ns	***	ns	ns	***	**	ns
CIE b*- Value at 0.5 h	***	***	*	***	***	ns	***	***	ns
CIE b*- Value Difference	***	***	ns	**	***	ns	***	***	ns

†Cult = Cultivar main effect; Harv = harvest time main effect, and C x H, corresponds to their interaction.
df = degrees of freedom; ns = not significant.
*Significant at the 0.01 probability level.
** Significant at the 0.05 probability level.
*** Significant at the 0.001 probability level.

Harvest time main effect was significant for CIE L* semolina dough sheet color at Langdon-14 and Prosper-14, but not at Prosper-15 (Table 22). At Langdon-14 brightness decreased between the second (74.51) and third (73.89; LSD=0.31) harvest times. At Prosper-14, brightness was similar for dough sheet made from grain harvested at the first (73.09), third (73.07) and fourth (72.82) harvest times and was greater than for dough sheet made from grain harvested at the second (72.27; LSD=0.53) harvest time.

Cultivar main effect was significant for dough sheet CIE L*- value at 0.5 h for Langdon-14, Prosper-14, and Prosper-15 (Table 22). Rankings of cultivars differed at each environment (Table 23). At Langdon-14, Carpio and Joppa had the highest CIE L*- value (brightness) and Alkabo, Dilse, Lebsock, and Strongfield had the lowest brightness. At Prosper-14, Carpio and Grenora had the highest brightness and Dilse, Lebsock, Mountrail, and Strongfield had the lowest brightness. At Prosper-15, Grenora and Joppa had the highest brightness and Dilse had the lowest brightness.

Table 23. Means for dough sheet CIE L*-value at 0.5 h, averaged across harvest time at Langdon-14, Prosper-14, and Prosper-15.

Cultivars	Langdon-14	Prosper-14	Prosper-15
	----------0.5h ----------		
Alkabo	73.50	73.08	75.25
Ben	74.06	73.31	75.01
Carpio	75.37	74.12	75.53
Dilse	73.45	72.07	72.70
Divide	74.88	73.54	74.71
Grenora	74.21	74.00	75.79
Joppa	75.52	73.42	76.02
Lebsock	73.41	71.49	74.93
Mountrail	74.06	71.52	74.04
Pierce	74.12	72.48	74.57
Strongfield	73.21	71.87	73.80
Tioga	74.77	72.81	74.58
LSD (0.05)	0.55	0.91	0.49

Gluten index was positively correlated with dough sheet brightness at Langdon-14 (r=0.44, *P*=0.002), Prosper-14 (r=0.55, *P*=0.0001), and Prosper-15 (r=0.39, *P*=0.006). Conversely, wet gluten was negatively correlated with dough sheet brightness at Langdon-14 (r=-0.64, *P*=0.0001), Prosper-14 (r=-0.63, *P*=0.0001), and Prosper-15 (r=-0.80, *P*=0.0001). In addition, vitreous kernel content was negatively correlated with dough brightness at Prosper-15 (r=-0.39, *P*=0.006) and Langdon-14 (r=-0.58, *P*=0.0001).

Dough Sheet CIE L* Difference

Cultivar x harvest time interaction was not significant for dough sheet CIE L*- value difference for Langdon-14, Prosper-14, or Prosper-15 (Table 22). Loss of brightness, averaged over cultivar and harvest time, was greatest for Langdon-14 (-3.92), intermediate for Prosper-15 (-2.44) and least for Prosper-14 (-2.27). Harvest time main effect was significant for loss of dough sheet CIE L*- value at Langdon-14 and Prosper-15, but not at Prosper-14. Loss of dough

sheet CIE L*- value was not affected by delayed harvest at Prosper-14. At Langdon-14, loss of brightness was greatest at the first (-4.99) harvest time, intermediate at the second (-4.35) harvest time, and lowest at the third (-3.30) and fourth (-3.01; LSD=0.57) harvest times. At Prosper-15, loss of brightness was greater at first (-2.65), second (-2.72), and fourth (-2.42) harvest times than at the third (-1.95; LSD=0.55) harvest time.

Cultivar main effect was significant for dough sheet CIE L*- value difference for all three environments. Rankings of cultivars for loss of brightness differed for each environment (Table 24). At Langdon-14, Carpio had the greatest loss of brightness (-6.59) and Alkabo (-2.61), Ben (-3.52), Dilse (-2.86), Lebsock (-2.83), and Mountrail (-3.19) had the smallest loss of brightness. At Prosper-14, Carpio (-4.23) and Grenora (-4.15) had the greatest loss of brightness and Mountrail (-0.23) had the smallest loss. At Prosper-15, Carpio (-5.03) had the greatest loss of brightness and Ben (-1.25), Lebsock (-1.33), and Mountrail (-0.61) had the smallest loss.

At Langdon-14 and Prosper-15, change in brightness was negatively correlated with polyphenol oxidase activity (r=-0.60, P=0.0001 and r=-0.56, P=0.0001, respectively) and yellow pigment content (r=-0.52, P=0.0001 and r=-0.67, P=0.0001, respectively).

Table 24. Means for loss of brightness (CIE L* semolina dough sheet color) between 0.5 and 24 h, averaged across harvest time at Langdon-14, Prosper-14, and Prosper-15.

Cultivar	Langdon-14	Prosper-14	Prosper-15
	----------24-0.5h----------		
Alkabo	-2.61	-1.91	-1.56
Ben	-3.52	-2.00	-1.25
Carpio	-6.59	-4.23	-5.03
Dilse	-2.86	-1.67	-2.38
Divide	-3.85	-2.26	-3.19
Grenora	-4.15	-3.88	-3.16
Joppa	-3.75	-1.93	-2.39
Lebsock	-2.83	-2.05	-1.33
Mountrail	-3.19	-0.23	-0.61
Pierce	-3.98	-1.47	-2.51
Strongfield	-4.98	-3.09	-3.41
Tioga	-4.64	-2.51	-2.46
LSD (0.05)	0.99	1.11	0.95

Dough Sheet CIE b*-Value at 0.5 h

Cultivar x harvest time interaction was significant for dough sheet CIE b* value at 0.5 h at Langdon-14, but not at Prosper-14 or Prosper-15 (Table 22). In general, dough sheet b*-value (yellowness) declined with delayed harvest, except for Alkabo and Carpio, where their b*-values remained relatively constant across harvest times (Table 25). At Langdon 2014, the first decrease in dough sheet b*-value relative to the first harvest occurred at the second harvest with Divide, Grenora, and Lebsock, at the third harvest with Ben, Carpio, Dilse, Joppa and Pierce, and at the fourth harvest with Mountrail. Ranking of cultivars at each harvest time did not vary greatly. For each harvest time, Mountrail had the lowest dough sheet b*-value and Carpio and Joppa had the highest b*-value.

Harvest time main effect was significant for dough sheet CIE b*-value for Prosper-14 and Prosper-15 (Table 22). CIE b*- value, averaged over cultivar and harvest time, was greatest

for Prosper-15 (32.49), intermediate for Langdon-14 (31.19), and least for Prosper-14 (30.79) (Table 25). There was a reduction of yellow color after 0.5h at Prosper-14 between the first (31.91) and second (30.62) harvest time with b*- value similar for second, third (30.46), and fourth (30.16; LSD 0.53) harvest time. At Prosper-15, b*-value was similar for first (32.80) and second (32.97) harvest but declined between the second and third (32.28) and between the third and fourth (31.89; LSD 0.30) harvest time.

Table 25. Means for dough sheet CIE b*-value at 0.5 h as affected by cultivar x harvest time interaction at Langdon-14 and averaged across harvest time at Prosper-14 and Prosper-15.

	Langdon-14						
	Harvest times						
Cultivar	1	2	3	4	Mean	Prosper-14	Prosper-15
	----------0.5h----------						
Alkabo	32.30	32.24	31.40	31.69	31.91	31.99	33.69
Ben	29.05	29.12	28.04	27.54	28.43	29.46	29.78
Carpio	34.37	34.49	32.90	33.95	33.93	32.53	35.36
Dilse	33.74	33.22	32.58	32.35	32.97	32.09	33.77
Divide	32.88	31.46	30.95	31.12	31.60	32.48	32.58
Grenora	31.88	30.63	29.59	29.71	30.45	28.97	31.82
Joppa	35.42	35.31	33.29	33.83	34.46	32.16	34.46
Lebsock	30.03	28.45	28.16	27.32	28.49	29.03	29.24
Mountrail	27.87	26.86	26.91	26.46	27.02	27.64	28.61
Pierce	33.22	32.80	31.37	31.40	32.19	31.70	33.48
Strongfield	31.46	31.98	30.80	30.35	31.15	30.22	34.18
Tioga	32.57	32.25	31.21	30.61	31.66	31.19	32.86
LSD (0.05)	----------1.00----------					0.91	0.52

Cultivar main effect was significant for dough sheet CIE b*- value at 0.5 h for Prosper-14 and Prosper-15 (Table 22). Mountrail had the lowest b*-value at Prosper-14 (27.64) and Prosper-15 (28.61). Alkabo (31.99), Carpio (32.53), Dilse (32.09), Divide (32.48), Joppa (32.16) and

Pierce (31.70) had high b*-values at Prosper-14 and Carpio had the highest b*-value (35.36) at Prosper-15.

Dough sheet b*-value at 0.5 h was positively correlated with b*-value for semolina (r=0.94, *P*=0.0001, r=0.91, *P*=0.0001, r=0.94, *P*=0.0001) and yellow pigment content (r=0.92, *P*=0.0001, r=0.65, *P*=0.0001, r=0.93, *P*=0.0001) at Langdon-14, Prosper-14, and Prosper-15, respectively.

As mentioned above, delayed harvest had a negative effect on dough sheet yellowness. The early evaluation of pasta color base on semolina color is helpful in selecting durum lines with good pasta color. Fu et al. (2011) determined that the measure of CIE b* dough sheet at 0.5 h was enough to make predictions, the measure at 24 h (after storage) did not show any better prediction. During pasta processing, a portion of carotenoids pigments are oxidized, which results in a loss of yellow color (Matsuo et al., 1970). Fu et al. (2011) determined that dough sheet color showed a more accurate prediction of pigment content and their degradation by oxidation.

Dough Sheet CIE b*-Value Difference

Cultivar x harvest time interaction was not significant for dough sheet CIE b*-value difference for Langdon-14, Prosper-14, or Prosper-15 (Table 22). Harvest time main effect was significant for change in CIE b*-value (yellowness) for Langdon-14, Prosper-14, and Prosper-15. Loss of b*-value, averaged over cultivar and harvest time was greatest for Langdon-14 (-4.79), intermediate for Prosper-15 (-2.30), and least for Prosper-14 (-0.55). At Langdon 2014, loss of b*-value was greater at first (-5.67) harvest than at the second (-4.56), third (-4.47) or fourth (-4.47; LSD=0.42) harvest. Similarly, at Prosper-14, loss of b*-value was greater at the first (-1.13) harvest than at the second (-0.29), third (-0.20) or fourth (-0.02; LSD= 0.58) harvest. At

Prosper-15, loss of b*-value was greater at the first (-2.69) and third (-2.52) harvest times than at the second (-1.92) or fourth (-2.06; LSD=0.37) harvest times.

Cultivar main effect was significant for loss of dough sheet CIE b*-value at Langdon-14, Prosper-14, and Prosper-15 (Table 22). Ranking of cultivars varied with environment (Table 26). At Langdon-14 greatest loss of b*-value (yellowness) occurred with Alkabo, Dilse, Joppa and Pierce and lowest loss of yellowness occurred with Grenora. At Prosper-14, greatest loss of yellowness occurred with Ben, Dilse, Divide, and Pierce while interestingly, Alkabo and Grenora had an increase in yellowness. At Prosper-15, greatest loss of yellowness occurred with Dilse, Joppa and Pierce and lowest loss with Ben Lebsock and Mountrail. Fu et al. (2011) found that all the genotypes that increased in dough sheet color after 24 h storage also had better pasta yellowness. The cause of that increase can be linkage to the lipoxygenase gene type found on chromosome 4B of durum, the deletion of Lpx-B1.1 is associated with low LOX activity in semolina (Carrera et al., 2007).

According to the data almost all the parameters evaluated in dough sheet color are correlated with yellow pigment content. Yellowness at 0.5 h is positive correlated with yellow pigments at all three environments (Table 27).

In general delayed harvest seems to have a bigger impact on the yellowness than brightness of the dough sheet. Dilse, Strongfield and Lebsock tended to have lower dough sheet brightness at 0.5 h, while Carpio, Joppa, and Grenora tended to have increased brightness. The difference in brightness between 0.5 and 24 h was low for Mountrail and Ben and high for Carpio. Carpio and Joppa were the cultivars with the best dough sheet color at 0.5 h, while the worst was Mountrail. Interestingly, the difference in yellow color between 0.5 and 24 h showed

Dilse, Joppa, and Pierce as the cultivars with high losses, while Grenora at Langdon-14 and Prosper-14 had the lowest loss of yellowness.

Table 26. Means for loss of yellowness (CIE b* semolina dough sheet color) between 0.5 and 24 h, averaged across harvest time at Langdon-14, Prosper-14, and Prosper-15.

Cultivars	Langdon-14	Prosper-14	Prosper-15
	----------24-0.5h----------		
Alkabo	-5.45	0.10	-1.99
Ben	-4.12	-0.94	-1.37
Carpio	-4.10	-0.47	-2.67
Dilse	-5.55	-0.73	-3.20
Divide	-4.80	-1.54	-2.46
Grenora	-3.76	0.81	-1.57
Joppa	-5.50	-0.38	-3.57
Lebsock	-4.12	-0.20	-1.34
Mountrail	-4.80	-0.18	-1.35
Pierce	-5.59	-0.62	-3.65
Strongfield	-5.14	-0.47	-1.94
Tioga	-4.61	-0.27	-2.47
LSD (0.05)	0.73	1.00	0.64

Table 27. Correlations for dough sheet CIE L*-value (brightness) and CIE b*-value (yellowness) and grain yellow pigment content at Langdon-14, Prosper-14, and Prosper-15, ND.

Parameters	Langdon-14	Prosper-14	Prosper-15
CIE L* Dough Sheet Color at 0.5h	0.49***	0.33*	0.14
CIE L* Dough Sheet Color difference	-0.52***	-0.24***	-0.67***
CIE b* Dough Sheet Color at 0.5h	0.92***	0.65	0.93***
CIE b* Dough Sheet Color difference	-0.31*	0.31*	-0.58***

CONCLUSION

In general, delayed harvest caused a decline of yield, test weight, vitreous kernels, falling number, yellow pigment content, wet gluten content, and semolina and dough sheet b*-values (yellowness); while parameters, such as, large kernel size content, kernel CIE L*-value (brightness), and gluten index increased. Gluten index was the only parameter where delayed harvest had a clear positive effect at all three environments. Ranking of cultivars varied with environment and on the performed grain and semolina analyses. In general, for grain quality Carpio, Joppa, and Dilse showed the best results, Divide intermediate, and Strongfield and Lebsock showed the worst results. To semolina quality the best cultivars were Dilse and Carpio, intermediate were Joppa and Lebsock, and worst was Mountrail. Overall, delayed harvest affected grain and semolina quality of durum wheat. The effect was dependent on the environment and cultivar. Grain quality tended to be more affected than semolina quality.

INDUSTRIAL APPLICATION AND FUTURE RESEARCH

Based on the results, it was determined that delayed harvest has a negative impact on durum wheat quality. Thus, durum producers should avoid harvest delay, because loss of quality means loss of money to them.

More research is necessary to understand why the quality of protein increased during delayed harvest and why some cultivars varied in their response to delayed harvest. This information would be very useful for the farmers in North Dakota, as well as, breeders of durum wheat.

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APPENDIX

Table A-1. Correlations for grain quality parameters at Langdon-14

Parameter	Yield	Test Weight	Thousand Kernel Weight	Large Kernel Content	CIE L* Color	Vitreous Kernel Content	Kernel Protein Content	Falling Number	Yellow Pigment Content	Polyphenol Oxidase Activity
Yield	1.00	0.34*	-0.15	-0.41***	-0.35**	-0.27	-0.19	0.38	0.25	0.05
Test Weight	0.34*	1.00	-0.20	-0.23	-0.26	0.02	-0.22	0.44***	-0.20	-0.30*
Thousand Kernel Weight	-0.15	-0.20	1.00	0.69***	-0.03	-0.19	0.11	-0.27	-0.20	-0.13
Large kernel Content	-0.41***	-0.23	0.69***	1.00	0.04	-0.09	0.50***	-0.33*	-0.22	0.06
CIE L* Color	-0.35**	-0.26	-0.03	0.04	1.00	0.36**	-0.05	-0.21	0.03	-0.03
Vitreous Kernel Content	-0.27	0.02	-0.19	-0.09	0.36**	1.00	0.02	0.001	-0.40**	-0.36**
Kernel Protein Content	-0.19	-0.22	0.11	0.50***	-0.05	0.02	1.00	-0.36**	-0.03	0.37**
Falling Number	0.38**	0.44***	-0.27	-0.33*	-0.21	0.00	-0.36**	1.00	-0.13	-0.25
Yellow Pigment Content	0.25	-0.20	-0.20	-0.22	0.03	-0.40**	-0.03	-0.13	1.00	0.45***
Polyphenol Oxidase Activity	0.05	-0.30*	-0.13	0.06	-0.03	-0.36**	0.37**	-0.25	0.45***	1.00

*Significant at the 0.01 probability level.
** Significant at the 0.05 probability level.
*** Significant at the 0.001 probability level.

Table A-2. Correlations for grain quality parameters at Prosper-14.

Parameter	Yield	Test Weight	Thousand Kernel Weight	Large Kernel Content	CIE L* Color	Vitreous Kernel Content	Kernel Protein Content	Falling Number	Yellow Pigment Content	Polyphenol Oxidase Activity
Yield	1.00	0.32*	0.08	-0.24	-0.38**	-0.26	-0.56***	0.38**	0.51***	-0.13
Test Weight	0.32*	1.00	0.18	-0.12	-0.51***	0.18	-0.04	0.48***	0.32*	0.11
Thousand Kernel Weight	0.08	0.18	1.00	0.68***	0.24	-0.15	-0.36**	-0.21	-0.19	0.05
Large Kernel Content	-0.24	-0.12	0.68***	1.00	0.37**	-0.12	-0.10	-0.62***	-0.25	0.20
CIE L* Color	-0.38**	-0.51***	0.24	0.37**	1.00	0.01	-0.05	-0.64***	-0.62***	-0.22
Vitreous Kernel Content	-0.26	0.18	-0.15	-0.12	0.01	1.00	0.42***	0.07	-0.21	-0.10
Kernel Protein Content	-0.56***	-0.04	-0.36**	-0.10	-0.05	0.42***	1.00	-0.01	-0.14	-0.04
Falling Number	0.38**	0.48***	-0.21	-0.62***	-0.64***	0.07	-0.01	1.00	0.43***	-0.06
Yellow Pigment Content	0.51***	0.32*	-0.19	-0.25	-0.62***	-0.21	-0.14	0.43***	1.00	0.22
Polyphenol Oxidase Activity	-0.13	0.11	0.05	0.20	-0.22	-0.10	-0.04	-0.06	0.22	1.00

*Significant at the 0.01 probability level.
** Significant at the 0.05 probability level.
*** Significant at the 0.001 probability level.

Table A-3. Correlations for grain quality parameters at Prosper-15.

Parameter	Yield	Test Weight	Thousand Kernel Weight	Large Kernel Content	CIE L* Color	Vitreous Kernel Content	Kernel Protein Content	Falling Number	Yellow Pigment Content	Polyphenol Oxidase Activity
Yield	1.00	0.55***	0.46***	0.33*	-0.63***	0.41***	-0.19	-0.07	0.33*	0.33*
Test Weight	0.55***	1.00	0.56***	0.38**	-0.39**	0.14	-0.10	-0.40***	-0.02	-0.01
Thousand Kernel Weight	0.46***	0.56***	1.00	0.92***	-0.18	-0.13	0.05	-0.22	0.04	0.23
Large kernel Content	0.33**	0.38**	0.92***	1.00	-0.02	-0.32*	0.03	-0.02	0.05	0.37**
CIE L* Color	-0.63***	-0.39**	-0.18	-0.02	1.00	-0.70***	0.08	0.003	-0.30*	-0.11
Vitreous Kernel Content	0.41***	0.14	-0.13	-0.32*	-0.70***	1.00	0.03	0.24	0.20	0.13
Kernel Protein Content	-0.19	-0.10	0.05	0.03	0.08	0.03	1.00	-0.01	-0.18	-0.00
Falling Number	-0.07	-0.40***	-0.22	-0.02	0.00	0.24	-0.01	1.00	0.15	0.56***
Yellow Pigment Content	0.33*	-0.02	0.04	0.05	-0.30*	0.20	-0.18	0.15	1.00	0.49***
Polyphenol Oxidase Activity	0.33*	-0.01	0.23	0.37**	-0.11	0.13	0.00	0.56***	0.49***	1.00

*Significant at the 0.01 probability level.
** Significant at the 0.05 probability level.
*** Significant at the 0.001 probability level.

Table A-4. Means for test weight as affected by cultivar x harvest time interaction at Prosper-14.

	Harvest time			
Cultivars	1	2	3	4
	----------Kg hL----------			
Alkabo	80.1	77.9	77.8	76.7
Ben	81.0	78.3	78.7	78.2
Carpio	80.2	78.7	79.1	78.5
Dilse	80.0	77.0	78.3	77.1
Divide	79.3	77.5	78.0	77.2
Grenora	78.4	75.2	75.8	74.1
Joppa	79.6	76.7	77.5	76.5
Lebsock	80.2	77.5	78.0	77.4
Mountrail	77.6	75.8	76.3	75.3
Pierce	81.1	78.4	79.2	65.5
Strongfield	76.6	75.2	75.4	74.7
Tioga	79.6	76.3	76.5	76.0
LSD (0.05)	----------4.9----------			

Table A-5. Means for end height mixograph, averaged across cultivar at Langdon-14, Prosper-14, and Prosper-15.

Harvest times	Langdon-14	Prosper-14	Prosper-15
	----------cm----------		
1	5.7	5.1	5.1
2	5.5	5.1	4.9
3	5.4	5.1	5.6
4	5.4	4.6	5.4
LSD (0.05)	0.1	0.2	0.2

Table A-6. Means for end width mixograph, averaged across harvest time at Langdon-14 and Prosper-15 and cultivar x harvest time interaction at Prosper-14.

		Prosper-14				
		Harvest times				
Cultivar	Langdon -14	1	2	3	4	Prosper-15
	----------cm----------					
Alkabo	1.47	1.70	1.85	1.83	1.28	1.66
Ben	1.54	1.33	2.28	2.18	1.23	1.75
Carpio	2.49	2.40	1.65	1.68	1.28	1.75
Dilse	1.59	1.30	1.95	1.90	1.43	1.64
Divide	1.95	2.10	1.30	1.45	1.13	1.49
Grenora	1.61	1.35	1.80	1.43	1.18	1.44
Joppa	2.48	2.15	1.90	1.78	1.50	1.83
Lebsock	1.33	1.80	1.45	1.50	1.38	1.53
Mountrail	1.01	0.88	2.20	2.00	1.40	1.62
Pierce	1.86	1.65	2.00	1.65	1.28	1.64
Strongfield	1.99	1.73	1.28	1.08	1.15	1.31
Tioga	1.90	2.20	0.85	0.80	0.95	1.20
LSD (0.05)	0.30	----------0.63----------				0.54

Table A-7. Correlations for semolina quality parameter at Langdon-14.

Parameter	Semolina Extraction	Semolina CIE L*-value	Semolina CIE b*-value	Ash Content	Semolina Protein Content	Wet Gluten Content	Gluten Index	Peak Time	Peak Height	Peak Width
Semolina Extraction	1.00	-0.03	-0.24	-0.07	-0.10	0.03	-0.21	-0.08	-0.23	-0.16
Semolina CIE L*-valuer	-0.03	1.00	0.23	-0.21	-0.30*	-0.13	0.08	0.11	0.23	0.01
Semolina CIE b*-value	-0.24	0.23	1.00	-0.27	-0.25	-0.59***	0.66***	0.56***	0.16	0.46***
Ash Content	-0.07	-0.21	-0.27	1.00	0.27	0.06	0.02	0.07	-0.10	0.06
Semolina Protein Content	-0.10	-0.30*	-0.25	0.27	1.00	0.57***	-0.29	-0.24	0.08	0.00
Wet Gluten Content	0.03	-0.13	-0.59***	0.06	0.57***	1.00	-0.79***	-0.78***	0.26	-0.49***
Gluten Index	-0.21	0.08	0.66***	0.02	-0.29*	-0.79***	1.00	0.78***	-0.16	0.47***
Peak Time	-0.08	0.11	0.56***	0.07	-0.24	-0.78***	0.78***	1.00	-0.38***	0.31*
Peak Height	-0.23	0.23	0.16	-0.10	0.08	0.26	-0.16	-0.38***	1.00	0.24
Peak Width	-0.16	0.01	0.46***	0.05	0.00	-0.49***	0.47***	0.31*	0.24	1.00

*Significant at the 0.01 probability level.
** Significant at the 0.05 probability level.
*** Significant at the 0.001 probability level.

Table A-8. Correlations for semolina quality parameter at Prosper-14.

Parameter	Semolina Extraction	Semolina CIE L*-value	Semolina CIE b*-value	Ash Content	Semolina Protein Content	Wet Gluten Content	Gluten Index	Peak Time	Peak Height	Peak Width
Semolina Extraction	1.00	-0.22	0.14	0.10	0.14	0.03	0.01	-0.16	0.14	-0.09
Semolina CIE L*-value	-0.22	1.00	0.19	-0.32*	-0.22	-0.11	0.22	0.30*	-0.17	0.06
Semolina CIE b*-value	0.14	0.19	1.00	0.08	0.02	-0.34*	0.39**	0.13	0.11	0.17
Ash Content	0.10	-0.32*	0.08	1.00	0.28	0.20	-0.11	-0.30*	0.37**	-0.01
Semolina Protein Content	0.14	-0.22	0.02	0.28	1.00	0.53***	-0.38**	-0.24	0.44***	0.08
Wet Gluten Content	0.03	-0.11	-0.34*	0.20	0.53***	1.00	-0.73***	-0.26	0.45***	0.06
Gluten Index	0.01	0.22	0.39**	-0.11	-0.38**	-0.73***	1.00	0.14	-0.54***	-0.16
Peak Time	-0.16	0.30*	0.13	-0.30*	-0.24	-0.26	0.14	1.00	-0.21	0.27
Peak Height	0.14	-0.17	0.11	0.37**	0.44***	0.45***	-0.54***	-0.21	1.00	0.42***
Peak Width	-0.09	0.06	0.17	-0.01	0.08	0.06	-0.16	0.27	0.42***	1.00

*Significant at the 0.01 probability level.
** Significant at the 0.05 probability level.
*** Significant at the 0.001 probability level.

Table A-9. Correlations for semolina quality parameter at Prosper-15.

Parameter	Semolina Extraction	Semolina CIE L*-value	Semolina CIE b*-value	Ash Content	Semolina Protein Content	Wet Gluten Content	Gluten Index	Peak Time	Peak Height	Peak Width
Semolina Extraction	1.00	-0.18	-0.23	0.06	0.37**	-0.53***	0.19	-0.11	0.02	-0.21
Semolina CIE L*-value	-0.18	1.00	0.10	-0.38**	-0.29*	-0.08	-0.14	-0.06	-0.03	-0.12
Semolina CIE b*-value	-0.23	0.10	1.00	-0.13	-0.16	0.07	0.42***	-0.38**	0.05	0.18
Ash Content	0.06	-0.38**	-0.13	1.00	0.31*	0.26	-0.05	0.05	-0.20	0.01
Semolina Protein Content	0.37**	-0.29*	-0.16	0.31*	1.00	-0.22	0.15	0.10	-0.09	-0.19
Wet Gluten Content	-0.53***	-0.08	0.07	0.26	-0.22	1.00	-0.47***	0.04	-0.14	0.18
Gluten Index	0.19	-0.14	0.42***	-0.05	0.15	-0.47***	1.00	-0.20	-0.08	-0.01
Peak Time	-0.11	-0.06	-0.38**	0.05	0.10	0.04	-0.20	1.00	0.54***	0.28*
Peak Height	0.02	-0.03	0.05	-0.20	-0.09	-0.14	-0.08	0.54***	1.00	0.23
Peak Width	-0.21	-0.12	0.18	0.01	-0.19	0.18	-0.01	0.28*	0.23	1.00

*Significant at the 0.01 probability level.
** Significant at the 0.05 probability level.
*** Significant at the 0.001 probability level.

Table A-10. Correlations for semolina dough sheet at Langdon-14, Prosper-14, and Prosper-15.

Parameter	Langdon-14				Prosper-14				Prosper-15			
	CL† 0.5	Cb† 0.5	CL‡ diff	Cb‡ diff	CL 0.5	Cb 0.5	CL diff	Cb diff	CL 0.5	Cb 0.5	CL diff	Cb diff
CL 0.5	1.00	0.49***	-0.62***	-0.11	1.00	0.36**	-0.46***	0.64***	1.00	0.06	-0.18	0.01
Cb 0.5	0.50***	1.00	-0.40***	-0.44***	0.37**	1.00	-0.50***	0.14	0.06	1.00	-0.63***	-0.64***
CL diff	-0.62***	-0.40***	1.00	0.17	-0.46***	-0.50***	1.00	0.02	-0.18	-0.63***	1.00	0.27
Cb diff	-0.11	-0.44***	0.17	1.00	0.64***	0.14	0.02	1.00	0.01	-0.64***	0.27	1.00

†CL= CIE L* dough sheet color at 0.5h; Cb=CIE b* dough sheet color at 0.5h.
‡ CL diff= CIE L* dough sheet color difference (24-0.5h); Cb diff= CIE b* dough sheet color difference (24-0.5h)
*Significant at the 0.01 probability level.
** Significant at the 0.05 probability level.
*** Significant at the 0.001 probability level.