MANUFACTURING OPTIMIZATION OF NON-TRADITIONAL PASTA PRODUCTS

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The Supervisory Committee certifies that this ***disquisition*** complies with North Dakota State University's regulations and meets the accepted standards for the degree of

DOCTOR OF PHILOSOPHY

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ABSTRACT

A study was conducted to determine the effect of including non-traditional ingredients on the flow and agglomeration properties of flour formulations, the rheological behavior of pasta dough, and the physical and cooked quality of the spaghetti. The effect of including non-traditional ingredients on the chemical changes that occurred in pasta during pasta making and cooking was also investigated. The formulations used were semolina 100% (S), whole wheat flour 100% (WW), semolina-whole wheat flour (49:51) (SWW), semolina-flaxseed flour (90:10), whole wheat flour-flaxseed flour (90:10), and semolina-whole wheat flour-flaxseed flour (39:51:10). Flaxseed flour was incorporated as fine (FF) or coarse (CF) particles. Depending on the experiment, formulations were hydrated to different levels that ranged between 10 and 34%.

Study of the flow and agglomeration properties of the formulations indicated that samples containing whole wheat flour and or flaxseed flour were more cohesive and less prone to flow than samples with 100 or 90% semolina. Large agglomerate formation occurred with SFF hydrated beyond 30% moisture; whereas limited large agglomerate formation was detected in whole wheat samples with or without flaxseed flour hydrated up to 34% moisture.

Rheological experiments showed that traditional and non-traditional pasta dough behaved like a shear thinning fluid that can be described by the Power Law model. Increased hydration levels and/or presence of flaxseed flour in the dough formulation decreased the apparent viscosity of the dough, which correlated with reduced extrusion pressure, mechanical energy, and specific mechanical energy required to extrude the dough.

Better cooked quality was obtained for SWW than for WW, indicating that it is better to have some semolina in the formulation than replacing all the semolina with whole wheat flour.

Inclusion of flaxseed flour gave better results when adding the flaxseed to 90% semolina than when adding it to WW or SWW. No chemical interaction was observed between the different ingredients during pasta making and/or cooking.

Overall results indicated that the formulation-hydration combinations that optimized the processing and quality of non-traditional pasta products were: 30% for SFF, SCF, SWWFF, and SWWCF, 32% for S, WWFF, and WWCF, 33% for SWW, and 34% for WW.

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DEDICATION

I would like to dedicate this dissertation to grandpa Bernabé, uncle Pablo, grandma Aurea, uncle Erasmo, and grandpa Ciro. I could not be next to you in your final days, but you have been greatly missed and remembered at every single step of this journey.

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GENERAL INTRODUCTION

Pasta is a food product that has been traditionally made out of refined semolina (Agnesi 1996). Its culinary possibilities and inexpensive price have made this product a worldwide staple food. Nowadays, the current eating culture in developed countries abuses the consumption of refined cereal based products like pasta, and has pushed to the background the consumption of much nutrient rich food sources with healthier attributes (Nachay 2014). The increase of awareness of society about this problem is creating a new trend in which consumers are starting to demand healthier products without changing their eating habits. This situation has made the pasta industry to shift their ingredient composition based on refined semolina into a product formulation that includes ingredients not traditionally present in pasta that provide additional nutritional benefits on the consumer (Roda 2013). Some of the non-traditional ingredients chosen to fortify pasta products are whole wheat flour and flaxseed flour (Marconi and Carcea 2001).

Whole wheat flour and flaxseed flour contain phytochemicals that have been reported to have a beneficial effect on human health. Consumption of whole wheat flour has been reported to have an effect in reducing the risk of developing chronic diseases such as cardiovascular disease, type-2 diabetes, and some cancers (Hirawan 2010; Liu 2007). Likewise, consumption of flaxseed flour has been associated with anti-inflammation, antioxidant, hypocholesterolemic, anticarcinogenic effects and with attenuation of the postprandial insulin response among other benefits (Cardoso-Carraro et al 2012). Because of the aforementioned properties, whole wheat flour and flaxseed flour are considered two excellent fortification ingredients for pasta products.

Non-traditional ingredients like whole wheat flour and flaxseed flour have different physical properties and chemical composition than semolina. Semolina contains 2-3% of non-starch polysaccharides, 77-78% of starch, around 12-13% of protein, of which ~80% are gluten proteins, about 1-2% lipids, and a particle size distribution that ranges within 425 and 250 µm (Dick and Youngs 1988; Manthey and Twombly 2006). Whole wheat flour contains the bran and germ fractions of the wheat kernel, which are absent in semolina, and has a much smaller particle size than the semolina particles (<1 µm-200 µm) (Hoseney 1998). The presence of bran and germ has a diluting effect in the total starch and gluten protein content of the flour; however, the structural composition of the protein, lipid, and starch fraction is similar to semolina. Flaxseed differs greatly in amount and structural composition of protein, carbohydrates, and lipids in comparison to semolina. Flaxseed contains about 40% oil, 30% dietary fiber, 20% protein and lacks starch and gluten forming proteins (Daun et al 2003). These attributes give a completely new set of properties to the flour and dough formulations containing whole wheat and flaxseed flour that are going to be reflected in the processing and end the quality of the dry and cooked pasta product.

The transition from traditional to non-traditional pasta manufacturing represents a challenge to those factories or manufacturing lines that were originally designed to handle and process traditional pasta products. One of the challenges of using non-traditional ingredients in pasta making is the movement of ingredients in the processing line (Roda 2013). The easiness or difficulty encountered when moving cereal powders e.g. whole wheat flour, is ascribed to the flowing properties of the powder and the surface material over which the powder flows (Augenstein and Hogg 1978; Landillon et al 2008). In pasta manufacturing, the flowing properties of ingredients are very important since ingredient discharge into the mixing chamber, ingredient hydration-mixing, and pasta extrusion are continuous processes (Dalbon et al 1996). Therefore, any change in the flow of ingredients can affect the efficiency of the entire manufacturing process. Improper flowing of the ingredients leads to material build-up at the walls that can cause partial or complete disruption of the flow of ingredients (Twombly and Manthey 2006). Peleg (1983) indicated that knowing the flow properties of non-traditional ingredients is needed when designing new or retrofitting existing pasta production lines. Some of the tests more commonly used to determine the flowing properties of flour are the measurement of the angle of slide and angle of repose (Chang et al 1998).

Another problem derived from the utilization of non-traditional ingredients in pasta manufacturing is the formation of large agglomerates by non-traditional pasta ingredients during the hydration step prior to dough extrusion (de la Peña and Manthey 2012). Large agglomerate formation has been detected when non-traditional formulations have been hydrated at the same level that a traditional formulation (32%) (Yalla and Manthey 2006). This agglomerate formation has been attributed to the different hydration properties of non-traditional ingredients when compared to semolina (Kratzer 2007; Traynham et al 2007). Agglomerate formation has been associated with bridging that prevents the movement of hydrated ingredients between the high speed mixer and the main mixer and between the main mixer and the extrusion auger (de la Peña and Manthey 2012). Bridging of the hydrated material at any of these two connecting points will cause the disruption of the flow of material and the shutdown of the line. For this to be avoided, the optimum hydration level of non-traditional formulations needs to be identified.

The incorporation of non-traditional ingredients in pasta formulations has also been related with the alteration of the rheological behavior of the dough during extrusion (de la Peña et al 2014; Roda 2013). Yalla and Manthey (2006) reported that the presence of flaxseed flour or whole wheat flour, can alter the extrusion pressure, mechanical energy, extrusion rate and specific mechanical energy of pasta products. Chillo et al (2010) indicated that the changes in the machinability, processing conditions, and quality of non-traditional pasta products are influenced by the rheology of the dough during the extrusion process. Consequently, the study of dough rheology is important to the understanding of the flow properties of pasta dough during extrusion and to the evaluation of a given dough formulation to the extrusion processes.

The problems derived from the manufacturing of non-traditional pasta products mentioned above are related to the manufacturing process; however, the main challenge of incorporating non-traditional ingredients in a pasta product is to keep the quality of the dry and cooked product similar to the traditional product. Cooked pasta quality has been reported as the major factor influencing repeated purchasing and consumption of pasta by consumers (Hahn 1990). Several authors have reported the effect of a wide variety of non-traditional ingredients on the quality of pasta (Borneo and Aguirre 2008; Gallegos-Infante et al 2010; Hernández-Nava et al 2009; Martínez-Villaluenga et al 2010; Tudorica et al 2002). Cooked pasta quality is traditionally measured as cooking loss, cooked weight and cooked firmness, and presence of non-traditional ingredients can either improve or worsen these attributes (Dick and Youngs 1988). The degree to which these changes take place has been attributed to the chemical composition of the non-traditional ingredients and to their interaction with the starch and gluten proteins that hold together the matrix found in pasta products (Lamacchia et al 2010). Therefore, the study of the effect of non-traditional ingredients on the cooked properties of the pasta products should be considered when choosing a formulation.

The objectives of this research were to determine:

1. the flow properties of semolina and whole wheat flour with and without flaxseed flour on different surface materials;
2. the wet agglomeration properties of hydrated blends of semolina, whole wheat flour and flaxseed flour during pasta production;

3. the effect of dough hydration and formulation on rheological behavior of non-traditional pasta dough under extrusion conditions;
4. the effect of formulation and dough hydration level on the physical and cooked quality of pasta; and
5. the changes in the chemical components of non-traditional spaghetti that take place during cooking and how these changes reflect the quality of the cooked product.

It is important to remember that the best formulation-hydration combination that optimizes the manufacturing process does not necessarily yield the best pasta quality; henceforth, a compromise between these two parameters should be attained to improve the pasta making process and still provide a high quality product. Based on the information compiled in the five studies outlined above, the final goal of this research was to identify the best dough formulation-hydration combination for the manufacturing of non-traditional pasta products containing different amounts of semolina, whole wheat flour and flaxseed flour.

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LITERATURE REVIEW

Semolina

Semolina is the primary material obtained after milling durum wheat (*Triticum turgidum* L. var. *durum*) and it is the main ingredient for the manufacturing of pasta products. Durum wheat differs from common wheats in their genetic information. Durum is a tetraploid; whereas common wheats are hexploids (Bozzini 1988). Durum wheat is most commonly a spring wheat. Its grain is usually amber in color. Endosperm of durum wheat is high in carotenoid pigments which give the pasta its yellow color and consumer acceptance. Semolina particle size normally ranges between 425 and 250 µm (Dick and Youngs 1988). The particles of semolina have a yellowish color given by the high carotenoid content of the durum wheat endosperm. In order to be acceptable semolina cannot have an ash content higher than 0.80%.

Protein

Semolina typically contains 12 to 14% protein. Semolina protein is composed of about 20% metabolic proteins, mostly enzymes, and 80% storage proteins (Feillet 1988). The most important enzymes in semolina that affect pasta color are lipoxygenase (LOX), peroxidase (POX), and polyphenoloxidase (PPO) (Borrelli et al 2008). LOX is responsible for the degradation of semolina carotenoids, the pigment that gives semolina and pasta products their yellow color. Semolina with high LOX activity will result in a bleach dull color on pasta. POX and PPO are also responsible of the formation brown compounds that decreases the yellowness of pasta products.

Another important enzyme in semolina is α-amylase. This enzyme is absent in mature grain and it appears upon germination. It a an endosplitting enzyme that hydrolyzed the α-1,4 linkages of starch molecules. α-amylase activity induces higher level of sugars, particularly maltose, in spaghetti and subsequently increases the losses of solids in cooking water (Feillet 1988).

Semolina storage proteins are composed of two classes of proteins, gliadins and glutenins. The gliadins are soluble in 70% aqueous ethanol, and they have been divided into subgroups (α-, β-, ɤ-, and ω-gliadins) by electrophoretic mobility at low pH values. The ω-gliadins are unique with respect the other subgroups in that they do not contain sulfur-containing amino acids (Shewry et al 1986). The main stabilizing forces in the α-, β-, and ɤ-gliadins are covalent disulfide bonds and non-covalent hydrogen bonds, whereas ω-gliadins are stabilized by hydrophobic interactions (Tatham and Shewry 1985). The α-, β-, and ɤ-gliadins have molecular weight in the range of 30-40 kDa whereas ω-gliadins have higher molecular weight ranging within 60-80 kDa. There is also a high molecular weight fraction of gliadin with a molecular range of 100-200 kDa, which is composed of smaller subunits and are of similar weight to low molecular weight glutenin subunits (Bietz and Wall 1980).

The molecular weight of native glutenin is estimated to be several million; therefore, this protein is normally reduced to monomer ranging from 10 to 130 kDa to make possible its study. Seilmeier et al (1987) indicated that the reduction of the glutenin protein gives rise to the formation of subunits that can be classified according to their apparent molecular weight determined by SDS-PAGE as high molecular weight subunits, middle molecular weight subunits and, low molecular weight subunits.

The most important property of gliadin and glutenin is their ability to form gluten, the dough forming protein fraction in wheat (Feillet 1988). Gliadins protein provide cohesion to the gluten matrix and are responsible for the gluten extensibility and viscosity. Glutenin on the other hand are responsible for the strength and elastic properties of gluten.

Carbohydrates

Carbohydrate content in semolina can be up to 83%. Carbohydrates found in semolina can be divided in starch and non-starch polysaccharides.

Starch polysaccharides

Durum wheat starches contain between 0.21-0.29 % ash, 0.44-0.57 % protein, 0.39-0.54 % lipid, and 26.2-28.5 % amylose (Vansteelandt and Delcour 1999). The amylose content detected in durum wheat is slightly higher than the one found in common wheat starches (Dexter and Matsuo 1979; Vansteelandt and Delcour 1999). The remainder of the composition corresponds to amylopectin. Amylose is generally assumed to be a linear polymer of α-D-glucose units linked through an α-1,4 linkage. Although this polymer is generally assumed to be linear, there are amylose molecules that have a low degree of branching. The amylose branches are also composed of glucose units linked together through α-1,4 linkages (Buléon et al 1998). The branches are linked to the main backbone chain through α-1,6 linkages. These branches on amylose are so long and so few in comparison to the length of the polymer that it tends to behave like an unbranched molecule (Whistler and BeMiller 1997).

The axial-equatorial position coupling of the 1,4-linked α-D-glucose units is the amylose chains gives the molecules a right-handed spiral or helical shape. The interior of the helix contains predominantly hydrogen atoms and is lipophilic, while the hydroxyl (OH) group are positioned on the exterior of the coil. The lipophilic nature of the interior of the amylose helix allows it to form complexes with linear hydrophobic portions of molecules that fit within the lumen of the helix e.g. iodine, and mono and diacylglycerols (Matsuo et al 1992).

Like in amylose, amylopectin is composed of α-D-glucose units linked through an α-1,4 linkage (Buléon et al 1998). Amylopectin is branched to a much greater extent than is amylose, with 4-5% of the glycosidic bonds being α-1,6 bonds. Amylopectin is thought to be randomly branched. The molecule has three types of chains: A-Chains, composed of glucose linked α-1,4, and are branch chains; B-chains, composed of glucose linked α-1,4 and α-1,6, and are branch chains that also contain branches; and C-chain, the original chain that is made up of glucose linked by α-1,4 and α-1,6 linkages and that has a reducing group.

Starch is biosynthesized inside amyloplasts as semi-crystalline granules. Starch granules are composed of amylopectin with repeating regions that are highly branched and sparsely branched. The highly branched regions are polycrystalline and the sparsely branched regions are noncrystalline (Thomas and Atwell 1999a). The branches of amylopectin occur as packed double helices. These double helices form the crystalline regions of the starch granule in the dense layers of starch granules that alternate with amorphous layers. Vansteelandt and Delcour (1999) showed through NMR studies that durum wheat starches have a lower relative double helix content than common wheat starches. Amylose is generally found in the noncrystalline regions and can diffuse out of partially water-swollen granules. The radial arrangement of starch molecules in granule gives it a quasicrystalline nature. Starch granules can have different polymorphic types and degrees of crystallinity (Buléon et al 1998).

When starch is heated in the presence of water the order within the starch granules is lost. This phenomenon is known as gelatinization (Thomas and Atwell 1999b). Evidence for the loss of the organized structure includes irreversible granule swelling, loss of birefringence, and loss of crystallinity. Leaching of amylose occurs during gelatinization, but some leaching of amylose also occurs at temperatures below the gelatinization temperature due to its location in noncrystalline regions and the fact that it is a relatively small and linear molecule. Durum wheat starches start gelatinizing at about 50°C. Gelatinization continues for a temperature range of 12.5 °C, being the peak at about 55.5-60.0 °C, as measured with Differential Scanning Calorimetry (DSC) (Vansteelandt and Delcour 1999). Soulaka and Morrison (1985) recorded that durum wheat starch gelatinizes at a slightly lower temperature than starch from other wheat classes when evaluated by DSC. These finding may relate to a less-compact granule structure of durum starch (Lintas 1988).

The viscosity of gelatinized durum wheat starch has been reported to be higher than that of gelatinized spring wheat starch (Vansteelandt and Delcour 1999). These researchers also reported differences between the gelatinization behavior determined by Differential Scanning Calorimetry and Rapid Visco-Analyzer measurements and by the determination of amylose leaching of durum wheat starches and common wheat starches e.g. the points of initial swelling during gelatinization and granule rigidity. The authors concluded that these differences in gelatinization could be explained in terms of lipid content and amount of amylose-lipid complexes since durum wheat starches contained more crude fat than the soft wheat starches they studied.

On cooling, some starch molecules begin to re-associate, forming a precipitate or gel (Riva et al 2000). This process is called retrogradation. Retrogradation involves the formation of junction zone between both amylose and amylopectin. Both gelatinization and retrogradation processes are irreversible and involve a complete change in the physical and structural properties of the native starch granules.

In addition to starch gelatinization there are other changes to starch structure that can occur in presence of water and heat. Annealing and heat-moisture treatment are two hydrothermal treatments that modify the physicochemical properties of starch, without destroying granular structure (Jacobs and Delcour 1998). According to Jacobs and Delcour (1998) these treatments involve incubation of starch granules in excess (<60% w:w) or at intermediate (40-55% w:w) content (annealing) or at low moisture (>35% w:w) levels (heat-moisture treatment) for period of time, at a temperature above the glass transition temperature but below the gelatinization temperature. Other authors (Collado and Corke 1999) stated that annealing represents physical modification of starch slurries in water at temperatures below gelatinization whereas heat-moisture treatment refers to the exposure of starch to higher temperatures at very restricted moisture content (18-27%). Similar to gelatinized starch, annealed and heat-treated starch granules also damage the granular integrity and the loss of birefringence of the starch giving the resulted granules a completely new set of properties.

Non-starch polysaccharides

Non-starch polysaccharides in semolina are mostly composed of arabinoxylans (Ingelbrecht et al 2000). Arabinoxylans account for up to 65% of wheat endosperm cell wall dry matter (Turner et al 2008). They consist of a linear β-1,4-linked xylan backbone to which α-L arabinofuranose units are attached as residues via α-1,3 and or α-1,2 linkages (Izydorczyk and Biliaderis 1995). Non-starch polysaccharides have high-water biding activity.

Lipids

Semolina contains 1 to 2% lipid (Hoseney 1998). The lipids of the endosperm, and hence of the semolina, comprise membrane components, lipids associated with starch, and some spheronomes (Simmonds 1989). Spheromones are storage droplets of lipid dispersed in the endosperm. Starch lipids are those contained within the starch granules. These lipids contain phosphate groups and are termed lysophospholipids. Their structure allows them to form a complex with amylose by attaching the fatty acid group in the core of the amylose helix. Carotenoids pigments like xanthophylls and lutein are found in semolina and give pasta its characteristic yellow color and contribute towards its lipid content.

Whole Hard White Wheat Flour

Hard white wheat flour is the product obtained after the milling of *Triticum aestivum* L. Hard white wheat is normally used in whole-wheat and high-extraction flour applications, such as pan breads, flat breads and specialty noodles (Ranson et al 2006). The protein, carbohydrate, and lipid composition of the hard white flour is very similar to that found in semolina. Because both durum wheat and whole wheat flour belong to the same plant species, *Triticum spp.*, the structure of the protein, carbohydrates, and lipids found in white wheat flour are similar to that in semolina. The main difference in the composition of semolina and hard white winter wheat flour is the level of carotenoid pigment found in the endosperm, with semolina containing more than white wheat.

However, when whole wheat flour is considered, then the presence of the bran and germ of the kernel in the flour will affect the overall composition of the flour. The proteins in germ and bran are mainly composed of albumins and globulins, which might result in dilution of gluten proteins and starch content of the flour (Hirawan 2010). Both bran and germ are rich in phytochemicals that are absent or in low content in the refined flours. The most important groups of phytochemicals found in the bran and germ of wheat can be classified as phenolics, carotenoids, vitamin E compounds, lignans, β-glucan, and inulin (Hirawan 2010; Liu 2007; Pussayanawin and Wetzel 1987). These phytochemicals have been reported to have beneficial effect in reducing the risk of developing chronic diseases such as cardiovascular disease, type 2 diabetes, and some cancers (Hirawan 2010; Liu 2007).

Wheat bran

Wheat bran is the outer layer of the wheat kernel. It represents about 15% of the wheat seed and is composed predominantly of non-starch polysaccharides (∼58%), starch (∼19%) and crude protein (∼18%), with the non-starch polysaccharides being primarily ∼70% arabinoxylans, ∼24% cellulose and ∼6% β-(1,3) (1,4)-glucan (Maes and Delcour 2002). It has been reported that bran has the capability of absorbing several times its own weight in water (Seyer and Gélinas 2009). The main consequence of this effect is that when bran is incorporated into a flour, much more water is needed to be added to the flour in order to compensate for the amount of water absorbed by the bran (Roozendaal et al 2012; Seyer and Gélinas 2009).

Wheat germ

Wheat germ is composes 2.5-3.5% of the kernel. The germ is quite high in protein (~25%), sugar (~18%), oil (~9%), and ash (~5%) (Hernot et al 2008; Hoseney et al 1998). It contains no starch but it does contain high levels of B vitamins, vitamin E, and contains many enzymes (Hoseney et al 1998).

Flaxseed Flour

Flaxseed is the seed of the plant *Linum usitatissimum* L. Flaxseed has a flat and oval shape pointed in one end and is on average 5mm in length, 2.5mm in width and 1.5mm in thickness (Daun et al 2003). Flaxseed is made up of 55% cotyledon, 36% seed coat and endosperm (hull), and 4% embryo. The proximate composition of flaxseed is about 40% oil, 30% dietary fiber, 20% protein, 4% ash, and 6% moisture. In flaxseed milling, the entire seed is milled together. Unlike milling wheat for refined flour, the particles are not separated from each other like the bran, germ and flour. Therefore, the composition of flaxseed flour is the same as the composition of the whole flaxseed seed.

The use of flaxseed as a functional food has received considerable attention in North America due to the health-related benefits attributed to its consumption. The presence of n-3 fatty acids, soluble fibers, vitamin E, lignans, and other phenolic and peptide compounds found in flaxseed have anti-inflammation, antioxidant, hypocholesterolemic, anticarcinogenic effects, and helps in vessel relaxation and attenuation of the postprandial insulin response among other among others benefits (Cardoso-Carraro et al 2012).

Protein

Flaxseed does not have gluten forming proteins, making it unable to form a gluten matrix. Flaxseed can contain from 10 to 37% of protein, depending on the genetics and environmental conditions under which the seed develops (Oomah and Mazza 1993). Proteins in flaxseed are about 20% albumins (1.6S and 2S), whose molecular weight ranges between 16-17 kDa, and 80% legumin-like proteins (11S and 12S), whose molecular weight ranges between 252-298 kDa (Madhusudhan and Singh 1985). The legumin-like fractions is richer in sulfur amino acids than does the albumin proteins.

Carbohydrates

Flaxseed may contain a small percentage of soluble sugars of about 1-2% (Daun et al 2003). The majority of the carbohydrates present in flaxseed are of the group that are resistant to the action of human digestive enzymes, and are considered to be dietary fiber. Flaxseed carbohydrates can be divided into soluble and insoluble fiber, which account for about 9% and 20% of the total seed composition, respectively.

The outermost layer of the flaxseed seed coat or hull is also known as mucilage. Mucilage corresponds to approximately 8% of the seed weight (Oomah et al 1995). The flaxseed mucilage is composed primarily of two polysaccharides: a neutral and an acidic polymer. The neutral polymer consists of a β-1,4-linked xylose backbone which contain side chains of arabinose and galactose at positions 2 and or 3. The acidic polymer consists of α-1,2-linked D-rhamnopyranose residues and 1,4-linked D-galactopyranosyluronic acid residues with side chains of fucose and galactose (Oomah et al 1995). Flaxseed mucilage has potential to be used as a food hydrocolloid due to its thickening and emulsifying properties (Chen et al 2006). Some of health benefits attributed to the consumption of the flaxseed mucilage are the lowering effect of the cholesterol/triglyceride lipid level in the blood, blood glucose attenuation, fat absorption by fecal excretion, and protective effect against diabetes risk (Cardoso-Carraro et al 2012).

Lipids

Flaxseed is characterized for having about 45% oil and 55% meal on a dry basis (Daun et al 2003). The cotyledons are the major oil storage tissue, containing about three quarters of the seed oil. About 22% of the oil is found in the seed coat and 3% in the embryo. The most predominant lipid classes are acylglycerols, mostly in the form of triacylglycerols, and fatty acids stored in oil bodies. Sterol, including Vitamin E, esters, glycolipids, and phospholipids make up about 5% of the total lipids.

Flaxseed has very high levels of α-linolenic acid (ALA, 18:3 n-3) usually representing more than 50% of the total fatty acids (Gonçalves de Oliveira et al 2012). Other fatty acids include palmitic, stearic, oleic, and linoleic acid.

ALA is the major n-3 polyunsaturated fatty acid in the human diet. Consumption of ALA has been associated with having an effect in: reducing the effect of obesity, reducing the incidence of cardiovascular diseases and diabetes, controlling the levels of blood sugar, and reducing the incidence on hormonal dependent cancers e.g. prostate and breast cancers (Cardoso-Carraro et al 2012). In addition, ALA it is also related to the modulation of the immune response, inhibiting excessive inflammatory responses and being beneficial in allergic conditions (Saini et al 2010).

Minor components

Potassium and phosphorus are the major mineral components of flaxseed and also contains a range of vitamin B (Daun et al 2003); however, the most important minor component in flaxseed are the phenolic compounds known as lignans. The lignan component in flaxseed of particular interest is secoisolariciresinol diglucoside which can be present on the order of 1-10µM/g seed. Lignan consumption reduces cardiovascular risk, has anticarcinogen properties, and inhibits the development of some types of diabetes (Mueller et al 2010). Health benefits of flaxseed lignans reside in their antioxidant capacity as sequestrators of hydroxyl radicals, and as estrogenic compounds due to their structural similarity to 17-β-estradiol. The latter is a human sex hormone and steroid important in the regulation of the estrous and menstrual female reproductive cycles.

Flow Properties of Powders

Wheat powders are used as raw materials in the production of cereal-based food products i.e. pasta. Characterization of food powder flowability is required for predicting powder flow from hoppers in small-scale systems or at the industrial scale from storage silos or bins dispensing into powder mixing systems or packaging machines (Juliano and Barbosa-Cánovas

2010). Part of the design of processing plants involves ensuring the satisfactory flow of materials, which is essential to the profitability of the process.

Lee and Yoon (2015) reported that most of the problems associated with powders handling take place when discharging material from a storage hopper without interruption and at the required rate. These problems are caused by failure to incorporate accurate flowability measurements into the design. The end result is frequent stoppage of the process, involving costly loss of production time and inefficient use of staff to restore the flow (Abu-hardan and Hill 2010).

Peleg (1978) described that the forces opposing flow are friction, attraction between particles (cohesion), attraction between the particles and system walls (adhesion), and mechanical resistance or interlocking i.e. formation of stable arches. Liquid layers on the surface of particles promote cohesion by creating a meniscus between the particles (Yan and Barbosa-Cánovas 1997).

Abu-hardan and Hill (2010) described the flow of powders as a complex phenomenon in which both the powder characteristics and physical and chemical features of the system determine the behavior of the powder flow system. Physical characteristics that determine the flowability of a given powder include particle surface properties, and particle shape and size distribution (Yan and Barbosa-Cánovas 1997). Regarding the chemical features of the system, flowability can be affected by the amount of free and associated water inside each particle. Other components such as fats, sugars, proteins, and fibers also determine the flowability of a powder.

Juliano and Barbosa-Cánovas (2010) indicated that the flow characterization is not only relevant for equipment design, storage location of bulk solids, and transporting or otherwise handling of solids, but is also needed to fulfill the requirements of quality control and for modeling processes. Classic methods that have been utilized for the determination of the flow properties of powder are the angle of repose and the angle of slide (Chang et al 1998). The static angle of repose is defined as the angle at which a material will rest on a stationary heap or the angle θ formed by the heap slope and the horizontal when the powder is dropped on a platform (Chang et al 1998; Fitzpatrick et al 2004). The angle of slide is the minimum angle of incline (measured relative to the horizontal) that will allow a bulk solid to flow from under its own weight. This angle is presumed to be useful in designing stationary chutes, but its measurement has not been designated as standard as reported by Peleg (1978). The value measured is thought to be highly influenced by the construction material of the chute, the amount of material used, and humidity.

Wet Agglomeration of Powders

Wet agglomeration is a process widely used in the drug, chemical, and food industry. The process consists in wetting a native powder with a liquid binder during a mixing stage (Iveson et al 2001). Schubert (1981) reported that the cohesion of agglomerates depends on the interactions established between native particles inside their structure. The adhesion forces are either capillary forces (liquid bridges) or viscous forces (sinter bridges). Particle adhesion strength depends on the surface viscosity, the contact angle of the liquid/solid system, the surface tension of the liquid at the particle surface, the contact time, and the kinetic energy of the particles during the agglomeration process (Rondet et al 2013).

The theory of agglomerate formation has been studied by several researchers (Iveson et al 2001; Rondet et al 2013; Saad et al 2008; Schubert 1981). When a single droplet hits a powder bed, the droplet is drawn into the powder by capillary forces. There are two stages in the growth of agglomerates: Initially fast growth takes place during wetting of the powder particles by a droplet, which results in the formation of an initial agglomerate (Schaafsma et al 1998). The whole void volume of the initially formed agglomerate is filled with binder solution. The second stage of growth is initiated by liquid transport from the initially formed agglomerate to the free particles surrounding the agglomerate. Growth of the agglomerate takes place by the attachment of new layers of particles (Iveson et al 2001). If a pore at the surface of an agglomerate is sufficiently filled with binder liquid then a free particle can reach the liquid phase on the pore and a liquid bridge can be formed. During growth, the liquid is drawn from the voids inside the agglomerate to the newly wetted pores formed by the attached particles at the surface of the agglomerate. The liquid flow is induced by the capillary pressure difference between the pores inside the agglomerate and the pores with a lower saturation near the surface of the agglomerates. According to Schaafsma et al (1998), the second growth stage takes place until the saturation reaches a minimum value also called the wetting saturation.

Wet agglomeration is a phenomenon that takes place during the hydration of semolina as the preparation step for the manufacturing of couscous and pasta products (Barkouti et al 2014; Hahn 1990). When the agglomeration has taken place, the semolina structure formed can be classified based on their moisture content. Saad et al (2011) reported that when the water content of semolina particles is lower than the capillary condensation water content, around 0.21 g water /g dry matter, structures are native particles because there is not enough water to generate capillary bonds between grains. The same author also stated that when the water content of semolina particles is higher than the capillary condensation water content and lower than the plastic limit value, around 0.52 g water /g dry matter, structures are nuclei or agglomerates. The water content promotes the growth of agglomerates that result from the association of nuclei. According to Iveson et al (2001) the nuclei are the first agglomerated structures resulted from the association of several grains when a liquid drop falls on the dry powder. When the structure's water content is higher than the plastic limit but lower than the liquid limit then the structures are considered dough pieces.

The characterization of the agglomerate mechanical properties is considered a relevant way of describing the structure of agglomerate powders (Barkouti et al 2014). The study of agglomerate mechanical properties can be determined by compression tests performed on individual agglomerates (Rondet et al 2013). The determination of mechanical properties of agglomerates can also be considered under the same approach than powders, by measuring the mechanical behavior of an agglomerates bed bulk i.e. bulk density.

Capillary Rheology

Cereal chemists have used the rheological properties of durum wheat for many years to evaluate the potential of durum wheat varieties for pasta production (Hahn 1990). These methods include the use of the mixograph, farinograph, extensiograph, alveograph, and amylograph, which mainly measure the gluten strength of the product (Bahnassey and Khan 1986; Baiano et al 2011; Doxastakis et al 2007; Kovacs et al 1997). Many researchers have shown that gluten strength is an important factor in making high quality products from durum wheat (Cubadda et al 2007; Matsuo and Irvine 1970; Matsuo et al 1972). However, these tests have not proved to be satisfactory in predicting the performance of semolina or other ingredients during the extrusion process (Hahn 1990).

Capillary rheology allows the study of the flow of pressure driven matter through pipes and extrusion dies (Macosko 1994); therefore, it can simulate flow of the dough during pasta extrusion. Capillary rheology can then be considered as an alternative to having to make the pasta for testing the viability of newly developed pasta formulations (de la Peña et al 2014). This technique can provide information about shear stress and shear rate of the pasta dough during extrusion, which are related to extrusion pressure and extrusion rate, respectively (Le Roux and Vergnes 1995). Consequently, this is a technique that can also be applied to study the rheological properties of pasta dough during extrusion. Knowing the rheological behavior of pasta dough is important because it affects the required extrusion pressure, extrusion rate, mechanical energy and specific mechanical energy requirements during the extrusion process (de la Peña et al 2014). Fluctuation of these parameters can cause problems downstream in the processing line. For example, alteration of extrusion rate such as too much or too little product needing to be processed through the drier. Using this technique will help identify ingredient effects on pasta extrusion and would allow the pasta processor to be pro-active in solving potential problems.

The schematic representation of capillary rheometer can be observed in Figure 1. The temperature of the capillary rheometer can be adjusted to the standard temperature expected to occur in the extrusion barrel of a pasta extruder (≈45 °C). The pre-hydrated pasta formulation is introduced in the pre-heated barrel, and a piston pushes the test sample e.g. dough, through the capillary die located at the very bottom of the heated barrel. The opening and length of the die can be customized so it simulates the opening expected to have in a pasta extrusion die. The data collected from the capillary rheometer will determine how much pressure needs to be exerted on the dough for it to extrude at a specific extrusion rate (Macosko 1994).

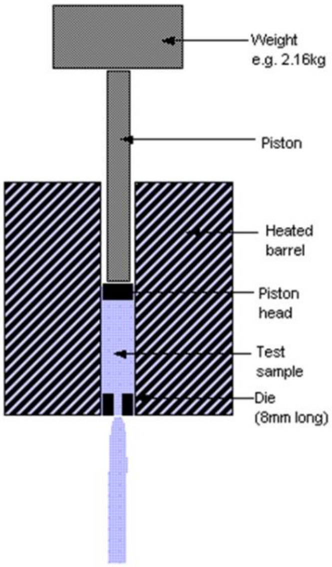

Figure 1. Diagram of a capillary rheometer.
http://www.adhesivestoolkit.com/Docs/test/Physical%20Analysis%20-%20Capillary%20Rheometers.xtp

Pasta Processing

Traditional pasta

Pasta processing can be divided into four stages: mixing, kneading, shaping and drying (Manthey and Twombly 2006). A pasta press is composed of an ingredient (semolina) feeding system, an ingredient hydration system (typically associated with a pre-mixer), a main mixer, and an extrusion barrel with an auger, extension tube, extrusion head, and pasta die (Figure 1).

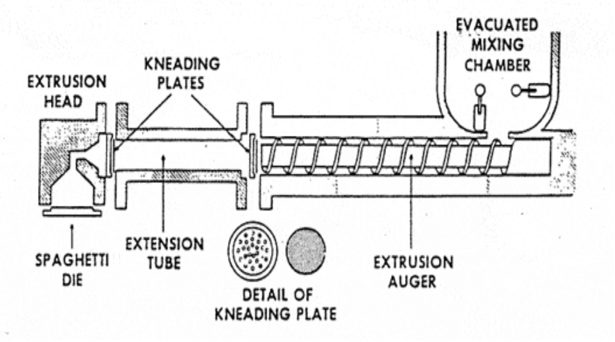

Figure 2. Diagram of a semi-commercial pasta extruder.

Before processing, semolina and warm water (35 to 40°C) are mixed in a paddle mixer (pre-mixer) (Dalbon et al 1996). Enough water is added to bring the moisture content of the semolina to 30-32%. The wetted semolina is then discharged then into the main mixer. The main mixer contains two horizontals shafts that rotate in opposing directions. The function of the paddle mixer is to hydrate the semolina gradually and homogenously. Mixing only wets the semolina particles and does not provide enough energy to develop gluten interactions (Hahn 1990). The main mixing chamber and rest of the extrusion line is kept under vacuum to remove air trapped in the dough. If the air is not removed small air bubbles can appear in the finish product giving a chalky appearance to the product and reducing its mechanical strength (Feillet and Dexter 1996).

Wet, mixed semolina is fed from the mixing chamber into the extrusion barrel. Inside the extrusion barrel there is an auger whose main purpose is to knead and convey the wetted semolina. A pressure gradient is formed as the hydrated material moves along the auger. This pressure gradient is initially formed due to the compaction of the dough against the kneading plate that is located at the end of the auger (Manthey and Twombly 2006). As the compressed dough goes through the kneading plate significant dough development occurs. The kneading plate is a perforated plate that situated between the end of the auger and the extrusion head. After going through the kneading plate the dough enters into an extension tube that allows the dough to relax before being extruded through the die. The shape of the die orifice will determine the shape of the pasta product. Parameters most commonly measured during pasta extrusion are extrusion temperature (°C), mechanical energy (J/s), extrusion rate (g/s) and specific mechanical energy (J/g). The latter is calculated by dividing mechanical energy by extrusion rate (Manthey et al 2004).

After the pasta has been extruded is can be fresh packaged or dried and the packaged. The latter is a more common practice. The pasta drying is generally divided into three stages: predrying, final drying, and cooling/stabilizing (Manthey and Twombly 2006). During drying the moisture content of the product changes from 30-32% to around 12%.

Non-traditional pasta

Inclusion of non-traditional ingredients in pasta composition provokes changes in the processing of the product in a greater or lesser degree depending on the type of ingredient, the physical state of the ingredient, e.g. particle size, fractionation, thermal treatment, fermentation, etc., and the degree of semolina substitution (Chillo et al 2008).

Particles of non-traditional ingredients have different particle size, shape, and surface than semolina particles. This creates a challenge to make even and homogeneous distribution of the non-traditional ingredients and semolina dry mixtures before hydration (Kuakpetoon et al 2001). Depending on the particle size and nature of the non-traditional ingredient, optimum mixing time of mixtures of semolina - non-traditional ingredients will be longer or shorter (Kuakpetoon et al 2001; Neel and Hoseney 1984).

Presence of non-traditional ingredients normally reduces or increases dough strength, increases water absorption in dough, and might also influence the mixing times prior to the extrusion step due to different rate of water distribution among the new ingredients (Haber et al 1978; Kordonowy and Youngs 1985; Manthey et al 2004; Yalla and Manthey 2006). Often the extrusion parameters of composite pasta yield different values (sometimes higher, sometimes lower), than compared to 100% semolina pastas (Manthey et al 2004; Yalla and Manthey 2006).

Pasta Quality

Physical quality

There are two main physical parameters that determine the quality of dried pasta: color/aesthetic appearance and mechanical strength (Feillet and Dexter 1996). Pasta color is the result of yellow and brown components. During pasta drying, the protein-carbohydrate reactions, which involve the terminal amino group of free amino acids, proteins and reducing sugars, Maillard reactions, lead to a change in color of pasta by non-enzymatic reactions (Acquistucci 2000). High occurrence of this reaction in pasta making is undesirable because it increases the redness and brownness of the pasta. Carotenoid pigments are responsible for the yellow color of pasta; whereas, naturally colored protein and enzymatic oxidation reactions are responsible for the undesired brown color (Fu et al 2011). These two color components refer only to traditional pasta products. Non-traditional pasta products rarely have yellow pigments and therefore the color requirements are more related to color evenness and brightness than to its yellowness (Chillo et al 2008). Checking, surface smoothness, and speckiness also affect the consumer acceptability of pasta (Feillet and Dexter 1996).

Mechanical strength is the measurement of dry pasta products ability to withstand compression forces as an indication of the resistance of the product to handling and transportation (Chillo et al 2008). Having a strong gluten matrix and an unchecked product is essential to for high mechanical strength (Feillet and Dexter 1996). Presence of non-traditional ingredients have shown to have an effect in decreasing the mechanical strength of non-traditional pasta product in comparison to the traditional, however this effect can be relatively compensated by applying an ultra-high drying treatment to the product (Abecassis et al 1989).

Cooked quality

Cooking quality is the characteristic of greatest importance to consumers (Hahn 1990). During pasta cooking, protein hydration and starch gelatinization begin from the outside and move towards the center of the pasta piece (Petitot et al 2009). In the interspaces between granules, protein coagulation and interaction lead to a continuous and strengthened network, which traps the starch while the latter, by swelling and gelatinization, occludes theses interspaces.

Cooked pasta quality is defined as cooked firmness, cooking loss, and cooked weight (Debbouz and Doetkott 1996). Cooking loss is a standardized pasta quality test performed in almost every pasta study. It is measured by evaporating cooking water to dryness overnight in a forced-air oven at 110°C. Cooking loss determines the amounts of solids that migrate from the pasta to the cooking water during cooking. Cooking losses are mostly amylose and soluble protein (Matsuo et al 1992).

Cooked weight is normally determined according the approved method 16.50 (AACC International 2013). It is a very simple method that consists on measuring the cooked weight of all rinsed spaghettis strands after being cooked till optimum cooking time. The difference between uncooked and cooked weight determines the weight gained upon cooking.

Cooked pasta firmness is the degree of resistance to the first bite and is defined from a sensorial point of view as the force required to penetrate the pasta with the teeth (D’Egidio and Nardi 1996). Spaghetti firmness is attributed to the protein content and protein quality of the semolina (Del Nobile et al 2005). Matsuo and Irvine (1969) proposed a method to measure cooked pasta tenderness (or firmness). The objective was to try to simulate the “bite test” to differentiate spaghetti made of different durum wheat varieties. That machine was the precursor of the texture analyzer with data-integration system which is currently used. This test measures the force required to cut five strands of spaghetti cooked at their optimum cooking time. This method has become the approved method 66-50.01 of AACC International (AACC International 2013).

Cooked quality tests are mostly done in pasta products cooked until the central starchy core in the sample disappears. That period of time elapsed between pasta product incorporated into the product and the disappearance of the central core is known as cooking time of pasta (AACC International 2013). Cooking time is traditionally determined by squeezing four or five pasta strands of 5 cm long between 2 plexiglass sheets after 30 sec intervals of hydration in boiling water.

Non-traditional ingredients

Cooking time, pasta color, cooking loss, cooked weight and cooked firmness are affected by the inclusion of non-traditional ingredients, sometimes improving certain characteristics of pasta, e.g. stickiness, and sometimes worsening other characteristics, e.g. firmness (Baiano et al 2011; Chillo et al 2008; Manthey and Schorno 2002; Petitot et al 2010; Tudorica et al 2002; Zhao et al 2005). The extent of any change produced in each quality parameter of pasta is independent for every ingredient type, percentage used, and every processing profile (Zhao et al 2005).

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PAPER 1. FLOW PROPERTIES OF SEMOLINA AND WHOLE WHEAT FLOUR FORTIFIED WITH FLAXSEED FLOUR

Abstract

Flow properties of ingredients affect their ease of handling during food processing. Experiments were conducted to determine the angle of repose and angle of slide properties of semolina and whole-wheat flour fortified with flaxseed flour and hydrated to different moisture contents. Semolina 100%, whole wheat 100%, semolina-whole wheat (49:51), semolina-flaxseed flour (90:10), whole wheat-flaxseed flour (90:10), and semolina-whole wheat-flaxseed flour (39:51:10) were the formulations used in the experiments. Flaxseed flour was incorporated as fine or coarse particles, for a total of nine treatments. Determinations of angle of repose and angle of slide were performed on both a stainless steel and an aluminum surface and each formulation was hydrated to 10, 15, 20, 25, 30 and 32% moisture content. Samples containing whole wheat flour and or flaxseed flour were more cohesive and less prone to flow than samples with semolina. A minimum angle of 46° was needed to make all formulations flow regardless of hydration level and/or surface material.

Introduction

Nowadays, a big trend in the food industry is the fortification of well-known staple products with functional ingredients whose consumption has been scientifically proven to promote health of consumers (Roda 2013). In order to satisfy this demand, the pasta industry has started to manufacture multigrain pasta products that contain whole grains such as flaxseed flour and whole wheat flour, ingredients not traditionally present in a pasta product (Marconi and Carcea 2001).

In a pasta manufacturing facility, ingredients are moved via pneumatic suction or via gravity through pipes and chutes of different shapes and sizes. It is fairly common that pipes and chutes are made out of metals, with stainless steel and coated aluminum being some of the most commonly utilized materials in the food industry (Roda 2013). Different surface materials have different static and kinetic friction coefficients meaning that how food powders accumulate or move on them will differ for a given ingredient.

Bulk properties of food powders such as their bulk density and flowability are characteristics that are highly dependent on size, geometry, and surface characteristics of particles (Fitzpatrick et al 2004) and their chemical composition (e.g. fats, proteins, and fibers) (Juliano and Barbosa-Cánovas 2010). Moisture content and the amount of free and bound water have also been reported to affect flow properties of food powders (Iqbal and Fitzpatrick 2006; Juliano and Barbosa-Cánovas 2010; Neel and Hoseney 1984; Teunou and Fitzpatrick 1999).

Semolina is a low cohesive material with good flowing properties, and pasta manufacturing lines are designed based on such properties. The development of new product lines with new product formulations has resulted in problems associated with handling and processing of non-traditional ingredients in facilities that were originally designed to handle only semolina, the ingredient of traditional pasta (Roda 2013). Non-traditional ingredients tend to have different particle size and chemical composition than semolina; therefore, use of non-traditional ingredients can lead to improper flow through the pipelines and accumulation on the pipe walls, which reduces or prevents flow (Twombly and Manthey 2006).

Flow properties of ingredients are very important in the pasta industry since ingredient discharge into the mixing chamber, ingredient hydration-mixing, and pasta extrusion are continuous processes. Therefore, any change in the flow of ingredients affects the efficiency of the entire manufacturing process. Information concerning the flow properties of non-traditional ingredients is needed when designing new or retrofitting existing pasta production lines (Peleg et al 1983). To the best of our knowledge no information is currently available regarding the flow properties of whole wheat flour and flaxseed flour alone or mixed with semolina. The objective of the current experiment was to determine and characterize the angle of repose and angle of slide of semolina and whole wheat flour with or without flaxseed flour and on different surface materials.

Materials and Methods

Experiments were performed with commercial standard semolina (Durakota No.1) from the North Dakota Mill (Grand Forks, ND) and whole hard white wheat flour (Ultragrain) from ConAgra Foods (Omaha, NE). Flaxseed flour was obtained by milling brown flaxseed on a pilot scale hammer mill (Fitzmill, Model DA506, Elmhurst, IL) (de la Peña and Manthey 2014). The ground flaxseed was separated into fine and coarse particles by using a sieve with a 34XXG 600 µm mesh screen.

Nine pasta formulations were evaluated: Semolina 100% (S), whole wheat flour 100% (WW), semolina:whole wheat (SWW) 49:51, semolina:fine flaxseed 90:10 (SFF), whole wheat:fine flaxseed 90:10 (WWFF), semolina:whole wheat:fine flaxseed 39:51:10 (SWWFF), semolina:coarse flaxseed 90:10 (SCF), whole wheat:coarse flaxseed 90:10 (WWCF), and semolina:whole wheat:coarse flaxseed 39:51:10 (SWWCF). Maintaining 51% of whole wheat flour in the formulations was necessary to be classified as a whole wheat product. Uniform blends were prepared by mixing ingredients for 5 min using a cross-flow blender (Patterson Kelly, East Stroudsburg, PA, USA). The 10% (w/w) level of fortification with flaxseed flour was selected because previous research indicated that at this level, the pasta dough had proper consistency for extrusion and the final product had acceptable quality (Sinha and Manthey 2008).

Particle size distribution

Particle size distribution of semolina, whole wheat, and the semolina-whole wheat mixtures was determined using a vibratory shaker (Retsch AS 200 sieve shaker; Retsch International, Haan, Germany) configured with a stack of seven sieves and a vibratory amplitude displacement of 3 mm for 15 sec intervals for 5 min. Sieves used were 50, 100, 150, 250, 425, 500, and 600 μm.

Flow properties

Flow properties were determined as a measurement of angle of repose and angle of slide. Semolina and whole wheat flour were air dried overnight at room temperature to ensure that their moisture contents were less than 10%. Nine formulations were prepared and their moisture content determined according to the Approved Method 44-15A (AACC International 2013). Six samples (200 g) of each formulation were hydrated with distilled water at 40°C to 10, 15, 20, 25, 30 and 32%, db. Dry samples were placed in the 4.2 L bowl and whisked for a few seconds. The samples were mixed at 60 rpm in a small Kitchen Aid mixer (Troy, Ohio, USA) and were whisked for a few seconds before adding water at 60 rpm. After adding the water, the mixing speed was increased to 77 rpm and then 128 rpm for 1 min. Afterwards, a rubber paddle was used to remove the material that adhered to the walls of the bowl to ensure that all ingredients were properly mixed with the whole batch. The sample was mixed for another minute at 128 rpm. Immediately after mixing, measurements of angle of slide were determined on two different metallic surfaces, food grade stainless steel 304 with 2B finish and aluminum. Information about the finish of the aluminum was not available.

Angle of repose

Angle of repose, also referred to as loose-base angle of repose (Ileleji and Zhou 2008) was determined by pouring 200 g of each ingredient sample onto the upper part of the chute of the angle of repose device (Fig. 3). The sample then cascaded through the hopper that directed it into a stainless steel cylinder placed on top of either the stainless steel or aluminum surface. The dimensions of the cylinder were 7.8 cm wide and 13.5 cm tall as described by Kuakpetoon (2001). The distance from the lower part of the chute to the base of the cylinder was 34 cm and the distance from the bottom of the hopper to the bottom of the cylinder was 15 cm. The cylinder was then quickly lifted vertically causing the material inside to spread into a conical heap. The height and diameter of the conical heap were measured using a ruler. The poured angle of repose was calculated using the following equation:

$$\alpha = \tan^{-1}(h/r) \tag{1}$$

where h is the height of the heap of material and r is the averaged radius of the heap obtained by recording the two diameters perpendicular from each other.

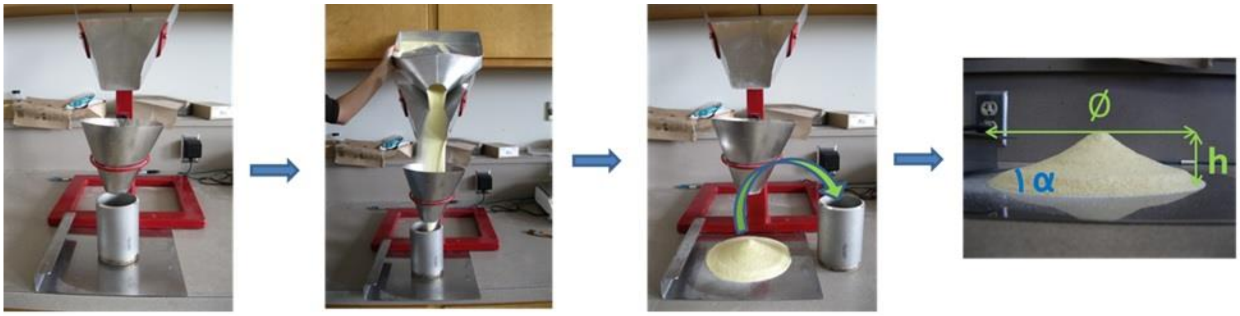


Figure 3. Illustration of the angle of repose test.

Angle of slide

Angle of slide, also known as sliding angle of repose (Ileleji and Zhou 2008) was calculated with an updated version of the method described by Chang et al (1998). The angle of slide used the same cylinder used for angle of repose. The cylinder was placed on the center of a metal sheet (stainless steel or aluminum) that was placed on top of a platform whose slope could be increased gradually by manually turning a screw (Fig. 4). Complete horizontal position of the platform was ensured by placing a protractor on top of it and verifying that it read 0°. After that, 200 g samples were poured into the cylinder which was then lifted vertically to obtain a conical heap of material similar to that used to measure the angle of repose. The heap did not touch the walls of the platform at any moment. The slope of the platform was slowly increased until the majority of the material slid down. The angle formed between the slope of the platform and its horizontal base was measured using a protractor.

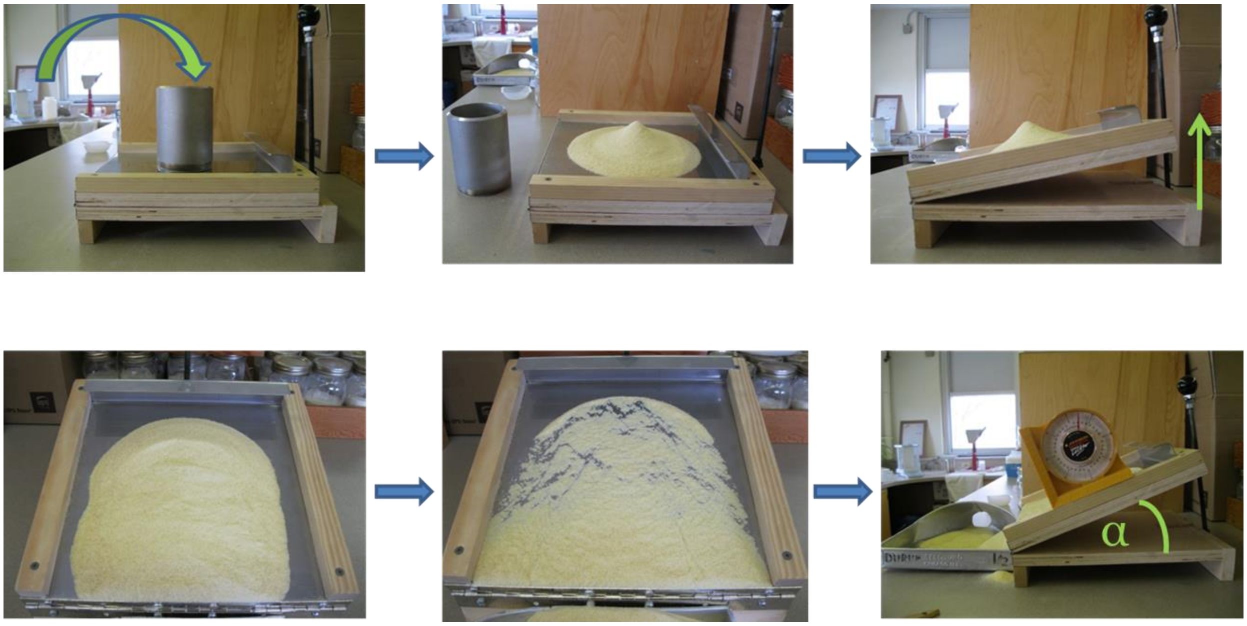


Figure 4. Illustration of angle of slide test.

Statistical analyses

Analysis of variance of the angle of slide and angle of repose was performed considering the experimental layout as a split plot arrangement in which the whole plot factor was the ingredient formulation (S, SFF, SCF, WW, WWFF, WWCF, SWW, SWWFF, and SWWCF) and the subplot factor were six hydration levels (10, 15, 20, 25, 30, 32%). Tests were replicated 3 times with each replicate on different days. The angle of slide and angle of repose tests were conducted on a stainless steel surface and on an aluminum surface. Each surface was analyzed separately. All data were subjected to analysis of variance using the ‘Mixed’ procedure in SAS System for Windows (v 9.3, SAS Institute, Cary, NC). Treatment means were separated by Fisher’s protected Least Significant Difference test calculated at *P*=0.05.

Results and Discussion

Particle size distribution

Particle size distribution of the tested samples were typical for these types of ingredients. Determination of particle size distribution of semolina showed that 50% of the particles were retained on the 250 μm sieve and 26.3% were retained on the 150 μm sieve (Fig. 5). As expected, particle size of whole-wheat flour was significantly smaller compared to the semolina. A total of 50% of the whole wheat flour was retained in the 100 μm sieve, followed by 34.9% of the flour which was retained on the 50 μm sieve. Particle size distribution of the semolina-whole wheat mixture had a bimodal distribution of particles reflecting the small particle size associated with whole wheat flour and large particle size associated with semolina. Ground non-fractioned flaxseed flour showed larger particle size than semolina or whole wheat flour with 23.8% retained in the 424 μm sieve, and 46.7 and 27.3% retained in the 500 and 600 μm sieve, respectively. Flaxseed flour has 40% lipid content (Rubilar et al 2010), and lipid has been proven to act as a very strong cohesive agent that does not allow the separation of the individual particles during the sieving process. Flaxseed flour formed aggregates during sieving. These aggregates were much larger than the sieves mesh size on which they were formed; therefore, the particle size distribution of the flaxseed flour might not represent completely the real particle size of this material.

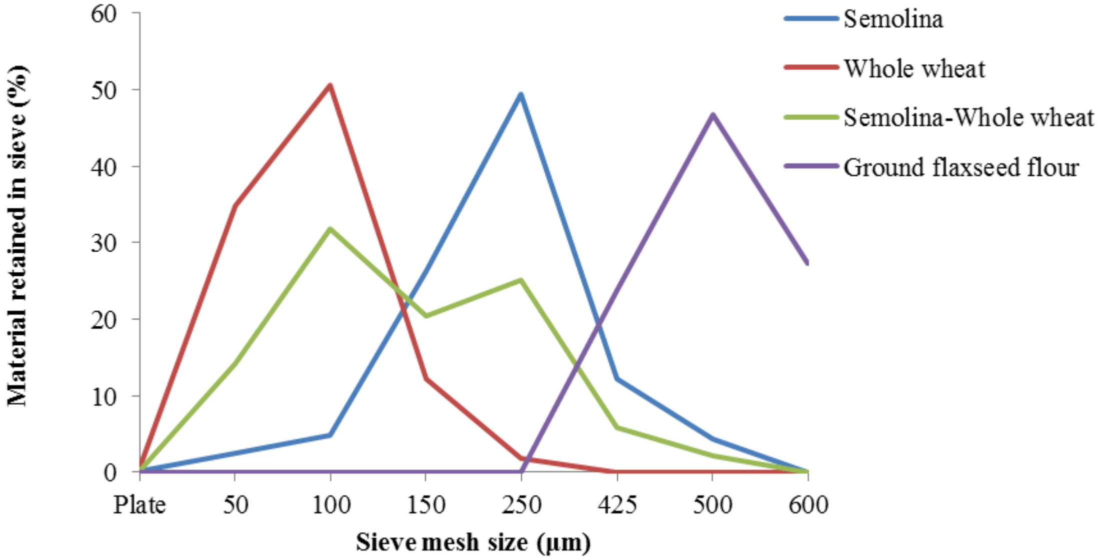
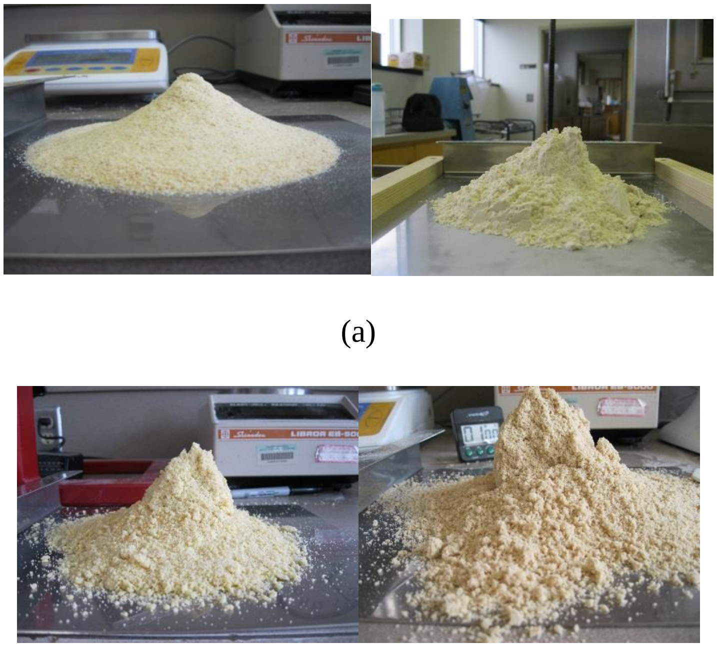


Figure 5. Particle size distribution of semolina, whole wheat flour, the combination of semolina and whole wheat flour, and flaxseed flour.

Flow properties

Angle of repose

The profile shape of the heap and the shape of the base were better defined and more circular, respectively, with S, SFF and SCF than with WW, WWFF, and WWCF, regardless of moisture content (Fig. 6-a). In all cases, ingredient formulations with higher moisture content formed heaps of material with both heap profile shape and base shape more irregular than for samples with less moisture content (Fig. 6-b). SFF and SCF hydrated at 30 and 32% displayed much more irregular heap profile shape than did S hydrated at the same moisture contents; however, this was not observed for WWFF, and WWCF in comparison to WW. In formulations with whole wheat flour as the main ingredient hydrated at 30 or 32% it was noticed that sometimes, after the cylinder used to hold the sample was lifted, the cylindrical shape of the cylinder was maintained and samples would not form any heap to measure the angle of repose (Fig. 7-a). Eventually, the cylinder-shaped sample would collapse (Fig. 7-b and -c), but the irregularity of the heap formed often did not allow for measuring diameter and height needed for angle of repose calculation. When this phenomenon took place the test was repeated until at least three acceptable measurements were recorded.



(a)

(b)

Figure 6. Heap shape of semolina-fine flaxseed (90:10) hydrated to 10% (left) vs whole wheat flour-fine flaxseed (90:10) hydrated to 10% (right) (a) and semolina-fine flaxseed (90:10) hydrated to 30% (left) vs whole wheat flour-fine flaxseed (90:10) hydrated to 30% (right) (b).

(a) (b) (c)

Figure 7. Evolution of the heap shape of 100% whole wheat flour hydrated to 30% over time.

Formulations with both semolina and whole wheat flour had a behavior between that observed for samples with 90% or 100% semolina or whole wheat flour. At low moisture contents, the shapes formed by the samples were similar to those formed by whole wheat but as moisture increased the heap shape and the base shape were more similar to those formed by semolina. Presence of flaxseed flour did not affect this behavior.

The formulation by flour blend hydration level interaction was significant for the angle of repose test when performed on either a stainless steel or on an aluminum sheet (Table 1). With the purpose of facilitating the visualization of the results, data points are represented in line graphs grouped according to the flour formulation (Fig. 8). Data in Figure 8 has been averaged across both surface materials because the measurements of the angle of repose recorded were extremely similar when the test was run on a stainless steel sheet or on an aluminum sheet, indicating that the material of the surface did not impact the results of this test. Samples with 90 or 100% whole wheat flour in the formulation had higher angle of repose than samples with semolina as the main ingredient. Formulations that contained both semolina and whole wheat flour had intermediate values of angle of repose. The presence of flaxseed flour in either fine or coarse particle size also contributed to an increase in the angle of repose of formulations containing 90% semolina (Fig. 8-a) but barely had any effect on samples with 90% whole wheat flour (Fig. 8-b) or in samples composed of 39:51 semolina and whole wheat flour (Fig. 8-c).

Table 1. Analysis of variance for the angle of repose of different flour formulations and hydration levels when measured on stainless steel and on aluminum surface.

Effect	Aluminum		Stainless steel	
	Angle of slide	Angle of repose	Angle of slide	Angle of repose
Formulation (F)	**	**	**	**
Hydration (H)	**	**	**	**
FxH	**	**	**	**

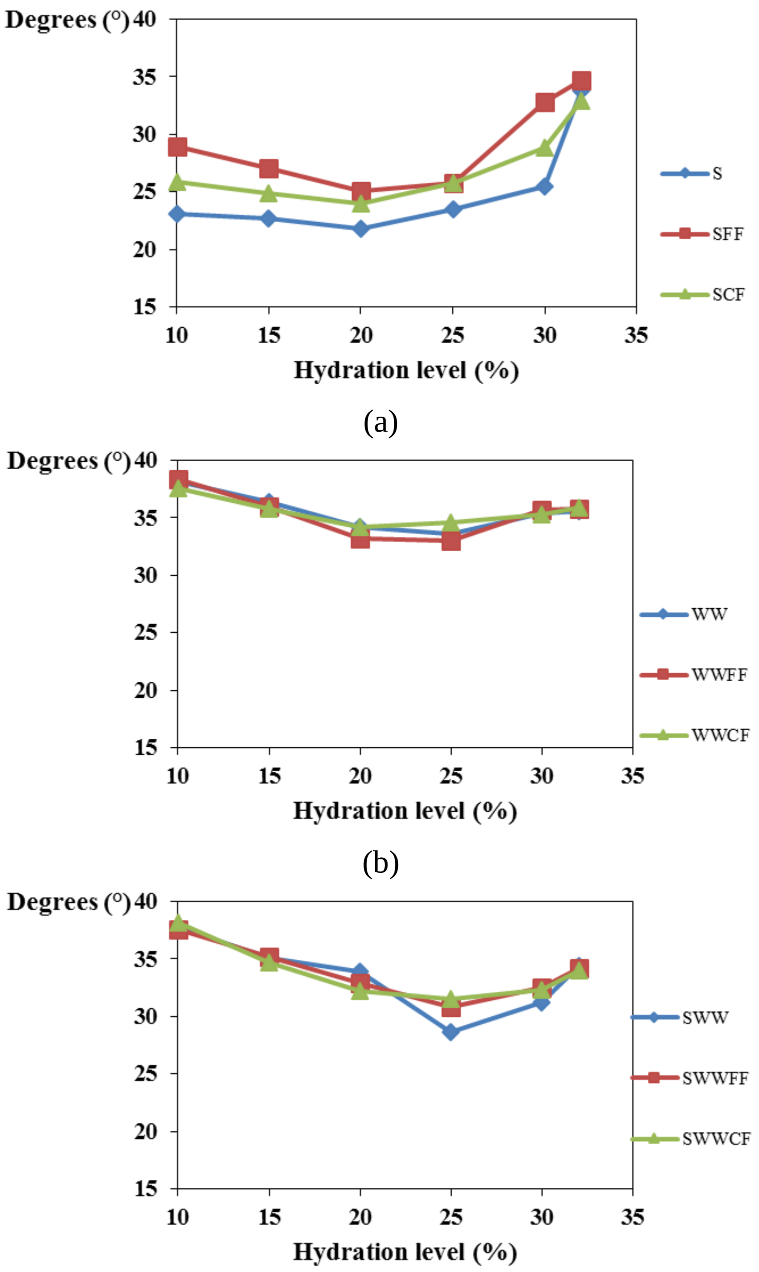
* Significant at $P \leq 0.05$.
** Significant at $P \leq 0.01$.

Differences in angle of repose observed among the samples were attributed to the difference in the cohesion forces among the whole wheat flour, semolina, and flaxseed flour particles. Cohesion is the attractive force between particles and is known to increase as particle size decreases (Fitzpatrick 2005). For the same weight, whole wheat flour contains more small particles than semolina does, as shown in Figure 5, which makes whole wheat flour particles more cohesive and favors the formation stable heaps with higher angle of repose than semolina samples. The high angle of repose when flaxseed flour was in combination with semolina was attributed to the high lipid content of flaxseed flour particles (Neel and Hoseney 1984) and to the flaxseed gum material that can leach from the hull fragments when the flour blend is hydrated at 25-30% (Marpalle et al 2014). Both the lipid and gum materials can act as a cohesive agents that increased the inter-particle forces within the flour and the degree of adhesion between flour and the metal surface and lead to the formation of higher angles of repose than when flaxseed flour was absent in the formulation. The increase of cohesiveness and adhesion due to the presence of whole wheat flour, flaxseed flour or both, lowers the flowability capability of the given sample, and increases the risk of bridging and/or flow interruption. Special attention should then be paid when handling these type of flours to avoid problems in the product line.

The angle of repose decreased progressively as the moisture content of the samples increased, reaching its minimum at 25% moisture content (Fig. 8). Once this minimum angle of repose was reached, the angle of repose increased significantly for moisture contents of 30 and 32%, reaching higher angle of repose values than at 10% moisture content.

The effect of moisture content on the flowing behavior of powders was attributed to nature of the cohesion forces generated by the increase of water in the formulation. For dry powders, cohesion forces can be classified as van der Waals, electrostatic, and magnetic forces, with the van der Waals forces of attraction usually being the most important (Alexander et al 2006; Fitzpatrick 2005). However, in the presence of liquid, i.e. water, formation of liquid bridges (capillary forces) becomes an important source of cohesion (Alexander et al 2006; Seville et al 2000). When samples have low moisture content, van der Waals forces are the main cause of cohesion since the liquid bridges are unstable because the liquid volume between particles is insufficient (Insopencu et al 2007). van der Waals forces occur on a molecular level due to the polarization of atoms and molecules resulting in attraction between atoms on the surfaces of each of the particles. The attraction among sample particles due to van der Waals forces decreased as moisture content increases because the water molecules interfere with the atoms in the flour particles. At low hydration levels the increase in capillary forces was not enough to compensate for the decrease in the van der Waals forces; hence, increasing the hydration level from 10 to 15 and 20% caused the drop in angle of repose values. At 25 or 30% hydration level, depending on the formulation, there was enough water in the sample to form enough liquid bridges among the particles to compensate for the decrease of the van der Waals forces and to increase the cohesiveness of the sample which led to an increase of angle of repose. This response reflected the role that moisture content has on the increase or decrease of cohesiveness of cereal powders. These results indicate the importance of knowing the flowability of a sample at different hydration levels when designing the pipes and/or chutes through which the sample will flow.

The data recorded with the angle of repose test can only be considered as qualitative and relative data that helps finding differences between samples (Ileleji and Zhou 2008). The angle of repose can be used as a rough flowability indicator; angles of up to about 35° indicate free flowability, 35° to 45° some cohesiveness, 45° to 55° cohesiveness (loss of free flowability), and 55° and above very high cohesiveness and very limited flowability (Chang et al 1998). Based on this classification S, SFF, and SCF possess free flowability; WW, WWFF, and WWCF had some cohesiveness when hydrated to 10 to15 and 30 to32% and free flowability when hydrated to 20 to 25%; SWW, SWWFF and SWWCF had some cohesiveness when hydrated to 10 to 15% and free flowability when hydrated from 20 to 32%. These results shed light onto the physical interactions of the different powders that are included in a given formulation at given hydration levels and help to characterize the formulation itself; however, it does not help towards the designing of the vessel or the pipes. Subsequently, the angle of slide was performed to complement the information provided by the angle of repose test.



(a)

(b)

(c)

Figure 8. Angle of repose for the different formulation hydrated at different moisture contents averaged for aluminum and stainless steel.

LSD=3.42°.
S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour-Fine Flaxseed (39:51:10), SWWCF: Semolina-Whole Wheat Flour-Coarse Flaxseed (39:51:10).

Angle of slide

Two different responses were observed for the angle of slide measurements of the different formulations on two different surface materials (Fig. 9). The most common response showed that the angle of slide decreased as moisture content increased from 10% to 20 or 25% moisture content, and then increased as the moisture content increased. The second response showed that the angle of slide increased with the increase in moisture content of the formulation. These different responses did not follow a particular trend regarding the composition of the formulations and were attributed to the interactions between the sample's particles and the surface material used to run the experiment.

The ingredient formulation by hydration level interaction was significant for the angle of slide test when performed on either a stainless steel or on an aluminum sheet (Table 1). To facilitate the visualization of the results, data points were represented in line graphs grouped according to the flour formulation (Fig. 9). There was a notable difference in the magnitude of the angle of slide values when comparing the values recorded on a stainless steel and on an aluminum surface. Because of this, the angle of slide data was presented separated for each surface material (Fig. 9-a and -b). S, SFF, and SCF always had a much lower angle of slide than any of the other formulations at any given moisture content, and samples with 90 or 100% whole wheat flour always had the highest angle of slide (Fig. 9). SWW, SWWFF, and SWWCF hydrated to 10, 15, and 20% displayed low angle of slide when compared to WW, WWFF, and WWCF, respectively, but these differences were almost non-existent when samples were hydrated at 25, 30, and 32% moisture content. Samples with fine flaxseed flour required steeper angles to slide than did samples with coarse flaxseed flour or with no flaxseed flour in the formulation e.g. SFF vs SCF and S. All these observations occurred on both surface materials studied in this experiment. Although not statistically analyzed together, angle of slide tended to be higher when the test was performed on the stainless steel surface than on the aluminum surface used in this experiment, especially after the 20% moisture content was reached (Fig. 9). This observation might be a function of the degree of polish of the surfaces, or of the finish treatment of the material; therefore, this result can be only applicable to the studied surfaces in experiment, and it cannot be generalized to all stainless steel and aluminum surfaces. The results recorded with this experiment confirmed then the importance choosing the right a chute and/or pipe material under which the materials show good flow properties to minimize the risk of bridging occurrence in the line.

Flow of powders implies the collapse of a mechanical structure formed by powder particles (Juliano and Barbosa-Cánovas 2010). The forces opposing flow are friction, attraction between particles (cohesion), attraction between the particles and system walls (adhesion), and mechanical resistance (Juliano and Barbosa-Cánovas 2010; Peleg 1978). Powders with small particle size have been reported to have increased cohesion forces between flour particles and between particles and surfaces as well as friction to resist flow (Fitzpatrick et al 2004; Geldart et al 2009). Accordingly, the high angles of slide in samples with 90% or 100% whole wheat flour are mostly attributed to large cohesion and adhesion forces generated due to the small particle size of the flour (Figs. 5 and 9). Adhesion was particularly observed for WW, WWFF, and WWCF hydrated to 10 and 15% moisture. These samples left a trail of whole wheat flour dust as they slid on the stainless steel or aluminum surfaces (Fig. 10). Semolina, with a much larger portion of particles over 150 µm than whole wheat flour, was much less cohesive and did not adhere as much to the metallic surface as whole wheat flour.

The tendency to adhere to surfaces makes the handling of whole wheat flour more complicated than handling semolina. Whole wheat flour can accumulate at the surface of a chute or pipe and eventually it could partially or completely block the flow of material and cause the processing line to shut down. The low level of cohesion and adhesion of semolina explains the low angles of slide displayed by S, SFF, and SCF. The intermediate results reported for SWW, SWWFF, and SWWCF at low moisture contents are the result of the combination of both semolina and whole wheat properties. These results agreed with those reported by the angle of repose test, indicating the relationship between these two tests to describe the flowing properties of flour blends.

The increase in the angle of slide in formulations containing fine flaxseed flour is attributed to the high lipid content inherent in flaxseed. Lipid content has been reported as a major factor influencing the flow properties of flours (Neel and Hoseney 1984). The lipid content of flaxseed flour corresponds to more than 40% of the total composition of the seed (Rubilar et al 2010). Lipid can act as a particle binder, increasing the cohesiveness in a powdery sample and the adhesion of the flour particles to the metallic surface. The presence of the gum that is located at the surface of the hull fragments (Oomah et al 1995) is also assumed to play a role in the flowing properties of the formulations, especially when formulations were hydrated at and over 25%. At that hydration level this gum material can start leaching and interact as a cohesive agent of the formulation particles as described in the angle of repose section. Fine flaxseed has smaller particle size than the coarse fraction, therefore it has greater surface area and a larger portion of the lipid and gum fraction exposed to the whole wheat flour, the semolina and/or the metal surface of the platform. This explains why the presence of fine flaxseed in a given formulation increased the angle required to make samples flow in comparison to the same sample with coarse flaxseed or with no flaxseed at all in the formulation. Coarse flaxseed particles did not affect the angle of slide of formulations containing S, WW or SWW when the formulations had 15% or higher moisture content.

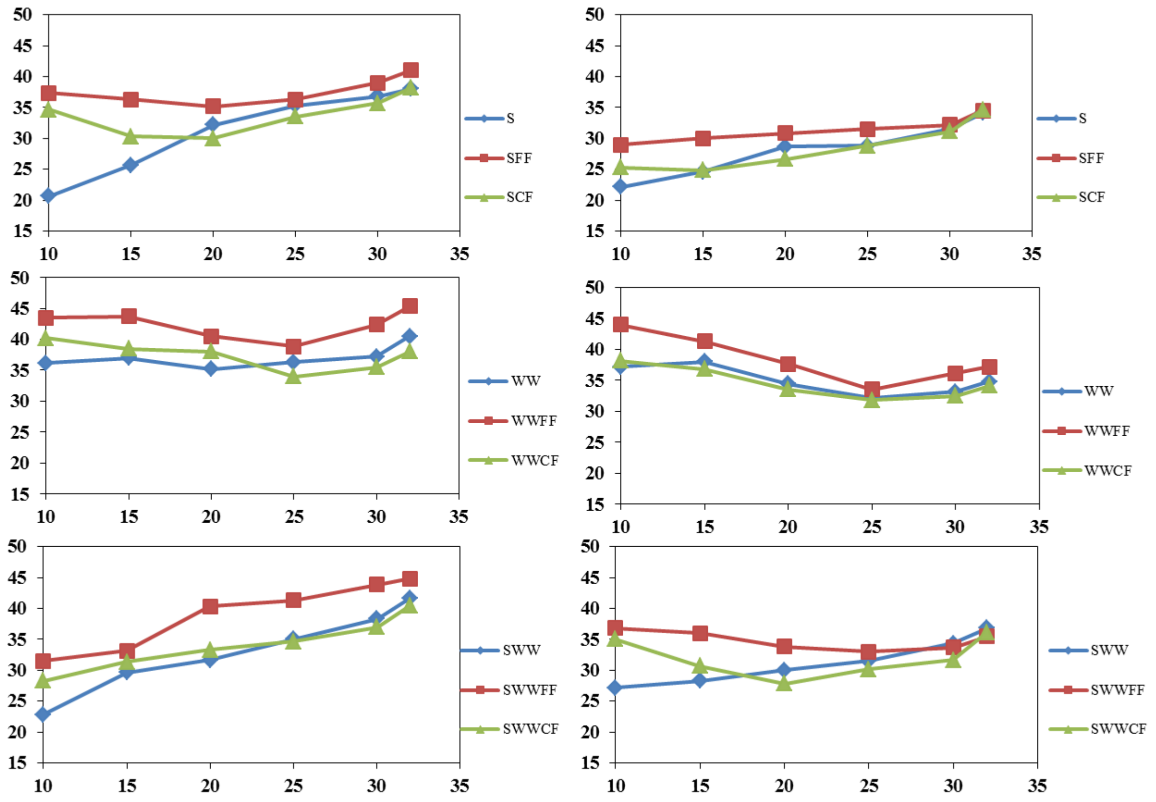
The way particles interact with each other not only affects the angle needed for materials to flow, but also to the way materials flow. In general, there are two types of flow behavior: free flow and floodable flow (flushing) (Ganesan et al 2008). During free-flow, materials flow in a steady and consistent way, as individual particles. Conversely, a floodable flow can be discontinuous, sudden and uncontrollable. Semolina and whole wheat flour demonstrated both types of flow. The flow of semolina tended to be closer to free-flow whereas dry whole wheat flour flow was more similar to a floodable flow.

Moisture content of the sample influenced the type of flow behavior. When working with S, SFF, and SCF samples with moisture contents from 10 to 20% moisture content, at some point during the angle of slide test, the heap of semolina would partially break away and start to flow downwards towards the edge of the platform, while the other part stayed static on the metal surface. As the steepness of the platform increased, more material slid at the surface (free-flow) until an angle was reached where all material flowed *en masse* (floodable flow) leaving little or no remnant semolina on the surface. The angle at which this floodable flow was attained was considered the angle of slide of the hydrated ingredient. At the higher moisture levels, 25-32%, the material became progressively more and more agglomerated and sticky due to higher water levels, especially for SFF and SCF. This change in the physical properties of the powder introduced a lot of shear in the flow which promoted the floodable flow behavior of these samples. Often, SFF and SCF hydrated to 32% moisture left behind a thick layer of sample material on the metal surface after the heap of material flowed. This indicates that 32% is very critical hydration for these two formulations since they are very likely to cause wall accumulation that leads to bridging and product flow disruption. Subsequently, the use of steeper and wider chutes or pipes is recommended when handling SFF and SCF hydrated at 32% than when using SFF and SCF hydrated at 30% or less. Other alternatives to the increase of steepness of pipes and chutes could be the use of a vibrating or wiping mechanisms to avoid or prevent the bridging of materials in the processing line.

The flow properties of WW, WWFF, and WWCF were opposite to those of formulations containing 90 or 100% semolina. At low hydration levels, the material would flow *en masse* after a certain angle was reached and a very thin film of remnant flour was observed on the metal surface. As previously explained, van der Waals inter-particle forces are high in wheat flour; so the whole heap remained together until the floodable flow occurred. The flow *en masse* behavior changed more into free flow once the hydration level exceeded 25%. This change in flow behavior can be attributed to the decrease in inter-particle van der Waals forces due to increased moisture content as described above. In addition, at high hydration levels, the particles began to group together into small agglomerates; hence, the inter-particle forces were diminished. Unlike semolina samples, the presence of flaxseed flour in combination with whole wheat flour did not affect how the material flowed on the platform.

Semolina-whole wheat formulations had intermediate flow behavior in comparison with semolina and whole wheat flours alone. Semolina-whole wheat flour formulations with low moisture contents showed a quick *en mass*e flow leaving no material behind, but as moisture content increased, the flow slowed, turned into free flow, and residual ingredients were seen on the platform surface.

The angle of slide is the minimum angle of incline relative to the horizontal that will allow a bulk solid to flow from under its own weight. This angle is presumed to be useful in designing stationary chutes (Juliano and Barbosa-Cánovas 2010) and it can also have applications in the designing of pipes; therefore, the values of angle of slide reported in Figure 9 indicate the minimum angle that a chute or a pipe should have for each formulation-hydration level-surface material combination in order to have free flow of the flour blend and avoid accumulation and/or obstruction on the walls. Based on results, the highest angles of slide required to flow on stainless steel was recorded for WWFF hydrated at 10, 15, and 32% (43.5, 43.7 and 45.2°, respectively) and SWWFF hydrated at 32% (44.8°). For aluminum, the highest angle of slide was recorded for WWFF hydrated at 10% (44.0°). These angle of slide values indicate that pipes or chutes should have a slope of at least 46° to guarantee the free flow of these formulations, regardless of their moisture content and the surface material in which the flow.



(a) (b)

Figure 9. Angle of slide for the each formulation hydrated at a different moisture content when the test was run on stainless steel (a) and aluminum (b) surface material. The X axis represents the formulation hydration level (%), and the Y axis represents the angle of slide (°).

LSD= 2.35° for aluminum; LSD=2.77° for stainless steel.
S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour-Fine Flaxseed (39:51:10), SWWCF: Semolina-Whole Wheat Flour-Coarse Flaxseed (39:51:10).

Figure 10. Residue left behind by the formulation 100% whole wheat flour hydrated at 15% on the stainless steel surface after running the angle of slide test.

Conclusions

Characterization of the flowability of pasta ingredient formulations by the angle of repose and angle of slide tests determined that samples with 90 or 100% whole wheat flour were more cohesive, and therefore less prone to flow than samples with 90 or 100% semolina. Intermediate results were obtained for the samples that contained 100% or 90% or the mixture of whole wheat and semolina flour. Presence of fine flaxseed flour tended to make whole wheat and semolina flours more cohesive and less prone to flow. The minimum steepness of chutes and/or pipes required for free flow of any of the studied formulations hydrated between 10 and 32% over a stainless steel or aluminum surface was 46° as determined by the angle of slide test.

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PAPER 2. AGGLOMERATION PROPERTIES OF SEMOLINA AND WHOLE-WHEAT FLOUR FORTIFIED WITH FLAXSEED FLOUR

Abstract

Experiments were conducted to determine the wet agglomeration properties of semolina and whole-wheat flour fortified with flaxseed flour. The formulations used were semolina 100% (S), whole wheat 100% (WW), semolina-whole wheat (49:51) (SWW), semolina-flaxseed flour (90:10), whole wheat-flaxseed flour (90:10), and semolina-whole wheat-flaxseed flour (39:51:10). Flaxseed flour was incorporated as fine (FF) or coarse (CF) particles, for a total of nine treatments. For determination of the wet agglomeration and extrusion properties, samples were hydrated to 30, 31, 32, 33, and 34% moisture content. Flour composition and starch damage had an important role in the formation of wet agglomerates during pasta processing. Greatest large agglomerate formation occurred with SFF hydrated over 30% moisture; whereas limited large agglomerate formation was detected in whole wheat samples with or without flaxseed flour at any of the hydration levels studied. Adhesion was the agglomerate textural property most affected by the dough formulation and the responsible one for the formation of large agglomerates. Subsequently, the maximum hydration level recommended for each formulation to avoid the formation of large aggregates was: 30% for SFF, 32% for SCF, 33% for S, WWFF and SWWFF, and 34% for WW, WWCF, SWW, and SWWCF. More research is needed to determine the effect of the agglomerates textural properties on the rheological behavior of dough during extrusion.

Introduction

Nowadays, one of the biggest trends in the food industry is the fortification of well-known staple products with functional ingredients whose consumption has been scientifically proven to promote the health of the consumer (Roda 2013). In order to satisfy this demand, the pasta industry has started to manufacture multigrain pasta products that contain whole grains such as flaxseed flour and whole-wheat flour, ingredients not traditionally present in a pasta product (Marconi and Carcea 2001). The development of a new product line has resulted in problems associated with handling and processing of non-traditional ingredients in facilities that were initially designed to handle only semolina, the original ingredient of traditional pasta (Roda 2013).

Agglomeration is the formation of agglomerates from dispersed materials into materials with large unit sizes (Yan and Barbosa-Cánovas 1997). Wet agglomeration occurs due to adhesion and cohesion forces caused by formation of liquid bridges between the partly or completely filled pore spaces of individual particles when enough water is available (Schubert 1981). The agglomerate size is dependent on the source and physical characteristics of the dispersed material, e.g. particle size and particle shape, and the amount of water added to given amount of dry material.

A problem derived from the utilization of non-traditional ingredients in pasta manufacturing is the formation of large agglomerates of non-traditional pasta ingredients during the hydration step prior the dough extrusion. Non-traditional ingredients have different hydration dynamics than regular semolina (Kratzer 2007; Traynham et al 2007); therefore, the water requirement of the ingredients during the hydration step are going to differ from that required by semolina. If the hydration level applied during the mixing process is not adjusted for the presence of non-traditional ingredients, it is possible that the non-traditional formulations will either under-hydrate and over-hydrate. The latter can product wet agglomeration and it was ~~will be~~ the focus of the current study.

Large agglomerates can be detrimental during the pasta processing process since they can block the flow of hydrated ingredients (de la Peña and Manthey 2012). Agglomerate formation has been associated with bridging that prevents the movement of hydrated ingredients within and out of the mixing chambers. Commercial pasta presses utilize two mixing chambers: high speed mixer and a main mixer. Dry ingredients and water are introduced into the high speed mixer. The rapid mixing action of the high speed mixer promotes uniform wetting of all ingredients. Wetted ingredients are discharged into the main mixer where they move slowly towards the discharge end. Retention time in the main mixer is generally 15 to 20 min, which allows moisture to penetrate the ingredients.

Agglomerate formation typically occurs in the main mixing chamber. Typically, the hydrated product will pass through a vacuum air lock and then be discharged onto the rotating auger located inside the extrusion barrel. If large dough agglomerates are formed, they can bridge over the discharge chutes or openings that connect the high speed mixer with the main mixer and the main mixer with the extrusion auger. Bridging of the hydrated material at any of these two connecting points will cause the disruption of the flow of material and the shutdown of the line. Consequently, the formation of large wet agglomerates is a significant problem in the manufacturing of pasta products, and the parameters that regulate the formation of these agglomerates should be adjusted to prevent or reduce this problem.

Several researchers have investigated the wet agglomeration phenomenon of semolina under different settings (Barkouti el at 2014; Mandato et al 2013; Rondet et al 2010; Rondet et al

2013; Saad et al 2011); however, none of these studies were applied to the pasta manufacturing industry or included the effect of ingredient mixes containing semolina/whole wheat flour and/or flaxseed flour on agglomeration. The objective of the current experiment was to determine and characterize the wet agglomeration properties of hydrated blends of semolina, whole wheat flour and flaxseed flour during pasta production.

Materials and Methods

Experiments were performed with commercial semolina (Durakota No.1) from North Dakota Mill (Grand Forks, ND) and whole hard white wheat flour (Ultragrain) from ConAgra Foods (Omaha, NE). Flaxseed flour was obtained by milling brown flaxseed on a pilot scale hammer mill (Fitzmill, Model DA506, Elmhurst, IL) as described by de la Peña and Manthey (2014). The ground flaxseed was separated into fine and coarse particles by using a sieve with a 34XXG 600 µm mesh screen.

Nine pasta formulations were evaluated: Semolina 100% (S), whole wheat flour 100% (WW), semolina:whole wheat (SWW) 49:51, semolina:fine flaxseed 90:10 (SFF), whole wheat:fine flaxseed 90:10 (WWFF), semolina:whole wheat:fine flaxseed 39:51:10 (SWWFF), whole wheat:coarse flaxseed 90:10 (WWCF), and semolina:whole wheat:coarse flaxseed 39:51:10 (SWWCF). Maintaining 51% of whole wheat flour in the formulations was necessary to be classified as a whole wheat product. Uniform blends were prepared by mixing ingredients for 5 min using a cross-flow blender (Patterson Kelly, East Stroudsburg, PA, USA). The 10% (w/w) level of fortification with flaxseed flour was selected because previous research indicated that at this level, the pasta dough had proper consistency for extrusion and the final product had acceptable quality (Sinha and Manthey 2008).

Proximate analysis

AACC International Approved Methods 08-12.01, 44-15.02, 56-30.01, 76-13.01, 76-31.01 and 46-30.01 were used to determine ash content, moisture content, water holding capacity, starch content, starch damage, and protein content, respectively, of the nine formulations used in this study (AACC International 2013). The conversion factor used to determine protein content was 5.7 for semolina and whole wheat flour and 5.41 for flaxseed flour (Tkachuk 1969). Lipid content was determined using a 16 h Soxhlet extraction with hexane according to Method Ba 3-38 (AOCS 1998).

Particle size distribution

Particle size distribution of dry semolina, whole wheat, and the semolina-whole wheat mixtures was determined using a vibratory shaker (Retsch AS 200 sieve shaker; Retsch International, Haan, Germany) configured with a stack of seven sieves and a vibratory amplitude displacement of 3 mm for 15 sec intervals for 5 min. Sieves had a mesh size of 50, 100, 150, 250, 425, 500, and 600 μm.

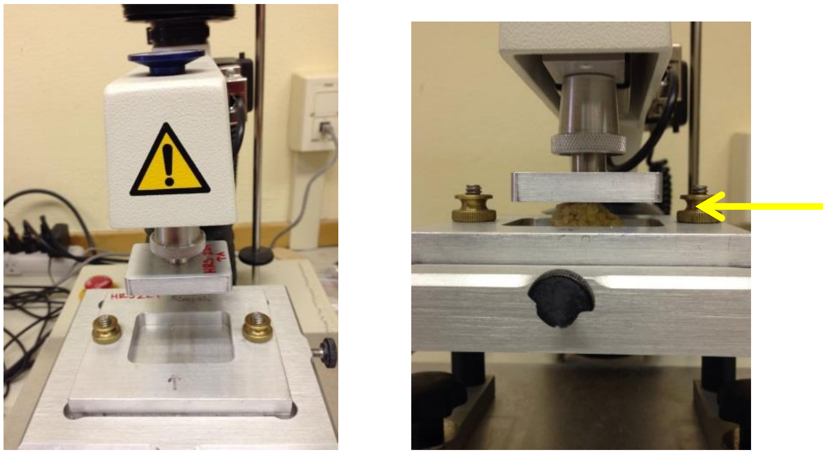
Wet agglomeration and bulk density of agglomerates

Each formulation (1000 g) was hydrated to 30, 31, 32, 33, and 34% moisture content using warm (40°C) distilled water and mixed for 4 min at 180 rpm in a 9.5 L Hobart mixer with a beater paddle attached. Agglomerates formed during mixing were separated by particle size by using sieves of different mesh number that were rotated at a constant speed (1Hz) for 30 sec by using a rotary sieve shaker (Strand Shaker Co. Minneapolis, MN). The different mesh sizes used were 0.28, 0.64, 1.27, and 1.91 cm. The weight of the portion of sample retained in each sieve was recorded and expressed as percentage of the total initial weight of the sample.

Loose bulk density of each sample hydrated to 30, 31, 32, 33, and 34% was determined as g/cm^3 by filling a 0.945 dm^3 container to the top. The height from which the material was poured into the container was always 15 cm. The excess sample was leveled off with a wood leveler to obtain a standard volume. The net weight of the wet material was calculated by subtracting the empty weight of the container from the total weight of both material and container. Bulk density was determined three times per treatment for average calculation.

Agglomerate compression

The Texture Profile Analysis (TPA) of the agglomerates retained in each sieve was determined with a Texture Analyzer (TA-XT2, Texture Technologies Corp., Scarsdale, NY). Agglomerate TPA was measured immediately after agglomerate formation. The TPA profile measured the hardness, adhesiveness, springiness, and resilience of the dough agglomerates formed during the mixing stage prior to pasta extrusion. The pasta firmness/stickiness rig probe was used (Fig. 11-a). Agglomerates were individually placed in the center of the platform opening. The probe was brought into contact with the top of the agglomerate right before the test started as shown in Figure 11-b. The level of strain was chosen based on the results of preliminary tests run for this experiment. At 75% of strain, the pressure exerted on the larger agglomerates was too high and the security switch that stops the machine was triggered. At 25% strain the magnitude of the curves was too small for the small agglomerates and the results were too variable. Subsequently, the level of strain chosen was 50% because at that strain it was possible to obtain good compression curves for all agglomerates sizes. Individual agglomerates were only used once for the compression test, and after that they were discarded. The formation of agglomerates larger than the 1.91cm-mesh sieve was not as abundant as the other agglomerates sizes; therefore, a minimum of three measurements were recorded per agglomerate size, but up to 10 measurements were made when enough agglomerates were available. The average of all measurements for each agglomerate size was calculated per replicate. The test was done in triplicate.



(a) (b)

Figure 11. Pasta firmness/stickiness rig probe attached to Texture Analyzer (a); position of dough agglomerate on probe right before starting the test (b).

Scanning electron microscopy

Scanning electron microscopy was conducted by the Electron Microscopy Center located at North Dakota State University, Fargo, ND. Agglomerates taken from samples hydrated to 34% were mounted on aluminum mounts with silver paint and were then coated with gold-palladium using a Balzars SCD 030 sputter coater (Model SCD 030, Liechtenstein) to make them electrically conductive. Samples were examined and photographed at 50 and 100 X magnifications using a JEOL JSM-6490LV scanning electron microscope (JEOL USA, Peabody, Massachusetts) at an accelerating voltage of 15 kV.

Statistical analysis

Analysis of variance of the water holding capacity, bulk density and TPA test was performed considering the experimental layout as a split plot arrangement in which the whole plot factor was the ingredient formulation (S, SFF, SCF, WW, WWFF, WWCF, SWW, SWWFF, and SWWCF) and the subplot factor were five hydration levels (30, 31, 32, 33, and 34%).

All data were subjected to analysis of variance using ‘Mixed’ procedure in SAS System for Windows (v 9.3, SAS Institute, Cary, NC). Treatment means were separated by Fisher’s protected Least Significant Difference test calculated at *P*=0.05.

Results and Discussion

Proximate analysis

Proximate analysis of the raw ingredients and the different formulations studied in this experiment is presented in Table 2. The results were typical for each ingredient. Lipid, protein, and ash contents were higher in all formulations that contained flaxseed flour either in coarse or fine form in comparison to the correspondent formulation with no flaxseed flour. This effect was expected since flaxseed flour is naturally higher in lipid, ash, and protein than semolina or whole wheat flour (Daun et al 2003; Manthey and Twombly 2006). Starch damage of formulations was 3.8% for S, SFF, and SCF, 5.6% for WW, WWFF, and WWCF, 4.4% for SWW, SWWFF and SWWCF. Flaxseed flour had little or no effect on starch damage since flaxseed lacks starch. Greater starch damage values in WW than in S were also anticipated since in the manufacturing of WW the material is subjected to a greater particle reduction process than semolina.

Table 2. Proximate composition of ingredient formulations and flaxseed flour in fine and coarse form.

Formulations	Lipid	Protein	Ash	Starch
	% db			
S	5.27 ± 0.16	14.05 ± 0.06	0.82 ± 0.08	72.00 ± 0.86
WW	7.04 ± 0.33	13.76 ± 0.04	1.87 ± 0.02	64.46 ± 0.09
FF	47.49 ± 0.22	22.13 ± 0.02	4.19 ± 0.02	0
CF	43.34 ± 0.40	19.13 ± 0.17	3.54 ± 0.02	0
SWW	6.20 ± 0.18	13.83 ± 0.03	1.44 ± 0.03	69.26 ± 0.48
SFF	9.33 ± 0.10	15.00 ± 0.09	1.29 ± 0.05	67.51 ± 1.11
WWFF	11.55 ± 0.28	15.42 ± 0.17	2.20 ± 0.03	57.84 ± 0.96
SWWFF	9.18 ± 0.23	15.12 ± 0.13	1.84 ± 0.01	62.38 ± 0.07
SCF	9.22 ± 0.76	15.06 ± 0.14	1.27 ± 0.02	67.45 ± 1.08
WWCF	10.99 ± 0.55	14.86 ± 0.04	2.07 ± 0.03	57.93 ± 0.91
SWWCF	8.84 ± 0.17	14.66 ± 0.14	1.82 ± 0.08	62.48 ± 0.12

Values in the same column followed by different letters are statistically different for $P \leq 0.05$.
S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour-Fine Flaxseed (39:51:10), SWWCF: Semolina-Whole Wheat Flour-Coarse Flaxseed (39:51:10), FF: Flaxseed Flour 100%, CF: Coarse Flaxseed Flour 100%.

Results of the water holding capacity (WHC) of the different formulations can be found in Table 3. Samples with 100 or 90% WW in the composition showed the highest WHC values, followed by formulations that contain both S and WW, and then by the formulations that contain 100 or 90% of S. This indicated that the WHC of whole wheat flour was much greater than the WHC of semolina. Bran fiber present in the whole wheat flour has the capacity to absorb water several times its dry weight (Robertson and Eastwood 1981). Flour particle size has also been reported as a factor that affects the WHC of flour formulations. Because flour is reduced to a smaller particle size than semolina it tends to have more starch damage (Grant et al 1993). Having more damaged starch granules enhances the amount of water absorbed by the flour, which can result in greater values of WHC (Kweon et al 2014).

The WHC for 100% fine flaxseed flour and 100% coarse flaxseed flour was of 2.84 and 1.70 ml of water per g of dry sample, indicating that for flaxseed flour a smaller particle size also increases the WHC of the flour. This is corroborated by the values of WHC reported in Table 3, in which all the formulations that contain fine flaxseed flour had a greater WHC than the same formulation with coarse flaxseed flour or with no flaxseed at all, e.g. SFF vs. SCF and S. The high WHC of flaxseed flour was attributed to the gum-like material or mucilage associated with the hull of the seed. This material corresponds to around 8% of the seed weight (Fedeniuk and Biliaderis 1994). The flaxseed gum has been reported to have excellent properties as a food hydrocolloid and can increase the WHC of the products where it is incorporated (Sun et al 2011).

WHC takes place by protein-water and carbohydrate-water interactions that occur in a large variety of food systems. WHC corresponds to the ability of a protein or carbohydrate matrix to absorb and retain water against gravity through bound, hydrodynamic, capillary, and physical mechanisms (Damodaran 1997; Mudgil and Barak 2013). WHC has an important role in hydration processes of cereal flours since it helps to determine how much water is needed to hydrate the flour for it to perform optimally (Kweon et al 2014).

Table 3: Water holding capacity and swelling power of different flour formulations.

Formulations	Water holding capacity
	ml water/g dry sample
S	0.57 k
SFF	0.79 h
SCF	0.64 j
WW	0.93 f
WWFF	1.21 c
WWCF	0.99 e
SWW	0.73 i
SWWFF	1.06 d
SWWCF	0.83 g

Values in the same column followed by different letters are statistically different for $P \leq 0.05$.
S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour-Fine Flaxseed (39:51:10), SWWCF: Semolina-Whole Wheat Flour-Coarse Flaxseed (39:51:10).

Particle size distribution

Particle size distribution of the tested samples studied in this experiment is presented in Figure 5 in Chapter 1. Semolina showed that 50% of the particles were retained on the 250 μm sieve and 26.3% were retained on the 150 μm sieve. A total of 50% of the whole wheat flour was retained in the 100 μm sieve, followed by 34.9% of the flour which was retained on the 50 μm sieve. Particle size distribution of the semolina-whole wheat mixture had a bimodal distribution of particles reflecting the small particle size associated with whole wheat flour and large particle size associated with semolina. Ground non-fractioned flaxseed flour showed larger particle size than semolina or whole wheat flour with 23.8% retained in the 424 μm sieve, and 46.7 and 27.3% retained in the 500 and 600 μm sieve, respectively.

Wet agglomeration of ingredient formulations

Agglomerate formation was extensively affected by the hydration level and the sample formulation and by the particle size of the ingredients included in the formulation. Agglomerate size increased as the hydration level increased and when semolina was used as the main ingredient vs. whole wheat flour. For the purpose of this paper, all agglomerates that can pass through a mesh size of 0.64 cm (0.25 in) were are deemed typical of pasta processing and do not cause problems during pasta extrusion. Conversely, large agglomerates (>0.64 cm) can cause bridging and disrupt the extrusion process (de la Peña and Manthey 2012).

SFF was the formulation with the highest percentage of agglomerates for all mesh sizes (Fig. 12). Particularly, for SFF hydrated at 34% moisture content, more than 49% (w/w) of the agglomerates did not pass through a 0.64 cm mesh sieve, of which 12.8% and 12.2% did not even pass through 1.27 and 1.91 cm mesh sieves, respectively (Fig. 12). For SFF hydrated at 33 and 32%, 34.0% and 10.2% of the material did not pass through the 0.64 cm mesh sieve, respectively. This extreme agglomeration behavior was not observed by any other formulation, regardless of hydration level. The ingredient formulation with the second highest rate of average agglomerate formation was SCF. For a hydration level of 32-34% the percentage-weight of agglomerates that did not go through the 0.64 cm mesh sieve ranged between 3.8-33.9% for this formulation. For the rest of the formulations, the percentage-weight of total wet formulation weight that did not go through a 0.64 cm sieve when the formulation was hydrated at between 32-34% ranged at follows:1.2-10.4% for S, 2.5-12.3% for WW, 9.3-14.5% for WWFF, 1.6-8.0% for WWCF, 0.9-3.4% for SWW, 12.0-13.6% for SWWFF, 0.8-3.6 for SWWCF.

Four major stages can be observed during the wet agglomeration of semolina and wheat flour (Barkouti et al 2014). The first stage is known as “nucleation” and it occurs at the beginning of the wetting stage. Nucleation consists of the instantaneous formation of nuclei from the native durum wheat particles as water droplets are incorporated in the bed bulk (Saad et al 2011). Nuclei are the result of the adhesion of two or more particles together. During this stage, the bulk powder is only composed of native semolina or wheat flour particles and nuclei (Barkouti et al 2014). Liquid bridges between semolina particles are involved in stabilizing the internal structure of the nuclei. As the water content increases, agglomerates begin to appear in the granular bed as a result of nuclei association. This second stage is called "nucleation and growth regime". At this stage, the granular bed is characterized by the presence of native semolina particles, nuclei, and agglomerates. Agglomerates can be differentiated from nuclei by their higher water contents and larger size. The nucleation and growth regime stage persists until all the native semolina particles disappear (Barkouti et al 2014). The third stage of wet agglomeration is called "growth of agglomerates", which involves the growth in size of the agglomerates formed in the previous stage. Consequently, the granular bed at this point is formed out of nuclei but mostly out of growing agglomerates. This is the wet agglomeration stage at which the formulations studied in this experiment were found after hydrating at 30-34%. When agglomerates become saturated with water their association results in dough pieces. The main difference between agglomerates and dough pieces is their moisture content. In addition, agglomerates show a plastic behavior, whereas dough pieces show an elasto-plastic behavior. Some dough pieces were found among the agglomerates collected in S, SFF, and SCF hydrated at 34%. The transition between the third and the fourth stage is called "plastic limit value", attributed to the change of the material behavior of the agglomerate described above, and it has been defined at a 52% hydration level for semolina (Rondet et al 2013). The fourth and last stage of wet agglomeration is called "paste transition" and it is observed when the moisture content is high enough to saturate all the agglomerates. At that point the agglomerates would be dough pieces that start to coalesce with each other to form a continuous dough.

Wet powder agglomeration is produced by adding a wetting liquid binder, water in this case, to a dry powder, i.e. semolina or whole wheat flour. The combined action of wetting and mechanical energy allows the wetted particles to bond together (Barkouti et al 2014). Semolina or flour particles bind together through capillary cohesion forces generated between the individual particles (Saad et al 2011; Schubert 1981). For wet agglomerates to form it is necessary that the individual semolina or whole wheat flour particles have a minimum water content known as capillary condensation value (Barkouti et al 2014). Capillary condensation is a process by which multilayer adsorption from the vapor phase into a porous medium proceeds to the point at which pore spaces become filled with condensed liquid from the vapor phase. In other words, capillary condensation corresponds to the minimum moisture content that an individual particle needs to have in order to be able to bond through capillary bridges with another particle. At that point, capillary bridges are formed causing two surfaces to adhere together. Barkouti et al (2014) indicated that dry native particles always had lower moisture content than the capillary condensation water content, agglomerated structures (nuclei and agglomerates) always had a moisture content higher than the capillary condensation value and lower than the “plastic limit value” for durum wheat semolina or wheat flour, and dough pieces always had a moisture content is found to be higher than the plastic limit.

The level of moisture content required to reach the capillary condensation value is ascribed to the sorption kinetics properties of the material. Semolina and whole wheat flour have different particle size and starch damage with gives then different hydration properties explaining why they agglomerate differently under same hydration levels (Hébrard et al 2013;

Roman-Gutierrez et al 2003). Because whole wheat flour has higher starch damage and smaller particle size than semolina, it is expected that flour has higher water absorption requirements to hydrate the same as semolina, with less starch damage (Oh et al 1985). This effect was corroborated by the WHC results. Smaller particle size increases water absorption because the surface area of the particles that need to be hydrated is increased. Starch damage increases water absorption because the intact granules can only absorb limited amounts of water in the absence of heat; however, a damaged granule can absorb large amounts of water because the structure of the granule has been physically broken (Farrand 1964). In addition, whole wheat flour has the bran particles mixed with the endosperm particles. Bran has the property of being able to absorb several times its own weight in water (Roozendaal et al 2012). Because of this characteristic, bran is going to compete for water with the flour particles when the water is incorporated. The water absorbed by the bran and the starch granules is not available anymore to saturate the protein fraction of the flour particles and form the necessary liquid capillary bridges required for agglomerate formation. Consequently, the result of the combination of starch damage, small particle size, and presence of bran would increase the capillary condensation value of whole wheat flour in comparison to semolina. This effect explains why samples with whole wheat in their formulation formed fewer large agglomerates than samples with semolina.

Fortification with flaxseed flour, especially flaxseed flour with fine particles, enhanced the agglomerate formation for all samples and increased the stickiness of the dough to the metal surface of the mixing bowl as also reported by Yalla and Manthey (2006). The stickiness of the dough was attributed to the soluble gum or mucilage that can leach from the hull of the flaxseed seed when exposed to moisture (Qian et al 2012) and to lipid expelled from the ground cotyledon part of the flaxseed flour. Both lipid and gum can also act as a particle binder increasing the effect of agglomerates formation as described in previously. Semolina seemed to be more susceptible than whole wheat to large agglomerate formation when blended with flaxseed flour. Flaxseed flour is a relatively hydrophobic material due to its high oil content which means that it will absorb less water per unit weight than either semolina or whole wheat. When 10% of the semolina, a highly hydrophilic material, was replaced with flaxseed flour, there is less semolina present to absorb the same amount of water than when no flaxseed fortification took place. This results in semolina particles becoming overhydrated which in combination with the presence of the flaxseed gum and lipid would favor the formation of large wet agglomerates. Whole wheat flour mixed with flaxseed flour did not form as large of agglomerates as did semolina. This behavior was mainly ascribed to the higher water absorption properties associated to whole wheat flour described above. Because of these properties, whole wheat flour samples containing flaxseed flour could be hydrated to up to 34% without detecting any extreme agglomeration problem. Similar behavior was observed for SWW, SWWFF and SWWCF.

The reason why fine flaxseed promoted agglomeration to a greater extent than coarse flaxseed is attributed to the smaller particle size of the material. Having a smaller particle size would enhance the exposure of the gums located at the hull and lipids in cotyledon of the seed to the semolina and whole wheat particles than the coarse flaxseed flour.

The standard hydration level of semolina for traditional pasta production is 32%; however, from an agglomerate formation point of view it could be hydrated to 33% and still not form large agglomerates that can cause flow disruption of ingredients. Results indicated that in order to avoid formation of large agglomerates, SFF should not be hydrated to more than 30%. For the rest of the formulations the maximum hydration level allowed to prevent extreme large agglomeration formation was: 32% for SCF, 33% for WWFF and SWWFF, and 34% for WW, WWCF, SWW, and SWWCF.

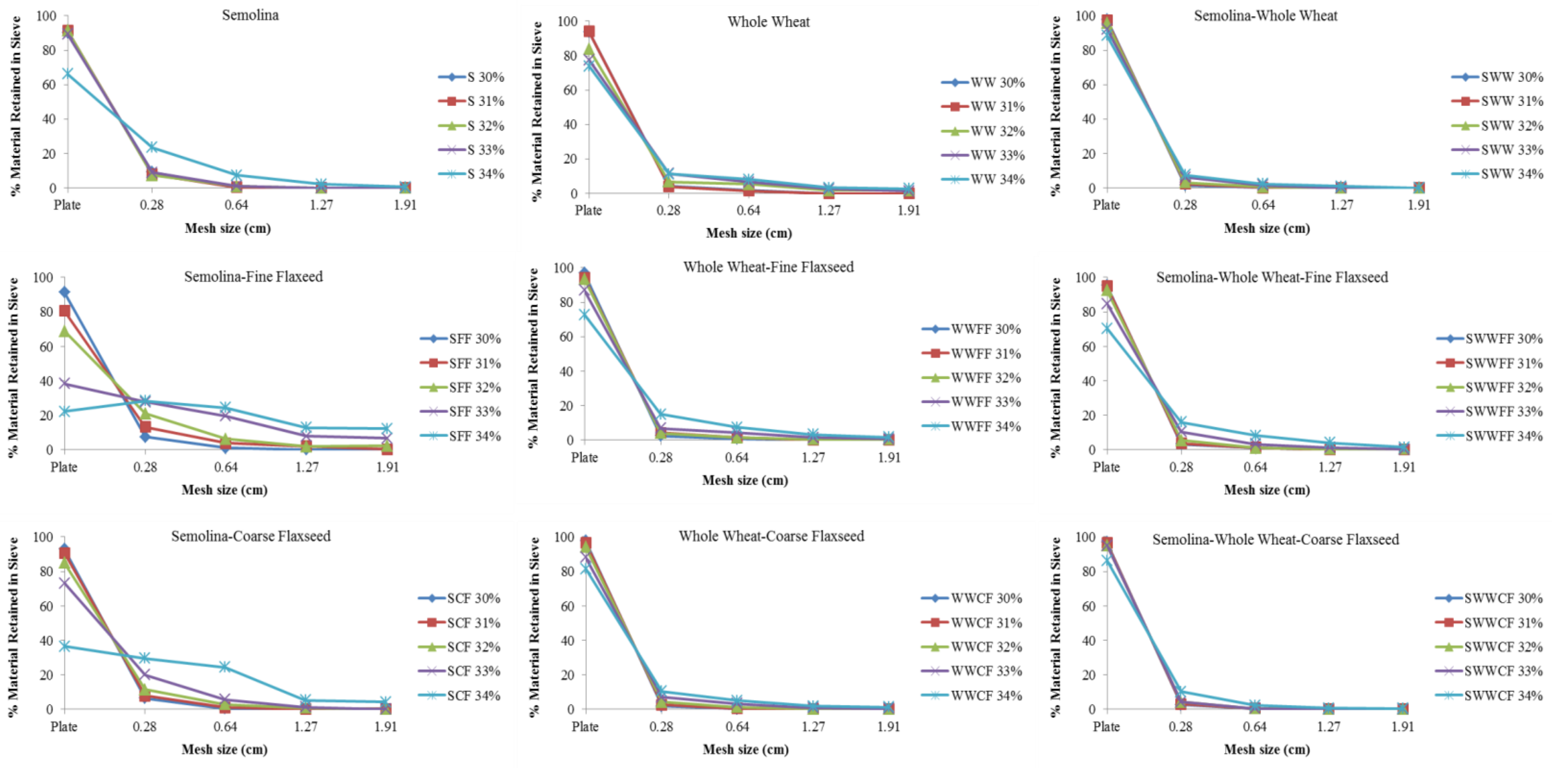


Figure 12. Particle size distribution of dough agglomerates using a rotary sieve shaker for the different formulations hydrated at different moisture content.

S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour-Fine Flaxseed (39:51:10), SWWCF: Semolina-Whole Wheat Flour-Coarse Flaxseed (39:51:10).

Scanning electron microscopy

The Scanning Electron micrographs of the raw ingredients are shown in Figure 13. Semolina particles (Fig. 13-a) were much larger than the whole wheat flour (Fig. 13-b) and particle size of coarse flaxseed (Fig. 13-d) was much larger and homogeneous than fine flaxseed particles (Fig. 13-c). The incorporation of water to these dry ingredients resulted in the formation of the agglomerates as discussed in the section above.

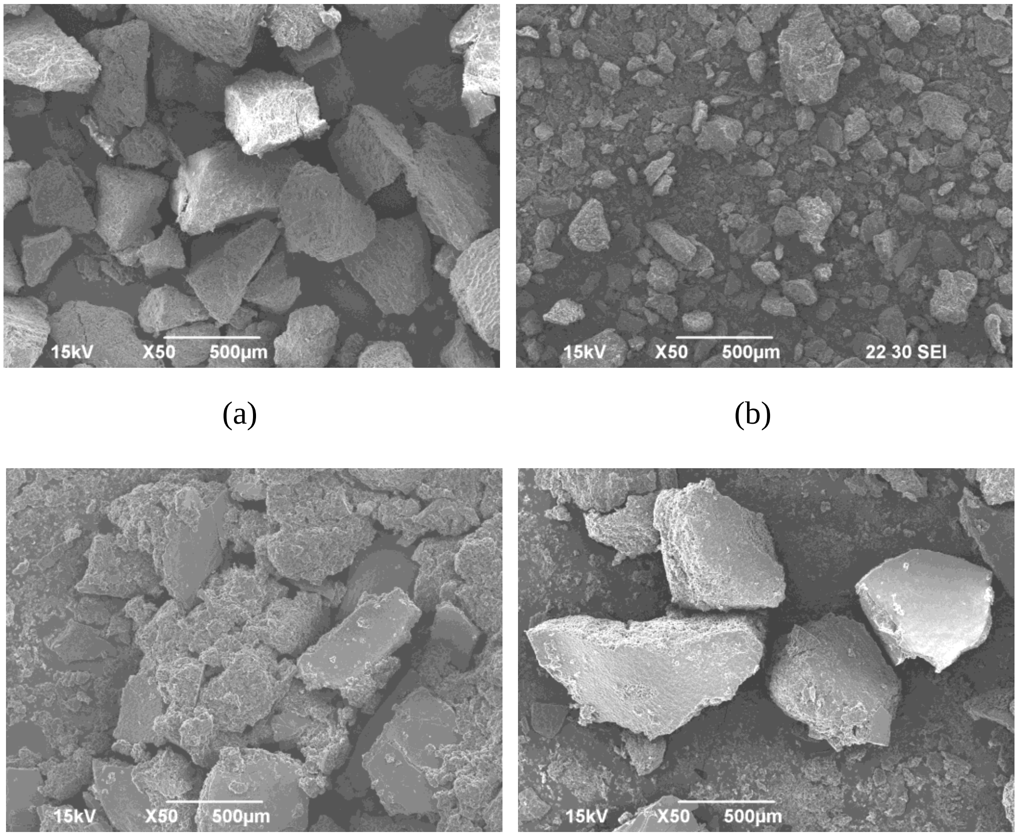
Scanning electron micrographs of agglomerate surfaces reveal the interaction of the individual particles among each other (Fig. 14). Only the micrographs of agglomerates obtained after hydrating the different formulations at 34% are presented since they best explain the results observed for the wet agglomeration. Semolina particles appear angular and are much larger than whole wheat particles. Particle size was greatest with coarse flaxseed, intermediate with fine flaxseed and semolina, and least with whole wheat particles.

Agglomerates of semolina appear to be less dense than agglomerates of whole wheat and this occurred with or without flaxseed particles. For S the SEM pictures (Fig. 14-a) showed that in some surface areas of the agglomerate it was possible semolina individually identify semolina native particles (Fig. 14-a1) of the same size and shape of native dry semolina particles (Fig. 14-a). In other areas of the agglomerate surface semolina particles seemed to be fused or molten altogether forming amorphous areas of variable size and shape that no longer retained the nature of the dry particle (Fig. 14-a2). This amorphous areas are made out of proteins that have been hydrated enough to lose their glassy state and that form a protein network in which the starch granules are dispersed (Icard-Vernière and Feillet 1999). The agglomerate structure observed for SCF (Fig. 14-g) was very similar to the one observed for S. For SFF (Fig. 14-d) no individual semolina particles could be observed since they are all fused together into much larger particles.

The structures seen in this case are like several “mini” aggregates (Fig. 14-d3) that together form a much larger aggregate with the flaxseed particles entrapped in between (Fig. 14-d4). These larger aggregates would be the physical units that are separated in the sieve in the wet agglomeration study described above. The more “mini” agglomerates formed, the more they combine together to form larger agglomerates that would be retained in the 1.27 cm and 1.91 cm mesh sieves. This observation verifies that the wet agglomeration stages in which the nine formulations are found is the stage three “growth of agglomerates”.

The surface of the WW agglomerate (Fig. 14-b) consisted mostly of individual flour particles in their native state (Fig. 14-b5) connected to each other. Individual particles of fiber could be identified in between the flour particles (Fig. 14-b6). Fewer and smaller amorphous areas were observed in comparison to S (Fig. 14-b2). WWCF (Fig. 14-e) displayed a similar agglomerate structure to WW. WWFF (Fig. 14-e) displayed more amorphous areas (Fig. 14-e2) than WW and WWCF, but such areas were fewer and much smaller than SFF. For both WWFF and WWCF it was hard to distinguish between bran and flaxseed particles embedded in the respective agglomerates.

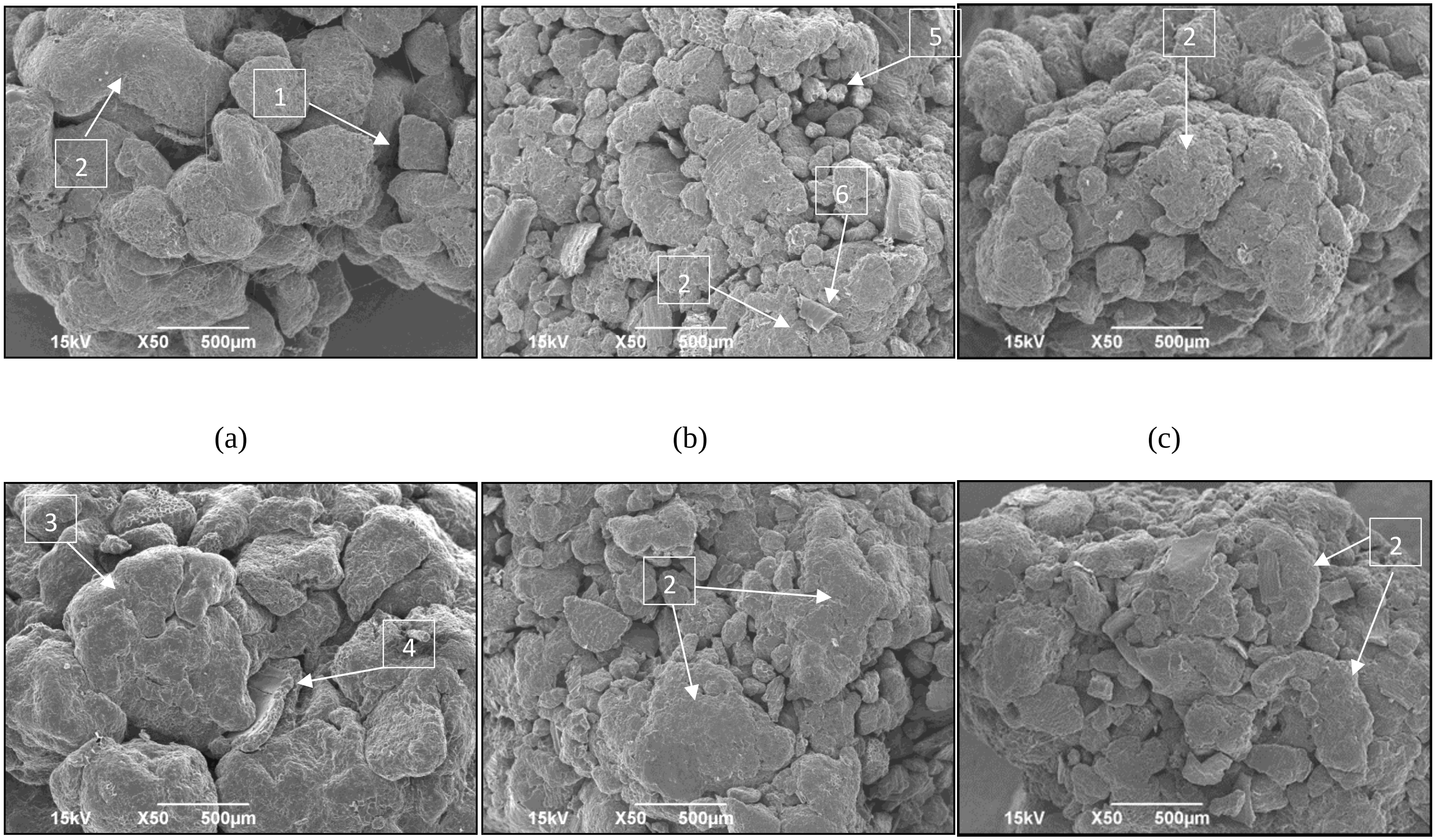
For SWW (Fig. 14-c), SWWFF (Fig. 14-f), and SWWCF (Fig. 14-i) the pictures exhibited characteristics intermediate to those with semolina or whole wheat as the main ingredient. In all three types of agglomerates, patches of both semolina and flour fused together to form amorphous zones are detected. It was possible to differentiate between semolina and whole wheat particles because of their difference in particle size; however, flaxseed and fiber were visible but not distinguishable in the agglomerates.



(a) (b)

(c) (d)

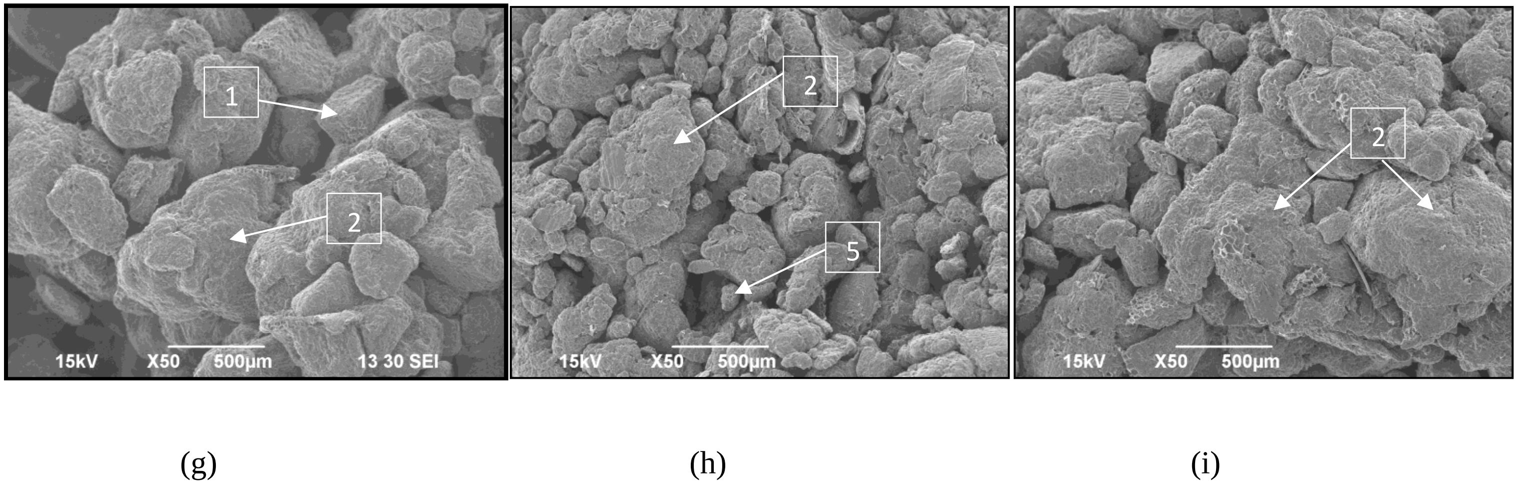
Figure 13. Micrograph of the dry ingredients used to make the formulations (a) semolina, (b) whole wheat flour, (c), fine flaxseed flour, (d) coarse flaxseed flour.



(a) (b) (c)

(d) (e) (f)

Figure 14. Micrograph of the agglomerates obtained from the nine formulations hydrated at 34% moisture content. 100% semolina (a), 100% whole wheat flour (b), semolina-whole wheat (49:51) (c), semolina-fine flaxseed flour (90:10) (d), whole wheat-fine flaxseed flour (90:10) (e), semolina-whole wheat-fine flaxseed (f), semolina-coarse flaxseed flour (90:10) (g), whole wheat-coarse flaxseed flour (90:10), (h), and semolina-whole wheat-coarse flaxseed (i). (1) Native semolina particles, (2) amorphous areas composed of fused particles, (3) mini aggregates, (4) flaxseed particle, (5) native flour particle, (6) fiber particle.



(g) (h) (i)

Figure 14. Micrograph of the agglomerates obtained from the nine formulations hydrated at 34% moisture content (continued). 100% semolina (a), 100% whole wheat flour (b), semolina-whole wheat (49:51) (c), semolina-fine flaxseed flour (90:10) (d), whole wheat-fine flaxseed flour (90:10) (e), semolina-whole wheat-fine flaxseed (f), semolina-coarse flaxseed flour (90:10) (g), whole wheat-coarse flaxseed flour (90:10), (h), and semolina-whole wheat-coarse flaxseed (i). (1) Native semolina particles, (2) amorphous areas composed of fused particles, (3) mini aggregates, (4) flaxseed particle, (5) native flour particle, (6) fiber particle.

Loose bulk density

The interaction between formulation and hydration level was significant ($P \leq 0.05$) for loose bulk density. The trend observed for a given formulation was that bulk density increased as the hydration level of the formulation increased, and that was true for all formulations (Fig. 15). It was anticipated that with increased moisture content, the bulk density values would be smaller than at low moisture levels due to over-hydration and large agglomerate formation having a less compacted structure but that was not the case. The explanation for this was that moisture displaced air and made particles more plastic and able to interact and stick with each other better.

For a given hydration level, samples with 90 or 100% semolina always had the highest bulk density, followed by samples with 100% or 90% of the combination of semolina and whole wheat, and then by samples with 90 or 100% whole wheat flour. Presence of flaxseed in SFF and SCF, and in SWWFF and SWWCF did not alter the bulk density from a statistical point of view for a given hydration level with respect to the correspondent formulation without flaxseed (S and SWW, respectively) but it did affect WWFF and WWCF in comparison to WW.

The loose bulk density results showed that using different hydration levels and formulations resulted in a product that for the same weight occupied a very different volume. The values recorded in this experiment can therefore provide useful information about the capacity that a mixing chamber and the size of the openings of the chutes should have when dealing with non-traditional ingredients or when the hydration level is changed.

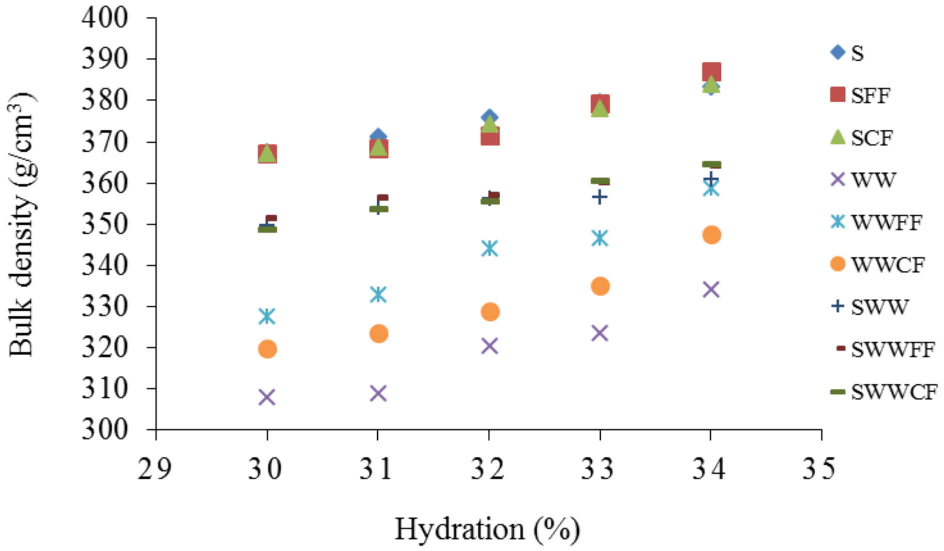


Figure 15. Bulk density of the different ingredient formulations when hydrated at different hydration levels.

S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour-Fine Flaxseed (39:51:10), SWWCF: Semolina-Whole Wheat Flour-Coarse Flaxseed (39:51:10).

Agglomerate compression

TPA analysis was only run in S, SFF, and SCF hydrated at 34% because they were the only samples for which enough agglomerates were retained on all four sieves to run the test. Figure 16 displays a typical TPA curve for an agglomerate. The Y-axis represents the force applied by the probe to compress the agglomerate whereas the X-axis displays the vertical distance travelled by the probe when compressing the agglomerate. Results of TPA test are summarized in Tables 5 and 6. The following are the descriptions for each parameter measured by TPA.

- Hardness is the peak force of the first compression of the product. It typically occurs at the point of deepest compression.

- Adhesiveness is the negative area for the first compression cycle representing the work needed to overcome the attractive forces between the surfaces of the probe and the food.
- Springiness is how well a product physically springs back after it has been deformed during the first compression.
- Resilience is how well a product "fights to regain its original position". It is possible to think of it as instant springiness since resilience is measured on the withdrawal of the first penetration, before the waiting period is started.

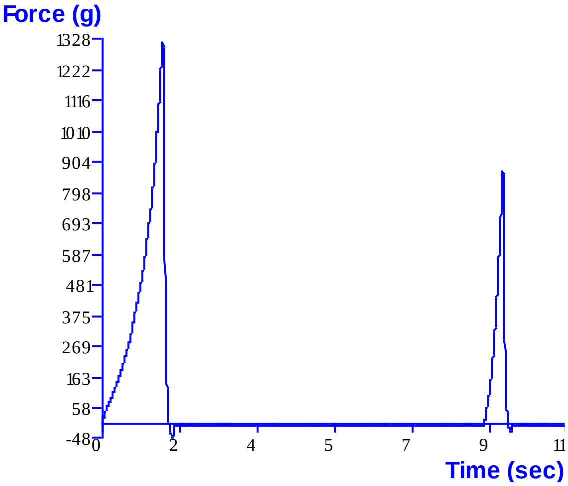


Figure 16. Typical agglomerate compression curve measured with the Texture Profile Analysis (TPA) with the Texture Analyzer. Agglomerate compression was 50% strain relative to the initial diameter for both compression cycles. Agglomerates were obtained from formulations hydrated at 34%.

Formulation by agglomerate size interaction was not significant for any of the TPA parameters measured. The formulation main effect was significant for hardness, and adhesiveness (Table 4), whereas the agglomerate size main effect was significant for hardness, adhesiveness, and springiness (Table 5) ($P \leq 0.05$). When averaged across agglomerate size, S and SCF displayed less hardness and more adhesiveness (higher negative value) than did SFF. The hardness results agree with the SEM micrographs displayed in Figure 14 that show much more compact agglomerates for the SFF, than for S, and SCF. The high adhesiveness recorded for SFF agreed with the extreme wet agglomeration phenomenon detected for this particular formulation in Figure 12. As mentioned above, fine flaxseed has lipid and gum exposed to the semolina. The adhesive properties of these compounds would explain the higher adhesive values detected in the SFF agglomerates. Coarse flaxseed flour has also lipid and gum like fine flaxseed flour, but its surface area is small compared to fine flaxseed flour, so that SCF adhesiveness is much closer to S, which lacks lipid and gum, than to SFF. The parameters springiness and resilience were very similar for all three formulations.

Table 4. Texture Profile Analysis (TPA) of pasta dough agglomerates hydrated to 34% and compressed at 50% strain. Data averaged across the mesh size of the sieve in which the agglomerates were retained (agglomerate size).

Formulation	Hardness	Adhesiveness	Springiness	Resilience
	g	g·sec	mm	-
S	1481.0 b	-30.57 a	0.435 a	0.145 a
SFF	1946.3 a	-68.89 b	0.395 a	0.147 a
SCF	1238.2 b	-37.63 a	0.412 a	0.131 a

Values in the same column followed by different letters are statistically different for $P \leq 0.05$.
S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), and SCF: Semolina-Coarse Flaxseed (90:10).

When data was averaged across formulation, it was possible to observe that the magnitude of the TPA parameters were very influenced by the agglomerate size (Table 5). Results showed that large agglomerates were harder and more adhesive, springy, and less resilient. This is ascribed to the moisture content of the agglomerates. Larger agglomerates have higher moisture contents (Barkouti et al 2014) which explains the differences recorded in their textural properties.

Table 5. Texture Profile Analysis (TPA) of pasta dough agglomerates averaged across the dough formulation.

Sieve mesh size where agglomerate retained	Hardness	Adhesiveness	Springiness	Resilience
cm	g	g·sec	mm	-
1.91	3481.2 a	-96.43 c	0.548 a	0.115 c
1.27	1661.5 b	-56.53 b	0.419 b	0.113 c
0.64	813.5 c	-26.97 ab	0.374 bc	0.136 b
0.28	254.7 d	-2.86 a	0.335 c	0.199 a

Values in the same column followed by different letters are statistically different for $P \leq 0.05$.

The data shown in Table 4 and 5 was collected only on those formulation-hydration levels combinations that formed agglomerates that were large enough to run the test. It is anticipated that if the other formulation-hydration levels combinations had resulted in agglomerates large enough to run this test, their textural differences would also relate to the different formulations and hydration levels.

The agglomerates formed during the mixing step, can range in numbers of hundreds or thousands for a given amount of flour, depending on the size of the agglomerates. After the mixing step, the agglomerates are moved into an extrusion auger inside the pasta extruder where they are pressed together and lose their individuality and form a continuous dough. Consequently, it is possible that the changes in the agglomerate textural properties could alter the texture of the dough formed and impact the way the dough behaves during the extrusion process. It seems logical to think that adding different hydration levels to the formulations would also influence the textural properties of the agglomerates, even if they are only a few microns long. Thus, additional research is needed to investigate the effect of dough hydration and spaghetti formulation on the processing properties of non-traditional pasta and relate these results to the changes to textural properties of the agglomerates.

Conclusions

This research identified the highest hydration level, within the studied range, that was possible to incorporate into a formulation without provoking the formation of large wet agglomerates. Those maximum hydration levels were 30% for SFF, 32% for SCF, 33% for S, WWFF and SWWFF, and 34% for WW, WWCF, SWW, and SWWCF. The formulations WW, WWFF, WWCF, SWW, SWWFF, and SWWCF formed much smaller agglomerates than S, SFF, and SCF. This indicated that the physical properties of the whole wheat flour, i.e. particle size, starch damage, and bran presence, helped to decrease the over-hydration of the protein matrix of the flour particles that lead to the formation of large agglomerates.

The formulation of the flour blends affected the textural properties of the agglomerates. It is possible that the changes in the agglomerate textural properties alter the rheology of the dough formed and impact the pasta extrusion process. Additional complimentary work is needed to investigate the effect of spaghetti formulation on extrusion properties of non-traditional pasta to assess the impact of changing the agglomerates textural properties on the pasta extrusion process. The effect of dough hydration level should also be studied since it is also assumed to contribute to the alteration of the textural properties of the agglomerates.

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PAPER 3[1]: RHEOLOGICAL PROPERTIES OF PASTA DOUGH DURING PASTA EXTRUSION: EFFECT OF MOISTURE AND DOUGH FORMULATION

Abstract

A study was conducted to investigate the effect of dough formulation and hydration level on the rheological properties of pasta dough during pasta extrusion. Semolina 100%, whole wheat 100%, and the following mixtures semolina-whole wheat (49:51), semolina-flaxseed flour (90:10), whole wheat-flaxseed flour (90:10), and semolina-whole wheat-flaxseed flour (39:51:10) were the formulations used for the experiments. Dough was hydrated at 30, 32, and 34% moisture content. Pasta was extruded with a capillary and a semi-commercial pasta extruder to determine the apparent viscosity of the dough during extrusion conditions and its relationship to the behavior of the dough during pasta processing. Results showed that non-traditional pasta dough behaved like a shear thinning fluid that can be described by the Power Law model. Increased hydration levels and/or presence of flaxseed flour on the dough formulation decreased the apparent viscosity of the dough, which correlated with extrusion pressure, mechanical energy, and specific mechanical energy that were required to extrude the dough in the pasta extruder. The strong correlations found between the apparent viscosity of the dough and the pasta extrusion parameters indicates the possibility of using a capillary rheometer to determine the appropriate hydration level of ingredient formulations before extruding with a pasta press.

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Introduction

Nowadays, consumption of fortified food products is becoming a new trend (Rodrigo and Vadillo 2004). In that regard, pasta is a staple food product that can be fortified with non-traditional ingredients, being especially important those that contribute to improve the essential amino acids and essential fatty acids profile or that increase the fiber, vitamins, and mineral content (Bahnassey and Khan 1986).

Both flaxseed flour and whole wheat flour can be considered as good candidates for pasta fortification due to the health benefits they both provide when consumed (Cardoso-Carraro et al 2012; Liu 2007). Fortification of pasta products with flaxseed flour or whole wheat flour have been previously studied by several authors (Hirawan et al 2010; Manthey and Schorno 2002; Manthey et al 2002; Sinha and Manthey 2008; Villeneuve et al 2013; Yalla and Manthey 2006), however the effect of the inclusion of both flaxseed and whole wheat flour together in pasta formulation has not been studied.

The addition of different raw materials during pasta dough preparation involves changes at different levels of the pasta production process (Roda 2013). It has been observed that when pasta dough is fortified with non-traditional ingredients it behaves differently than when only semolina is present (Rayas-Duartes et al 1996). The presence of flaxseed flour or whole wheat flour, alters the extrusion pressure, mechanical energy, extrusion rate and specific mechanical energy of pasta products (Yalla and Manthey 2006). These changes in pasta extrusion parameters are attributed to the effect of non-traditional ingredients in the rheological properties of the pasta dough. Rheology influences the machinability, processing conditions and quality of products (Chillo et al 2010). Subsequently, the study of dough rheology is important to understand the role of non-traditional ingredients in the flowing properties of pasta dough during extrusion and to evaluate the suitability of a given dough formulation to the extrusion processes. Rheology studies the flow and deformation of materials e.g. wheat doughs, under the action of stresses (Bagley et al 1998). Rheological characteristics of cereal doughs using a capillary rheometer, especially for bread making applications, have been previously described in the literature (Bagley 1998; Cuq et al 2002; Hicks and See 2010; Singh and Smith 1999). Cuq et al (2003) has reported the visco-plastic behavior of fresh pasta and the textural changes that pasta exhibits with changes in moisture content and temperature by using a modified TA-XT2 instrument. Other authors have studied the rheological behavior of plain and fortified semolina and flour dough through farinograph, and mixograph studies (Bahnassey and Khan 1986; Baiano et al 2011; Doxastakis et al 2007; Kovacs et al 1997). However, the behavior of semolina dough during extrusion has not been extensively studied from a rheological standpoint (Chillo et al 2010; Le Roux and Vergnes 1995).

Capillary rheology is a technique based on the application of high stresses and high shear rates during the extrusion of the material through a capillary die (Macosko 1994). Determination of the pressures necessary to force the material through the capillary die with a given flow rate as well the expansion swelling at the exit of the capillary enable the determination of shear and extensional viscosity of the material (Macosko 1994). The concept of capillary rheology is similar to fluid flow in pipes and extrusion dies, which makes it suitable to study the rheological properties of extruded products (Macosko 1994). A capillary rheometer can be adapted to the characterization of shear sensitive fluids such as wheat flour dough (Cuq et al 2002) therefore its utilization for the characterization of pasta dough is justifiable.

The objective of this experiment was to characterize the rheological behavior of pasta dough under extrusion conditions in a semi-commercial extruder with the help of a capillary rheometer when the dough formulation and hydration level was varied. For that purpose, pasta dough was fortified with whole wheat flour and/or with flaxseed flour and the hydration level of the dough was varied from 32%, commonly used for traditional semolina pasta products, to 30% and 34% which yield under-hydrated and over-hydrated traditional pasta products, respectively.

Materials and Methods

Raw material

Experiments were performed with commercial semolina (Durakota No.1) from North Dakota Mill (Grand Forks, ND) and whole hard white wheat flour (Ultragrain) from ConAgra Foods (Omaha, NE). Flaxseed flour was obtained by milling brown flaxseed on a pilot scale hammer mill (Fitzmill, Model DA506, Elmhurst, IL) as described by de la Peña and Manthey (2014). The ground flaxseed was separated into fine and coarse particles by using a sieve with a 34XXG 600 µm mesh screen.

A total number of six different pasta formulations were used for this experiment: Semolina 100% (S), whole wheat flour 100% (WW), and a blend of semolina:whole wheat (SWW) 49:51, semolina:fine flaxseed 90:10 (SFF), whole wheat:fine flaxseed 90:10 (WWFF), and semolina:whole wheat:fine flaxseed 39:51:10 (SWWFF). Maintaining 51% of whole wheat flour in the formulations was necessary to be classified as a whole wheat product. Uniform blends were prepared by mixing ingredients for 5 min using a cross-flow blender (Patterson Kelly, East Stroudsburg, PA, USA). The 10% (w/w) level of fortification with flaxseed flour was selected because previous research indicated that at this level, the pasta dough had proper consistency for extrusion and the final product had acceptable quality (Sinha and Manthey 2008).

Rheological studies

A twin-bore RH 2000 Rosand capillary rheometer (Malvern Instruments, Southborough, MA) was used to measure the shear viscosity of the pasta dough. The ingredients were mixed for 1 min in a Kitchen Aid^{TM} mixer (Troy, Ohio, USA) and then loaded on to the rheometer. The rheometer was preheated to 45 °C, temperature at which pasta extrusion normally occurs. Temperature was controlled by 3 heaters along the length of the barrel. For the current experiment only the left barrel was used. The diameter of the barrel was 24 mm and approximately 88 g of material was required to fill it. The capillary die used had an *L/D* ratio of 8 (32 x 4 mm). The rheometer piston was brought into contact with the dough for the preconditioning step. The dough was allowed to rest in the barrel for 3 min to allow the dough to relax and equilibrate at 45 °C without significant moisture loss (Hicks and See 2010). The experiment was performed by measuring the extrusion pressure required to extrude the dough at a piston speed of 21.4, 52.6, 129.8, and 320 mm/min. The software, Flowmaster (Mentor Graphics, Wilsonville, Oregon), was programmed to measure the viscosity readings only when pressure equilibrium was reached. Some samples took longer to reach pressure equilibrium, so more than one load was tested to the get a steady pressure measurement for the four selected piston speeds. Triplicate readings of pressure were recorded for each piston speed using freshly prepared dough.

Shear stress at the capillary wall (τ_w) was determined using Eq.2.

$$\tau_w = \Delta P/(4 \cdot L/D) \tag{2}$$

where τw is shear stress in kPa, ΔP is the pressure exerted in the dough in a kPa, and L/D is the ratio of length and diameter of the capillary, a dimensionless number.

The apparent shear rate was calculated by using the flow rate at which the dough was extruded, Q, and the radius R of the capillary die.

$$\dot{\gamma}_{\mathrm{app}} = 4 \cdot \mathrm{Q}/\pi \cdot \mathrm{R}^3 \tag{3}$$

Q (m^3/s), was calculated by multiplying the extrusion speed of the dough at the capillary die (m/s) by the area of the capillary die ($\pi \cdot R^2$) (m^2). The true wall shear rate ($\dot{\gamma}_w$) was obtained by applying the Rabinowitch correction:

$$\dot{\gamma}_{\mathrm{w}} = 1/4 \cdot (3 + \mathrm{d}\,(\log \dot{\gamma}_{\mathrm{app}})/\mathrm{d}(\,\log \tau_{\mathrm{w}}))\dot{\gamma}_{\mathrm{app}} \tag{4}$$

Using the shear stress and shear rate calculated from Eq. (2) and (4), respectively, the power law model was fitted to obtain the consistency index, *k*, and the flow index, *n*, for each dough formulation-hydration level combination. The power law model is described by Eq. (5).

$$\tau_{\mathrm{w}} = \mathrm{k}\,\dot{\gamma}_{\mathrm{w}}^{\mathrm{n}} \tag{5}$$

Units of the consistency index *k* are in kPa·s^{n} and the flow behavior index *n* is dimensionless. The power law model was utilized to build flow curves for each dough formulation-hydration levels combination according to the following equation:

$$\eta = \mathrm{f}(\dot{\gamma}) = \frac{\tau_{\mathrm{w}}}{\dot{\gamma}_{\mathrm{w}}} = \frac{\mathrm{k}\dot{\gamma}_{\mathrm{w}}^{\mathrm{n}}}{\dot{\gamma}_{\mathrm{w}}} = \mathrm{k}\gamma\dot{\mathrm{w}}^{\mathrm{n}-1} \tag{6}$$

The variable η is the apparent viscosity of the dough and is expressed in kPa·s.

Pasta processing

A total weight of 1000g of the different dough formulations were hydrated to 30, 32, and 34% moisture and extruded using circular dies that provided a spaghetti shape using a semi-commercial laboratory extruder (DEMACO, Melbourne, FL). Extrusion conditions were:

extrusion temperature, 45 °C; mixing chamber vacuum, 46 cm of Hg; auger rotating at a constant speed of 25 rpm. The extruder auger had a length to diameter ratio of 8.1:1, a constant root diameter 4.3 cm and uniform pitch of 2.4 cm along the entire auger length. Products were shaped on the die head that has 84 Teflon coated circular openings of 1.5 mm diameter and 2.3 mm long. Mechanical energy (J/s), extrusion rate (g/s), and extrusion pressure (kPa) were recorded for each sample as the pasta extrusion parameters. The mechanical energy required to operate the empty pasta press was subtracted from the mechanical energy used to operate the press under load as described by Manthey et al (2004). Accordingly, specific mechanical energy (J/g), was also calculated by dividing values of mechanical energy by values of extrusion rate.

In addition, extrudate lineal velocity (v) (m/s) was also measured for calculation of the dough flow rate of pasta dough, Q_e (m^3/s), in a single die opening during pasta extrusion (Eq. 7).

$$\mathrm{Q_e} = \mathrm{v} \times \mathrm{A} \tag{7}$$

The variable A is the extrusion area (m^2) of one opening of the extrusion die. Apparent shear rate of the dough during pasta extrusion ($\dot{\gamma}_{app_e}$) was calculated with Eq. (8), where the variable r is the radius of each of the openings in the extrusion die through which pasta dough was extruded.

$$\dot{\gamma}_{\mathrm{app}_e} = 4 \cdot \mathrm{Q}/\pi \cdot \mathrm{r}^3 \tag{8}$$

Shear stress at the capillary wall for the pasta extruder (τ_{w_e}) was calculated by Eq. (2). The Rabinowitch correction was then applied to calculate the true wall shear rate ($\dot{\gamma}_{we}$) at which each dough extruded in the semi-commercial pasta extruder as described in Eq. (5). Apparent viscosity of the pasta dough samples, η_e, was determined by entering $\dot{\gamma}_{we}$ in Eq. (6) and by entering the appropriate *k* and *n* value for each dough formulation-hydration determined experimentally with the capillary rheometer as explained above.

Statistical analysis

Analysis of variance of pasta extrusion parameters, consistency index, *k*, and flow index, *n*, and apparent viscosity was performed considering experimental layout as a split plot arrangement in which the whole plot factor was the spaghetti formulation (S, SFF, WW, WWFF, SWW, and SWWFF), and the subplot factor was three hydration levels (30, 32, and 34%). The experimental design for whole plot was a completely randomized design and all the experiments were performed in triplicate. Data were subjected to analysis of variance using ‘Mixed’ procedure in SAS System for Windows (v 9.3, SAS Institute, Cary, NC). *F*-Test was significant at $P<0.05$. Treatment means were separated by Fisher’s protected Least Significant Difference test calculated at $P=0.05$ using SAS 9.3 statistical package. Pearson’s correlation analysis between rheological and extrusion parameters were also calculated for $P\leq 0.05$.

Results and Discussion

Rheological studies

Two different capillary dies of 2 mm and 4 mm of diameter and 32 mm long were used to collect preliminary data. When the 2 mm-diameter capillary die was used, the pressure inside the barrel of the rheometer reached very high levels (approximately 28 MPa) for most of the formulation-hydration level combinations, which triggered the safety switch to stop the test. Similar problems had been previously reported by Le Roux and Vergnes (1995) who decided to use a large capillary diameter (3 mm) and a short capillary length (25 mm) to avoid the difficulty of obtaining unreliable measurements for pasta dough in a capillary rheometer. Thus, the 4 mm diameter capillary die was selected to complete the experiment. For samples S, WW and SWW hydrated at 30%, it was not possible to record pressure values for piston speeds above 215 mm/min because the pressure required for extruding the dough was too high and safety switch stopped the test. For the samples that contained flaxseed, it was possible to record pressure values at all hydration levels for the four piston speeds at which the dough was extruded in the capillary rheometer.

The shear stress and shear rate data obtained with the capillary rheometer and corrected using the Rabinowitsch formula were adequately described by the power law model as revealed by R^2 values greater than 0.90. The shape of the apparent viscosity curve described by Eq. (6) indicates that pasta dough is a pseudoplastic fluid shear-thinning fluid (Fig. 17), similar to what was reported by Le Roux and Vergnes (1995) and Chillo et al (2010). The pseudoplastic shear-thinning behavior observed in the dough is attributed to the macromolecular characteristics of the protein molecules. At low shear rates protein molecules are in random arrangement but at high shear rates they get aligned in the direction of shear, thus reducing the viscosity of the dough (Chillo et al 2010).

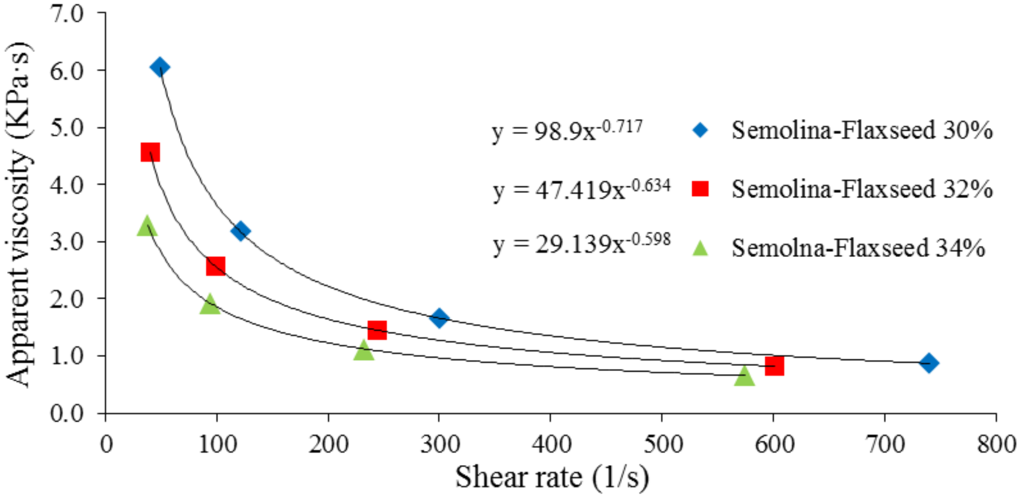

Figure 17. Flow curve for the Semolina-Flaxseed dough formulation hydrated at 30, 32, and 34% hydration levels.

The *k* values given by the power law model ranged between 236.9 kPa·s^n, for WWFF-30%, and 26.7 kPa·s^n, for SWWFF-34% (Table 6). Within the same dough formulation, *k* values decreased when the hydration level of the dough increased from 30 to 32 or 34%, which agrees with data reported by Singh and Smith (1999) and Martin et al (2003). The inclusion of flaxseed in the dough formulation reduced the *k* values regardless of the hydration level of the dough in comparison for the same dough formulation without flaxseed. The flow index, *n*, was also influenced by moisture content and the presence or absence of flaxseed (Table 6). Overall *n* values increased with moisture content, behavior that has been also reported by Singh and Smith (1999). Values of *n* ranged from 0.184, for WWFF-30%, and 0.412, for SWWFF-34%. In general, the presence of flaxseed increased the *n* value of all samples, except for WW and SWW hydrated at 30% in which higher *n* values were obtained when flaxseed was not present.

Table 6. Consistency coefficients, *k*, and flow index, *n*, values calculated with the capillary rheometer for each dough formulation-hydration level combination.

Sample	*k* (kPa·s^n)	n (-)
S 30%	175.1 bc	0.214 fg
S 32%	105.9 gfe	0.295 cde
S 34%	51.9 hi	0.372 ab
WW 30%	206.9 ab	0.254 efg
WW 32%	169.3 bc	0.273 def
WW 34%	118.9 def	0.279 def
SWW 30%	222.1 a	0.245 efg
SWW 32%	146.0 cde	0.287 def
SWW 34%	68.2 ghi	0.334 bcd
SFF 30%	101.1 efg	0.284 def
SFF 32%	46.8 hi	0.366 abc
SFF 34%	29.3 i	0.404 ab
WWFF 30%	236.9 a	0.184 g
WWFF 32%	90.4 fgh	0.272 def
WWFF 34%	32.0 i	0.399 ab
SWWFF 30%	156.4 cd	0.214 fg
SWWFF 32%	44.7 ghi	0.380 ab
SWWFF 34%	26.7 i	0.412 a

Values in each column followed by different letters are significantly different ($P \leq 0.05$).
S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour- Fine Flaxseed (39:51:10).

The consistency coefficient, *k*, and the flow index, *n*, are parameters that reflect the rheological properties of the dough during its extrusion in the capillary rheometer. Reduced *k* values when the moisture of the dough was increased from 30% to 32, and 34% (Table 6) indicates that water is a plasticizer that decreases the consistency of the dough and increases its fluidity, facilitating the extrusion of the dough in the capillary rheometer. The flow index, *n*, is a dimensionless number that indicates the level of non-Newtonian behavior of a dough. The further the *n* value of a fluid is from 1, the more non-Newtonian-like behavior that fluid displayed. Water is a Newtonian fluid, therefore, as expected, the *n* values of a given dough formulation became closer to 1 as the moisture content of the dough increased (Table 6).

Flaxseed also acted as a dough plasticizer as it reduced *k* values of samples when compared to the same type of dough with no flaxseed. The high lipid content of flaxseed (Manthey et al 2002) reduces the amount of water that flaxseed can absorb (Villeneuve et al 2013), making more water to be available for the other ingredients during ingredient hydration step. For a given hydration level, samples that were fortified with 10% of flaxseed, had 10% less of S or WW in the formulation to absorb the water as explained in Chapter 2. This resulted in increased hydration of the S or WW particles and in lower *k* values than the same sample without flaxseed. Thus, some samples containing flaxseed had a visual appearance of being wetter than the same sample without the presence of flaxseed. This effect was particularly noticed in samples containing S versus samples with WW as the main ingredient.

WW contains the bran fraction of the wheat kernel. Wheat bran has higher water absorption than S or wheat flour (Kaur et al 2012). Therefore, part of the water incorporated to samples with WW was absorbed by the bran decreasing the amount of water actually absorbed by the flour particles. In addition whole wheat flour had more starch damage and smaller particle size which increases the water absorption capacity of the flour in comparison to semolina as explained in Chapter 2. These properties made samples with WW behave like a drier dough than S samples hydrated to the same level, and that was reflected in the rheological behavior of the dough when extruded in the capillary rheometer.

Pasta processing

The true wall shear rate under which samples were extruded in the pasta extruder ranged between 41.6 s^{-1}, for SFF-34%, and 90.8 s^{-1}, for SWWFF-30% (data not presented). Those values were contained within the range of shear rates at which the dough was extruded in the capillary rheometer making possible the calculation of the apparent viscosity of the dough with the models generated with the capillary rheometer.

Results showed that the apparent viscosity of the pasta dough extruded in the semi-commercial pasta extruder was greatly affected by formulation and hydration level of the dough (Fig. 18). The higher the moisture content of the dough, the lower the apparent viscosity of the extrudate, as it has been reported by other researchers (Sandoval and Barreiro 2007). Flaxseed also reduced the apparent viscosity of the dough. This effect was especially noticeable for the WWFF for which the apparent viscosity of the dough was reduced nearly in half when compared to the apparent viscosity of that formulation when no flaxseed was present.

Reduction of the apparent viscosity of the dough when the hydration level was increased and when flaxseed was present reflects the plasticizing effect of both water and flaxseed on the rheological behavior of the dough under extrusion conditions (Fig. 18). The apparent viscosity of the dough is directly dependent on both the values of *k* and *n,* as described in Eq. 6. Since both *k* and *n* were affected by the hydration level and the presence of flaxseed it was expected that apparent viscosity of the dough would respond in the same way.

Traditional pasta dough, 100% semolina, typically is hydrated to 32% moisture content, and therefore the apparent viscosity of this formulation-dough hydration will be used as a reference value, marked with a horizontal line in Figure 18. According to Figure 18, the optimum hydration level of WW formulation to display the similar apparent viscosity as the traditional pasta dough was above 34%; whereas for SWW samples, the optimum hydration level would be included between 32-34%. For the remaining non-traditional dough formulations, the optimum hydration level was 30, 32, and 30% for SFF, WWFF and SWWFF, respectively.

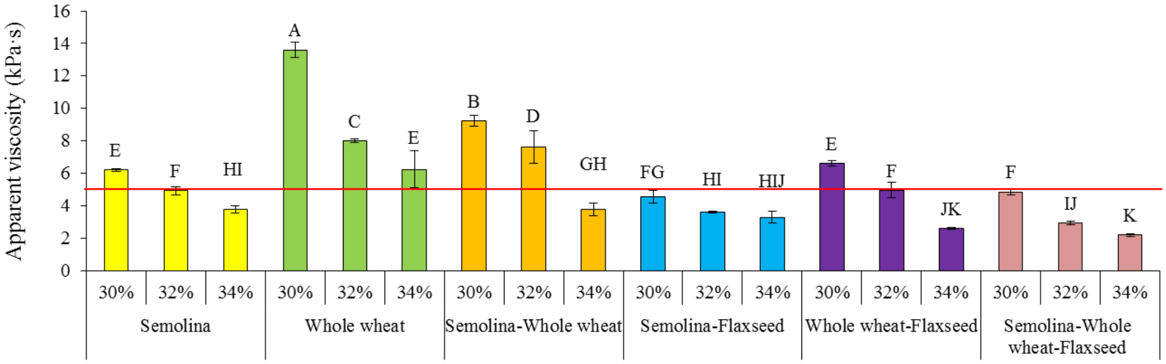

Figure 18. Apparent viscosity values for each dough formulation-hydration level combination. Values in each bar followed by different letters are significantly different (*P*≤0.05).

The fact that SFF samples required 2% less water to have the same apparent viscosity than the reference sample is attributed to the "wetting" effect of the flaxseed described above. On the other hand, WW samples displayed much higher values of apparent viscosity than the reference sample, even at a 34% of hydration. The bran fraction of the WW absorbed water therefore they need more water to display the same rheological behavior than the reference sample. The "buffer" capacity of whole wheat bran and the starch damage of flour on the hydration of dough made samples containing WW more tolerant to the presence of flaxseed than samples with no WW on the formulation, in agreement with the results showed in Chapter 2. This effect was particularly noticeable for the WWFF formulation that required 32% of hydration to have same rheological behavior than the reference sample, versus SFF that required a hydration of 30% to display the same behavior.

Dough with low moisture levels required the highest extrusion pressure, mechanical energy, and specific mechanical energy during the process. Intermediate values were recorded for 32%, followed by 34% moisture (Table 7). Extrusion pressure, mechanical energy, and specific mechanical energy were significantly lower for samples with flaxseed. Extrusion rate was also reduced by increased hydration level and presence of flaxseed but to a smaller extent than the other parameters.

Table 7. Extrusion data collected with the semi-commercial extruder for each dough-moisture combination.

Sample	Extrusion pressure	Extrusion rate	Mechanical energy	Specific mechanic energy
	kPa	g/s	J/s	J/g
S 30%	6598 a	3.9 ab	417.3 a	109.4 ab
S 32%	4541 de	3.8 ab	271.3 c	70.4 cde
S 34%	3068 hi	2.8 efg	168.7 hij	60.4 d-f
WW 30%	5582 b	3.3 a-f	355.7 b	117.4 a
WW 32%	4699 cd	3.1 c-g	262.5 c	79.7 c
WW 34%	3759 fg	3.0 d-g	187.0 fgh	63.1 d-g
SWW 30%	5175 bc	4.0 a	415.8 a	103.6 b
SWW 32%	4398 de	3.7 a-d	252.3 cde	68.7 cde
SWW 34%	3422 gh	3.4 a-e	186.3 fgh	54.3 fg
SFF 30%	3718 fg	3.4 a-e	220.7 def	65.1 def
SFF 32%	2500 ijk	2.9 efg	160.6 hij	54.7 fg
SFF 34%	1500 l	2.3 g	142.3 ij	63.7 d-g
WWFF 30%	3943 efg	3.0 d-g	214.9 efg	71.3 c-d
WWFF 32%	2999 hij	3.0 d-g	178.9 hij	59.2 efg
WWFF 34%	2380 jk	2.9 efg	158.4 hij	53.9 fg
SWWFF 30%	4297 def	3.7 abc	255.9 cd	69.2 cde
SWWFF 32%	2886 hij	3.3 b-f	173.1 hij	53.3 g
SWWFF 34%	2013 kl	2.6 fg	138.6 j	52.7 g

Values in each column followed by different letters are significantly different ($P \leq 0.05$).
S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour- Fine Flaxseed (39:51:10).

The rheological behavior displayed by the dough in the pasta extruder followed the same trend as the behavior of the dough displayed in the capillary rheometer. Extrusion pressure corresponded to the pressure that was needed to cause the dough to flow through the extrusion die and mechanical energy corresponded to the energy consumed by the pasta extruder while extruding a given dough. By increasing the hydration level of the dough, the dough became more malleable and fluid, reducing the extrusion pressure and mechanical energy required to extrude the dough. In pasta extruders, extrusion pressure is the result of interactions of screw speed, temperature, and flour hydration (Abecassis et al 1994). Since the screw speed and the temperature were kept constant during the extrusion process (25 rpm and 45 °C, respectively), variation in extrusion pressure values reflects the effect of dough hydration. Flaxseed also reduced the extrusion pressure and mechanical energy, which is attributed to the high lipid content of the flaxseed that acted as a lubricant reducing the friction between the dough and the screw elements and the barrel facilitating the dough extrusion process (Colonna and Mercier 1983; Yalla and Manthey 2006).

Specific mechanical energy measured the amount of energy that was consumed by the pasta extruder in order to extrude one gram of pasta. Low specific mechanical energy were expected in those samples were flaxseed was incorporated. This effect has been previously reported by Yalla and Manthey (2006) and it is attributed to the reduction of dough strength due to the disruption of the gluten matrix by the flaxseed.

Extrusion rate is determined by the screw speed and the open surface area of the die (Abecassis et al 1994). The slight decrease in extrusion rate displayed for samples with higher hydration levels and/or containing flaxseed on its composition was attributed to the slippage of the dough at the surface of the barrel due to the softening effect of the water added to the dough and/or to the lubricating effect of the lipid content of the flaxseed present in the dough.

Pearson correlation analysis revealed a strong correlation between *k* and all the pasta extrusion parameters except for extrusion rate (Table 8). Apparent viscosity and extrusion pressure, mechanical energy, and specific mechanical energy also displayed strong positive correlations. The correlations found between the consistency coefficients determined with the capillary rheometer, *k*, and extrusion pressure, mechanical energy, and specific mechanical energy measured with the pasta extruder are a good indication of the suitability of the capillary rheometer to study the rheological properties of pasta dough under extrusion conditions. The strong positive correlation found between the apparent viscosity and extrusion pressure, mechanical energy, and specific mechanical energy indicates that target extrusion pressure, mechanical energy, and specific mechanical energy values can be achieved during the extrusion process by adjusting the dough hydration.

Table 8. Pearson correlation coefficient, *r*, between rheological and extrusion parameters for $P<0.05$ ($n=27$).

Parameters	Extrusion pressure	Extrusion rate	Mechanical energy	Specific mechanical energy
Consistency coefficient, *k*	0.81	0.53	0.72	0.78
Apparent viscosity	0.75	0.34	0.74	0.84

Conclusions

The behavior of pasta dough observed during pasta extrusion can be explained by the rheological studies performed with a capillary rheometer. The consistency coefficient and flow index calculated after dough extrusion in the capillary rheometer were strongly affected by changes in the hydration level of only ±2% and by the formulation of the dough. Such effect was reflected in the processing parameters when the dough was extruded in a semi-commercial pasta extruder. An estimation of the apparent viscosity of a dough when dough was extruded in a semi-commercial pasta extruder could be determined thanks to the data provided by the capillary rheometer. The strong correlations found between the apparent viscosity of the dough and the pasta extrusion parameters indicates the possibility of using a capillary rheometer to determine the appropriate hydration level of ingredient formulations before extruding with a pasta press.

The level of hydration recommended for the studied formulations so that they display similar rheological behavior as traditional pasta dough was as follows: 30% for SFF and SWWFF, 32% for WWFF, 33% for SWW, and 34.5-35% for WW.

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PAPER 4: EFFECT OF FORMULATION AND DOUGH HYDRATIONLEVEL ON THE EXTRUSION, PHYSICAL PROPERTIES, AND COOKED QUALITY OF NON-TRADITIONAL SPAGHETTI

Abstract

An experiment was conducted to determine the effect of formulation and dough hydration on the extrusion and physical properties and on the cooked quality of non-traditional spaghetti. The formulations used for this experiment were semolina 100%, whole wheat 100%, semolina-whole wheat (49:51), semolina-flaxseed (90:10), whole wheat-flaxseed (90:10), and semolina-whole wheat-flaxseed (39:51:10). Flaxseed flour was incorporated as fine or coarse particles, for a total of nine treatments. Formulations were hydrated to 30, 31, 32, 33, and 34% before extrusion. Spaghetti required less pressure and mechanical energy during extrusion, had the brightest color, and required the shortest time to cook at 34% than at 30% hydration. Inclusion of flaxseed flour in the formulation also facilitated the extrusion process. Presence of whole wheat and flaxseed flour decreased the mechanical strength and flexibility of the spaghetti, decreased its brightness and yellowness, and increased its redness. Better cooked quality was obtained for whole wheat spaghetti with 49% semolina than for 100% whole wheat spaghetti. Spaghetti containing flaxseed flour had the best quality when in combination with 90% semolina. Small changes in cooked quality were observed after cooking non-traditional spaghetti beyond its optimum cooking time, regardless of the hydration level, indicating the tolerance of the spaghetti to overcooking.

Introduction

Consumption of fortified food products is a growing trend due to the awareness of consumers about the importance of following a healthy diet. Because of this situation, the pasta industry has started to manufacture non-traditional pasta products that contain ingredients different than semolina that provide additional nutritional value to the end product. In this regard, whole wheat flour and flaxseed flour are two excellent fortification ingredients because of their well-documented benefits on human health (Cardoso-Carraro et al 2012; Liu 2007).

The incorporation of non-traditional ingredients requires changes in the pasta manufacturing process (Roda 2013). Non-traditional ingredients possess different water absorption capacities than semolina that generally result in the alteration of the extrusion parameters (de la Peña and Manthey 2014a; Yalla and Manthey 2006). Rheological studies performed by de la Peña et al (2014) showed that pasta dough with non-traditional ingredients displayed different rheological properties than did traditional pasta dough causing changes in pasta extrusion parameters. By adjusting the dough hydration level, it was possible to obtain a proper dough consistency that would maintain the extrusion parameters within optimum range. However, the effect of changing the dough hydration on the quality of the pasta was not reported.

After hydration, moisture diffuses slowly into semolina particles hydrating storage proteins that are necessary for dough development by transforming the glassy protein polymers into a viscoelastic dough and allowing the formation of a protein network (Icard-Vernière and Feillet 1999). Altering the hydration levels of the dough might cause changes in the development of the gluten matrix with the possibility of affecting the quality of the cooked product. Under-hydration of the pasta dough during extrusion can result in an opaque pasta product with white spots and a tendency to crack. Conversely, excess hydration can result in sticky pasta with low mechanical strength, altered color and reduced cooking quality (Debbouz and Doetkott 1996; Walsh et al 1971).

The objective of the experiment was to determine the effect of spaghetti formulation and dough hydration level on the extrusion and physical properties of the spaghetti and on the change in cooked quality over time. The non-traditional ingredients used to fortify the spaghetti were different ratios of whole wheat flour and/or flaxseed flour. The different formulations were hydrated to 30, 31, 32, 33, and 34% before processing.

Materials and Methods

Experiments were performed with commercial standard semolina (Durakota No.1) from the North Dakota Mill (Grand Forks, ND) and whole hard white wheat flour (Ultragrain) from ConAgra Foods (Omaha, NE). Flaxseed flour was obtained by milling brown flaxseed on a pilot scale hammer mill (Fitzmill, Model DA506, Elmhurst, IL) as described by de la Peña and Manthey (2014b). The ground flaxseed was separated into fine and coarse particles by using a sieve with a 34XXG 600 µm mesh screen.

Nine pasta formulations were evaluated: Semolina 100% (S), whole wheat flour 100% (WW), semolina:whole wheat (SWW) 49:51, semolina:fine flaxseed 90:10 (SFF), whole wheat:fine flaxseed 90:10 (WWFF), semolina:whole wheat:fine flaxseed 39:51:10 (SWWFF), semolina:coarse flaxseed 90:10 (SCF), whole wheat:coarse flaxseed 90:10 (WWCF), and semolina:whole wheat:coarse flaxseed 39:51:10 (SWWCF). Maintaining 51% of whole wheat flour in the formulations was necessary to be classified as a whole wheat product. Uniform blends were prepared by mixing ingredients for 5 min using a cross-flow blender (Patterson Kelly, East Stroudsburg, PA, USA). The 10% (w/w) level of fortification with flaxseed flour was selected because previous research indicated that at this level, the pasta dough had proper consistency for extrusion and the final product had acceptable quality (Sinha and Manthey 2008).

Pasta extrusion and drying

Semolina and semolina-non-traditional formulations were hydrated to achieve proper dough consistency. The wetted ingredients were mixed at high speed in a Hobart mixer (Hobart Corp., Troy, OH, USA) for 4 min and placed in the mixing chamber of the pasta extruder. The mixtures were extruded under vacuum as spaghetti using a semi-commercial laboratory extruder (DEMACO, Melbourne, FL). Extrusion occurred under the following conditions: extrusion temperature, 45°C; mixing chamber vacuum, 46cm of Hg; an auger extrusion speed, 25 rpm. The extrusion auger had a length to diameter ratio of 8.1:1, a constant root diameter and a uniform pitch the entire length of the auger. After extrusion, pasta was dried in a laboratory dryer using a high temperature drying profile (length 10 h; peak temperature 73°C).

Extrusion pressure (kPa), mechanical energy ($J \cdot s^{-1}$), and extrusion rate ($g \cdot s^{-1}$) were recorded during extrusion of each sample. The mechanical energy required to operate the empty pasta press was subtracted from the mechanical energy required to operate the press under load. Specific mechanical energy ($J \cdot kg^{-1}$) was calculated as the mechanical energy ($J \cdot s^{-1}$) to extrude pasta divided by the amount of spaghetti extruded ($g \cdot s^{-1}$).

Pasta physical properties

Color of dried spaghetti was measured with a colorimeter (model CR310, Minolta Corp., Ramsey, NJ). Color reading was expressed by Hunter values for L-, *a*- and *b*-values. L-values measure black to white (0-100), *a*-values measure redness when positive; and *b*-values measure yellowness when positive. Mechanical strength was determined using the flexure rig and texture analyzer (TA-XT2, Texture Technologies Corp., Scarsdale, NY) by measuring both the vertical force (g) required to break a strand of spaghetti and the flexibility of the spaghetti strand (mm). Dry spaghetti dimensions (length and diameter) were measured using a digital caliper and strand weight was recorded. Measurements were conducted on at least 3 strands of pasta. Apparent density was calculated from the measured dimensions and the mass of spaghetti by considering each strand a perfect cylinder (Mercier et al 2011).

Pasta cooking

Pasta was cooked in boiling water (10 g:300 mL). Cooking time was determined using AACC International Approved Method 66-50.01 (AACC International 2013). Cooking time was estimated by squeezing four or five spaghetti strands of 5 cm long between 2 plexiglass sheets in 30 sec intervals. Cooking time corresponded to the disappearance of white in the central core of the spaghetti. Each test was performed in triplicate.

Pasta was cooked in boiling water (10 g:300 mL). Cooking quality of spaghetti was determined using the AACC International Approved Method 16-50.01 with minor modifications (AACC International 2013). Quality parameters of cooked pasta were monitored at each cooking time determined above and at increasing cooking time intervals of 2 min starting from 2 and up to 18 min. The quality parameters measured were cooking loss, cooked weight, and cooked firmness. Cooking loss was measured by evaporating the cooking water to dryness overnight in a forced-air oven at 110°C. The dried weight of material leached from the spaghetti was divided by the dry weight of spaghetti before cooking. Cooked weight was calculated as the increase in product weight and expressed as a percentage of sample weight before cooking. Cooked firmness was measured using the texture analyzer (TA-XT2, Texture Technologies Corp., Scarsdale, NY) by measuring the work (g·cm) required to shear five cooked strands of spaghetti with the pasta blade probe. The experiments were performed with the following set-up: the pasta blade was a 1 mm x 5 cm flat blade, the contact force was 15 g, the return distance was 5 mm, and the return speed was 10 mm-sec. Five measurements were done per sample.

Statistical analysis

Analysis of variance of the extrusion and physical properties of non-traditional spaghetti was performed considering the experimental layout as a split plot arrangement in which the whole plot factor was the spaghetti formulation (S, SFF, SCF, WW, WWFF, WWCF, SWW, SWWFF, and SWWCF) and the subplot factor was the five dough hydration levels (30, 31, 32, 33, and 34%). Tests were replicated three times on different days.

Analysis of variance of the spaghetti cooked quality of over cooking time was performed considering the experimental layout as a split-split plot arrangement in which the whole plot factor was the spaghetti formulation (S, SFF, SCF, WW, WWFF, WWCF, SWW, SWWFF, and SWWCF), the subplot factor was three dough hydration levels (30, 32, and 34%), and the sub-sub plot was the cooking time (2, 4, 6, 8, 10, 12, 14, 16, and 18 min). Tests were replicated three times with each replicate done on different days.

All data were subjected to analysis of variance using the ‘Mixed’ procedure in SAS System for Windows (v 9.3, SAS Institute, Cary, NC). Treatment means were separated by Fisher’s protected Least Significant Difference test calculated at $P=0.05$ using SAS 9.3 statistical package. Pearson correlation coefficient was also estimated between the pasta extrusion parameters, dry pasta physical parameters, and cooked pasta quality parameters ($P \leq 0.05$).

Results and Discussion

Extrusion

Formulation by hydration level interaction was significant for extrusion pressure, mechanical energy, and specific mechanical energy ($P \leq 0.01$). Spaghetti formulation and hydration level main effects were significant for extrusion rate ($P \leq 0.01$).

The fastest extrusion rates were recorded for S, WW, and SWW, followed very closely by their correspondent formulation with coarse flaxseed (Table 9). The incorporation of fine flaxseed caused a big drop in the extrusion rate compared to the formulations with coarse flaxseed or no flaxseed at all. Extrusion rate decreased as the hydration level of the dough increased.

Table 9. Effect of formulation and hydration level on the extrusion rate of spaghetti.

Formulation[a]	Extrusion rate
	g/s
S	3.52 ab
SFF	2.85 d
SCF	3.30 abc
WW	3.51 ab
WWFF	3.01 cd
WWCF	3.46 ab
SWW	3.59 a
SWWFF	3.27 bc
SWWCF	3.55 ab
Hydration (%)[b]	
30	3.65 a
31	3.52 a
32	3.41 b
33	3.06 c
34	2.81 d

Values in the within the column of the same main effect followed by different letters are statistically different for $P \leq 0.05$.

[a] Values averaged across dough hydration levels.

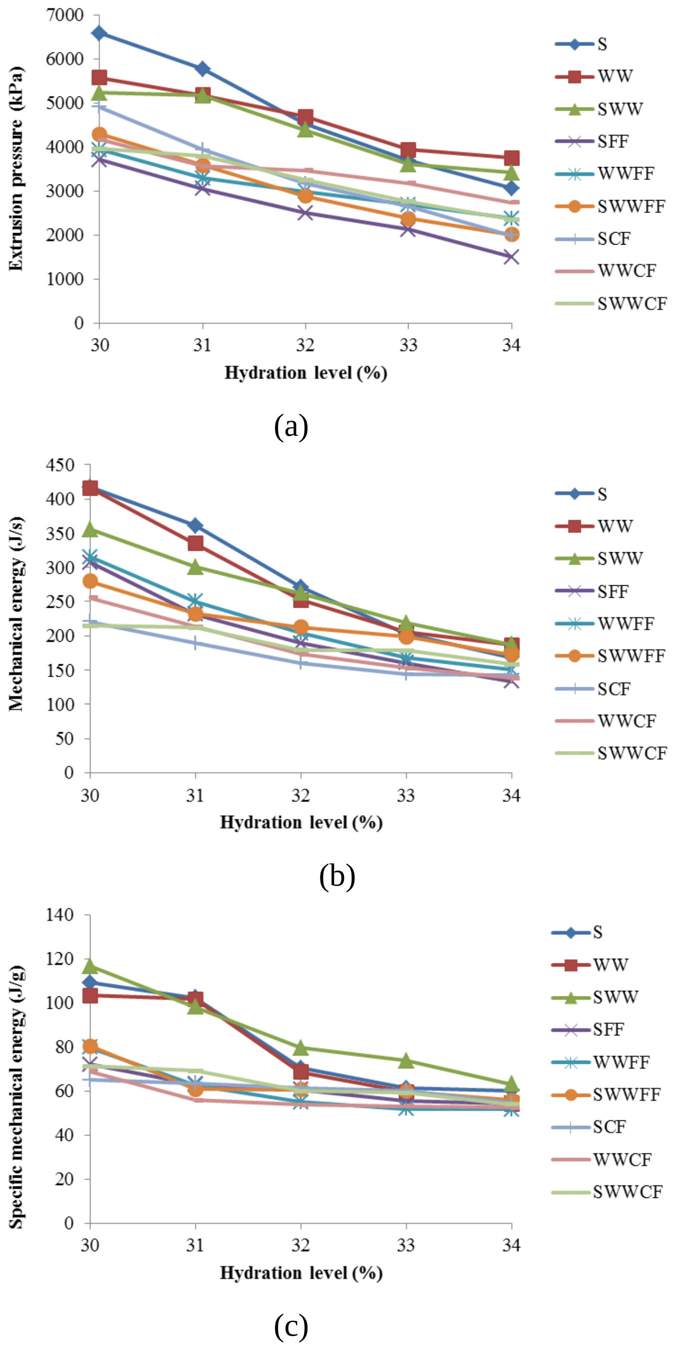
[b] Values averaged across spaghetti formulation.

S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour-Fine Flaxseed (39:51:10), SWWCF: Semolina-Whole Wheat Flour-Coarse Flaxseed (39:51:10).

Presence of flaxseed flour in either the fine or coarse form had a very dramatic effect in the decrease of extrusion pressure in comparison to the correspondent formulation with no flaxseed (Fig. 19-a). Extrusion pressure declined as hydration level increased. The greatest drop in extrusion pressure attributed to the dough hydration level was recorded for S, SFF and SCF. Extrusion of these samples at 34% moisture content required 59.6% less pressure for SFF and SCF and 53% for S than when the formulation were hydrated at 30%. The smallest decrease of extrusion pressure due to the hydration level was reported for WW which required a 32.6% less pressure to extrude when hydrated at 34% than at 30 %.

Mechanical energy required for extrusion was greater with formulations that did not contain flaxseed flour compared to those that did (Fig. 19-b). Mechanical energy during extrusion decreased as hydration level increased. Differences among formulations in the mechanical energy recorded during extrusion were much bigger with 30%, ranging within 220.7 and 417.3 J/s for SFF and S formulation, respectively, than with 34% hydration, ranging within 133.56 J/s and 186.3 for SFF and WW formulation, respectively. The biggest decrease in mechanical energy values recorded when comparing 30% and 34% hydration level was observed for S and SCF with 59.6% and 56.6% drop, respectively.

Specific mechanical energy during extrusion was greater for formulations that did not contain flaxseed flour compared to those that did (Fig. 19-c). Specific mechanical energy during extrusion decreased as hydration level increased. As was observed for extrusion pressure and mechanical energy, differences among formulations in the specific mechanical energy required for extrusion was much smaller with 34%, ranging within 63.2 and 51.8 J/g, for SWW and WWFF, respectively, than with 30% hydration, ranging within 117.0 and 65.1 J/g for SWW and SCF, respectively. The biggest drop of specific mechanical energy when the dough hydration increased from 30 to 34% took was for S, WW, and SWW, with a 44.8, 46.2 and 47.6% of specific mechanical energy drop, respectively.



(a)

(b)

(c)

Figure 19. Effect dough hydration level on the extrusion pressure (a), mechanical energy (b), and specific mechanical energy (c) of non-traditional spaghetti.

LSD (a)=84.79, LSD (b)=32.75, LSD (c)=13.32.
S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour-Fine Flaxseed (39:51:10), SWWCF: Semolina-Whole Wheat Flour-Coarse Flaxseed (39:51:10).

The results observed were in complete agreement with those reported in Chapter 3 where increased dough hydration and presence of flaxseed flour also decreased the values of the spaghetti extrusion parameters. The effect of hydration level and spaghetti formulation on the extrusion parameters also agreed with those results reported by Yalla and Manthey (2006). Reduced extrusion pressure, mechanical energy, and specific mechanical energy of formulations with flaxseed flour and with increased hydration level are attributed to: (1) the disruption of the gluten matrix by flaxseed flour particles that weakened the dough and (2) to the plasticizing effect of water and lipid, as discussed in Chapter 3. These results confirm the importance of adjusting the hydration level of the spaghetti dough in order to have a balanced and stable extrusion process.

Spaghetti physical properties

Formulation by hydration level interaction was significant for both the Hunter *a*-value and *b*-value color parameters ($P \leq 0.01$) (Table A8). Formulation main effect and hydration main effect were significant for L-value (Table A7).

Presence of whole wheat and flaxseed flour decreased lightness (L-value) and yellowness (*b*-value) and increased redness (*a*-value). This was attributed to the darker color of non-traditional ingredients in comparison to semolina. Similar results had been noted by Chillo et al (2008) when fortifying spaghetti with bran and buckwheat flour. The L-value was more affected by the incorporation of whole wheat flour than by flaxseed flour. No statistical differences were found for the L-values when incorporating fine or coarse flaxseed flour within the same type of spaghetti formulation.

Within the same formulation, the *a*-value tended to increase as the hydration level of the spaghetti dough was increased but this effect was not statistically different for S, SFF, and SCF.

This indicated that wetting the flour blend to a higher hydration level before processing enhanced more the redness of flaxseed flour or whole wheat flour than the redness semolina. No differences were detected when comparing the *a*-value of a given formulation with fine or coarse flaxseed when hydrated at the same level.

Hydration level did not affect *b*-value within the same formulation. This effect was different to what was observed by Sinha and Manthey (2008) who reporter higher *b*-values as the hydration level of fresh spaghetti was increased. In our case, pasta was subjected to a drying treatment which might explain the difference in the results. From the point of view of spaghetti formulation, higher *b*-values were found for the S formulation as it was expected, and it decreased as the percentage of non-traditional ingredients increased in the formulation. The incorporation of fine or coarse flaxseed did not affect the *b*-values when looking within the same type of spaghetti formulation e.g. SFF vs SCF.

These color parameters, especially the *b*-value, are very important when considering the acceptability of traditional spaghetti; however, when dealing with non-traditional ingredients, which rarely have a yellow color, they are expected to change. Customers generally desire a bright, uniformly yellow appearance to pasta products. Figure 20 displays the visual appearance of some of the studied formulations when hydrated at different hydration levels. All formulations hydrated at 30% displayed a dull whitish uneven color whereas those samples hydrated at 32% or more had a more even and brighter color. The most intense and even color was observed for samples hydrated to 34%.

Figure 20. Visual appearance of the spaghetti formulations 100% semolina (a), 100% whole wheat flour (b), whole wheat flour-fine flaxseed flour (90:10) (c), and whole wheat flour-coarse flaxseed flour (90:10) (d) when hydrated at 30, 31, 32, 33, and 34%.

Formulation main effect was significant for mechanical strength and hydration main effect was significant for mechanical strength, diameter, and density of dry spaghetti (Table 10). Formulation by hydration level interaction was significant for flexibility of dry spaghetti (Fig. 21).

The mechanical strength of dry spaghetti was the greatest with 100% semolina, although it was not statistically different from WW, and SWW (Table 10). Mechanical strength refers to the amount of force needed to break a strand of spaghetti. Inclusion of flaxseed flour decreased the mechanical strength of all formulations in comparison to the correspondent formulation with no flaxseed, indicating that presence of flaxseed flour weakened the spaghetti strands. This decrease in mechanical strength when non-traditional ingredients are present has been previously documented by Manthey and Schorno (2002) and is attributed to the disruption of the gluten matrix by the particles of non-traditional ingredients.

Flexibility was greatest for spaghetti made with 100% semolina, followed by both SWW and WW (Fig. 21). Flexibility was reduced more by coarse flaxseed flour than by fine flaxseed; contrary to what it was observe for the mechanical strength. This is attributed to the particle size of coarse flaxseed in comparison to the fine flour. Coarse flaxseed flour caused a greater disruption of the gluten matrix and made the spaghetti strand less flexible and prone to rupture than did fine flaxseed flour.

Density and diameter of the dry spaghetti did not differ with formulation but they decreased as the hydration level of the spaghetti increased (Table 10). The decrease in diameter was probably a result of the pressure/energy required to extrude the formulations. During pasta extrusion the dough goes through a teflon-coated die that gives pasta its final shape i.e. round openings form spaghetti. The teflon material that coats the pasta dies is made out of a plastic polymer of polytetrafluoroethylene that is solid at room temperature. Formulations hydrated to 30% are much tougher to extrude than formulations hydrated to 34% as it was reflected in the pasta extrusion parameters reported in Figure 16. It is possible that when formulations hydrated to 30% go through the teflon die, the teflon material is slightly compressed against the die plate wall and that resultant spaghetti strand has a slightly wider diameter. Conversely, formulations hydrated to 34% are much softer and flow much easily through the die, therefore they would not expand the teflon material during extrusion and the resultant strand would have a smaller diameter than formulations hydrated to 30%. Significant positive correlations of 0.42, 0.36, 0.44, and 0.31 ($P<0.01$) were found between the diameter and the extrusion pressure, extrusion rate, mechanical energy, and specific the mechanical energy parameters, respectively.

Formulations hydrated to 30% have more solids and less water than samples hydrated to 34%; therefore, it makes sense that samples hydrated to 30% formed denser strands than samples hydrated to 34%. During the pasta dehydration process water evaporates leaving more empty tiny pores filled with air (Mercier et al 2011). It is possible that those spaghetti samples with more initial moisture content (higher hydration level) leave more tiny pores behind than those samples that had less moisture content (lower hydration level). A positive correlation of $r=0.43$ ($P\leq 0.01$) was found between spaghetti diameter and spaghetti density. No correlation between density and pasta extrusion parameters was found.

Mechanical strength decreased as the hydration level increased, although the differences were only statistically different between 30-31% and 34% of level of hydration. As mentioned above, at 34% hydration spaghetti strands were less dense and had smaller diameters. Having thinner spaghetti and less dense strands would explain why samples hydrated to 34% need less mechanical energy to break and were more flexible than a sample hydrated to 30%; however, no correlations between mechanical energy and density and strand diameter were found.

Within the same formulation the flexibility of the spaghetti tended to increase as the hydration level increased (Fig. 21). The latter could also be attributed to the effect that hydration had on the diameter or density of the strands. A less dense strand would be less rigid and subsequently more flexible when subjected to bending. A significant correlation of *r*=-0.34 (*P*≤ 0.05) was found between spaghetti flexibility and density indicating that flexibility decreases as density increases. No significant correlation was found between flexibility and diameter.

Table 10. Effect of the main effects spaghetti formulation and hydration level on the mechanical strength, diameter, and density of dry spaghetti.

Formulation[a]	Mechanical strength	Diameter	Density
	g	mm	g/cm^3
S	29.1 a	1.483 a	0.722 a
SFF	21.5 c	1.477 a	0.747 a
SCF	22.6 bc	1.492 a	0.734 a
WW	28.7 a	1.455 a	0.726 a
WWFF	22.2 bc	1.471 a	0.758 a
WWCF	23.7 b	1.458 a	0.736 a
SWW	28.0 a	1.467 a	0.721 a
SWWFF	21.8 bc	1.484 a	0.753 a
SWWCF	23.5 bc	1.469 a	0.735 a
Hydration (%)[b]			
30	25.4 a	1.487 a	0.746 a
31	25.3 a	1.484 ab	0.741 a
32	24.9 ab	1.481 b	0.735 b
33	24.1 ab	1.466 c	0.732 b
34	23.2 b	1.451 d	0.730 b

Values within same main effect in the same column followed by different letters are statistically different for *P*≤0.05.

[a] Values averaged across dough hydration levels.

[b] Values averaged across spaghetti formulation.

S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW:Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour-Fine Flaxseed (39:51:10), SWWCF: Semolina-Whole Wheat Flour-Coarse Flaxseed (39:51:10).

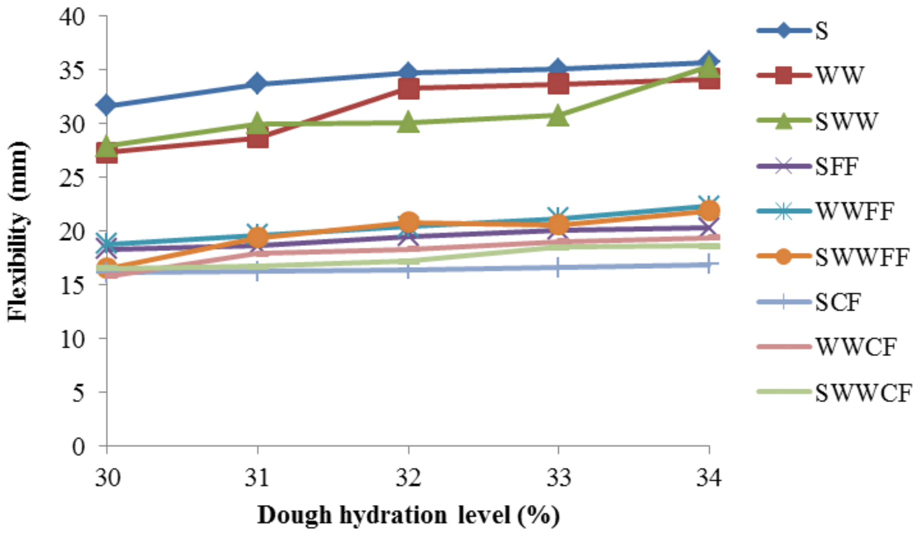
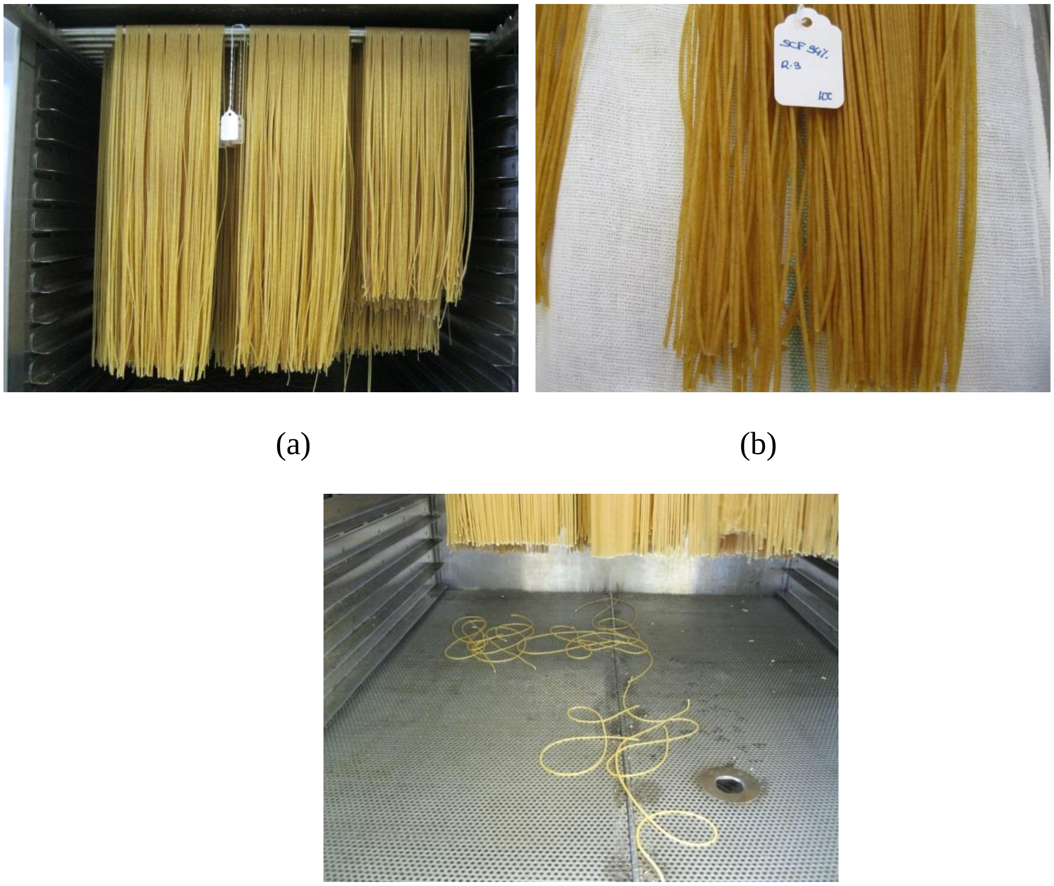


Figure 21. Effect of the spaghetti formulation-hydration level interaction on the flexibility of dry spaghetti.

LSD=2.478.
S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour-Fine Flaxseed (39:51:10), SWWCF: Semolina-Whole Wheat Flour-Coarse Flaxseed (39:51:10).

When some of the non-traditional formulations studied in this experiment were hydrated to 33-34%, and especially those with flaxseed flour the strands stuck to each other during the hanging process on the rod (Fig. 22-a). Some tangled among each other during the drying process resulting in curved strands that would not be acceptable by the consumer and that would cause problems during packaging (Fig. 22-b). Conversely, some samples that were hydrated to 30% were too dry and when hanging on the rod, caused the rupture of the strands and fell to the bottom of the dryer (Fig 22-c). This indicated the importance of finding an optimum hydration point for each formulation for which the color and the physical strength of the strand are acceptable.



(a) (b)

(c)

Figure 22. Visual appearance of pasta hanging from a drying rod after drying (a) and of dry strands entangled with each other (b) and after being hydrated at 34%. Broken spaghetti during drying when dough hydration was 30% (c).

Pasta cooking - optimum cooking time

Formulation by hydration level interaction was significant for cooked weight. Spaghetti formulation main effect and hydration level main effect were significant for cooking time and cooked firmness of non-traditional spaghetti (*P*≤0.01); however, only the spaghetti formulation was significant for cooking loss (*P*≤0.01). The interaction between spaghetti formulation and dough hydration was significant for cooked weight (*P*≤0.05). The cooked quality results of non-traditional spaghetti cooked to optimum can be found in Table A11 and Figure A2 in Appedix.

Cooking time was the longest for S, WW, and SWW, intermediate for samples containing fine flaxseed, and shortest for samples containing coarse flaxseed flour (Table 11). Shorter cooking times for samples with coarse flaxseed flour were attributed to a larger disruption of the protein matrix due the larger particle size found in the coarse flaxseed flour compared to the fine flaxseed flour (Chillo et al 2008; Manthey et al 2004). Short cooking times were also recorded for high hydration levels. This can be attributed to smaller diameter of the spaghetti, which decreased as the hydration level increased as mentioned in the section above and in Table 10. In agreement with this theory, a significant correlation of r=0.35 ($P\leq0.01$) was found between cooking time a spaghetti diameter. Having a smaller diameter would require less time for the water to penetrate within the strand and reduce the cooking time of spaghetti.

Table 11. Effect of spaghetti formulation and dough hydration level on the cooking time, cooking loss and firmness of non-traditional spaghetti.

Formulation[a]	Cooking time
	sec
S	563 a
SFF	521 b
SCF	491 cd
WW	553 a
WWFF	498 cd
WWCF	469 e
SWW	547 a
SWWFF	505 bc
SWWCF	482 de
Hydration (%)[b]	
30	535 a
31	519 b
32	509 c
33	492 d
34	491 e

Values in the same column within the same main effect followed by different letters are statistically different for $P \leq 0.05$.

[a] Values averaged across dough hydration levels.

[b] Values averaged across spaghetti formulation

S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW:Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour-Fine Flaxseed (39:51:10), SWWCF: Semolina-Whole Wheat Flour-Coarse Flaxseed (39:51:10).

Change in cooked quality with cooking time

The interaction between the main effects for the different spaghetti quality cooking parameters can be observed in Table 12.

Table 12. Statistical significance of the main effects spaghetti formulation, hydration level, and cooking time and their interactions on the cooked firmness, cooking loss, and cooked weight of spaghetti.

Effect	Firmness	Residue	Cooked weight
	g·cm	%	g/g dw
Formulation (F)	**	**	**
Hydration (H)	**	**	**
FxH	**	*	*
Cooking time (CT)	**	**	**
CT	**	**	**
FxCT	**	**	**
HxCT	**	**	ns
FxHxCT	**	ns	ns

* Significant at *P*≤0.05.

** Significant at *P*≤0.01.

The interaction of the spaghetti formulation, dough hydration, and cooking time for cooked firmness can be observed in Figure 23. The graphs showed that cooked firmness was different for the each of the formulations, especially during the initial minutes of cooking. The different firmness of each spaghetti formulation relates to the ability of the ingredients to interact with each other to form a compact and continuous protein matrix. As cooking time progressed, the firmness decreased because of the effects of the protein denaturation and starch gelatinization. Cooked firmness decreased the most during the first 4 min of cooking, and after that it decreased at a much slower rate. Samples hydrated at 30% before extrusion tended to show higher cooked firmness than the samples hydrated at 32 or 34%, but this effect was more pronounced in some formulations (e.g. S and WW) than in others (e.g. WWFF). The effect of hydration was more obvious during the first 4 to 8 min of cooking. Overall the lowest firmness was detected when a formulation had fine flaxseed flour, the hydration level of the dough before extrusion had been 34%, and the spaghetti had been cooked for 18 min.

When averaged across dough hydration and cooking time, greatest cooked firmness was recorded for S, closely followed by SWW, SFF and SCF. Formulations with coarse flaxseed ended to have much higher cooked firmness during the first 2 min of cooking than did the other formulations. After 2 min of cooking, this difference in cooked firmness disappeared.

Overall, presence of flaxseed flour in a fine or coarse form and whole wheat tended to decrease the cooked firmness of the spaghetti (data not presented). Better firmness was obtained when some semolina was present in the formulation than when samples had 90 or 100% whole wheat flour. Decreased firmness in non-traditional pasta has been previously reported and has been related to decreased continuity of the protein matrix (Manthey et al 2004). Cooked firmness decreased as the hydration level of the ingredients used to make spaghetti increased, which is probably related to the small diameter and low density of spaghetti made with high hydration levels. A significant correlation of $r=0.32$ ($P\leq0.01$) was found between cooked firmness and spaghetti diameter but no correlation was found between cooked firmness and spaghetti density.

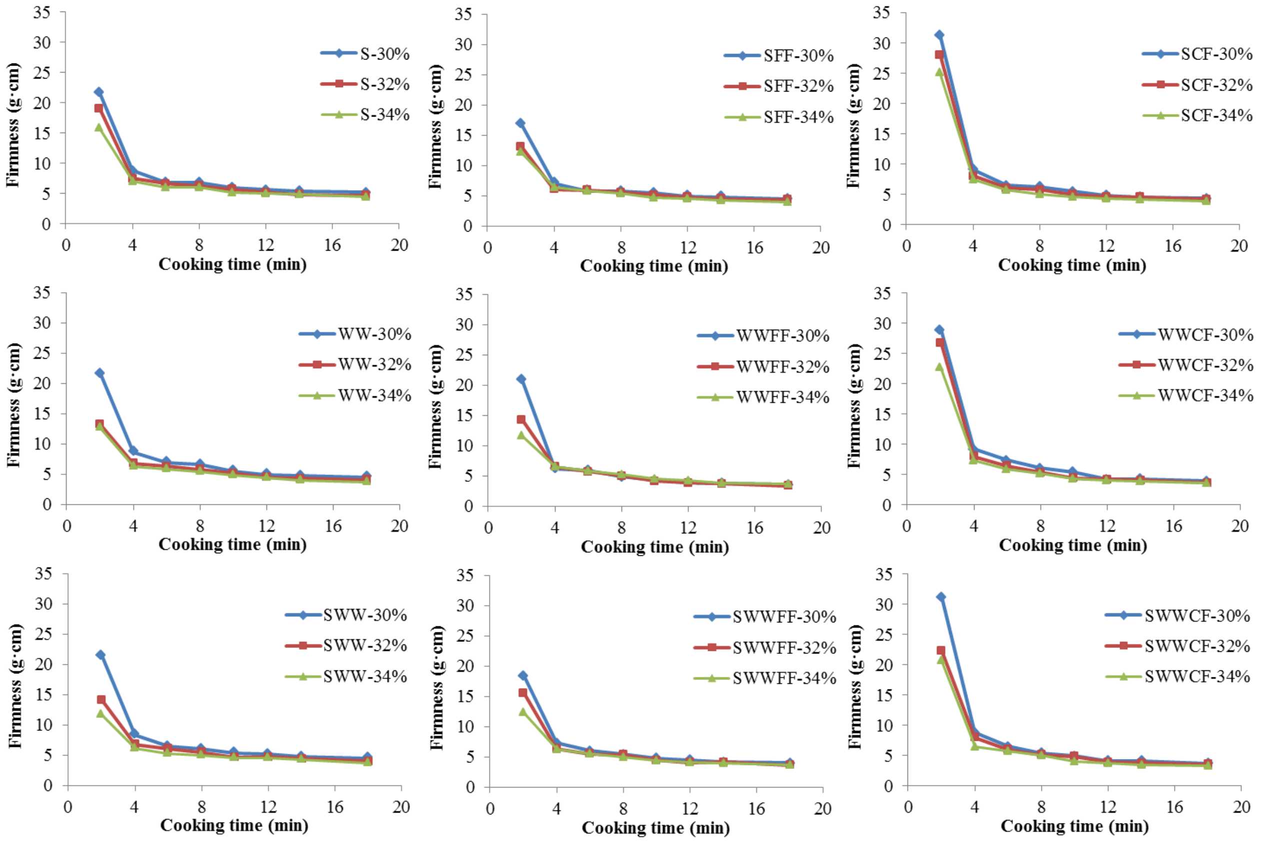


Figure 23. Effect of formulation, hydration level, and cooking time on the cooked firmness of spaghetti.

LSD 1 ($P \leq 0.05$)=13.3 Between two cooking times at the same formulation-hydration combination.
LSD 2 ($P \leq 0.05$)=13.2 Between hydration levels at the same formulation and same or different cooking time combination
LSD 3 ($P \leq 0.05$)=13.3 Between two formulations at the same or different hydration-cooking time combination.
S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour-Fine Flaxseed (39:51:10), SWWCF: Semolina-Whole Wheat Flour-Coarse Flaxseed (39:51:10).

The interaction between cooking time and dough hydration levels on cooking loss is displayed in Figure 24. Cooking loss increased as both the cooking time and dough hydration level increased. Cooking losses increased with cooking time because as water penetrates within the spaghetti amylose and soluble proteins leach into the cooking water (Sobota and Zarycki 2013). Differences in the cooking loss could be related to the density of the spaghetti. As mentioned above, spaghetti made from ingredients that had been hydrated at 30% before extrusion were denser than spaghetti made from ingredients hydrated at 32 or 34%. Having denser strands could have slowed water migration into the strand and reduced the leaching of amylose from the starch granules embedded in the protein matrix; however, no correlation was found between spaghetti density and cooking loss.

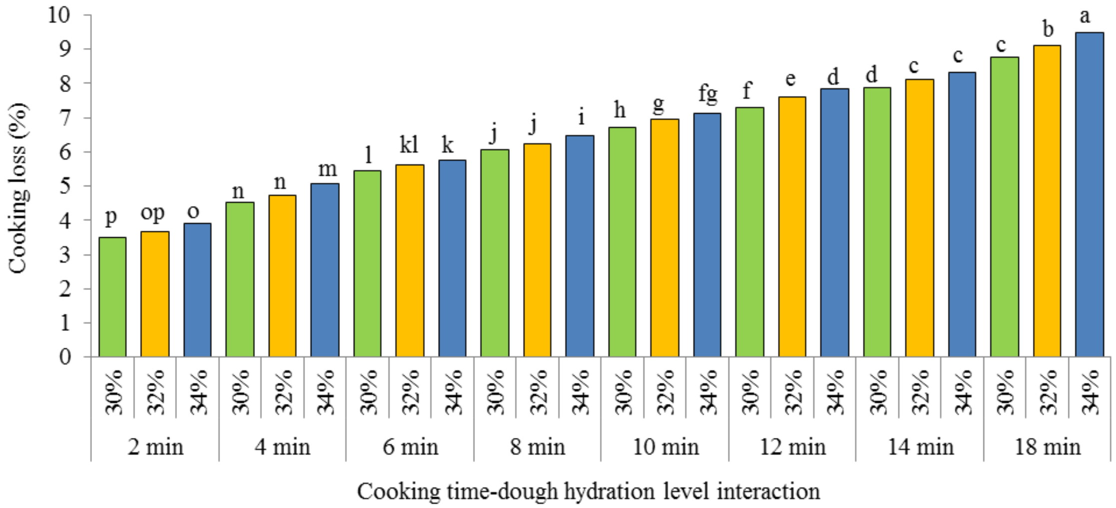
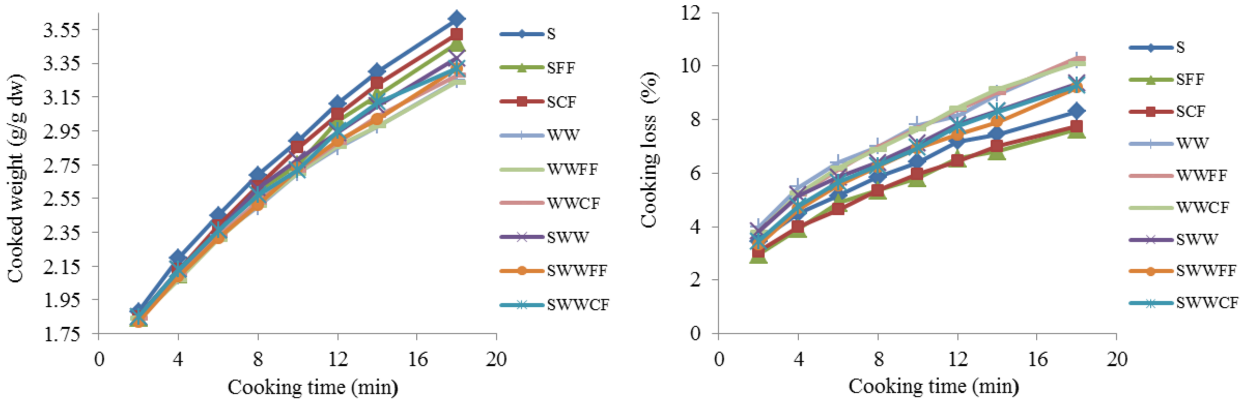


Figure 24. Effect of cooking time and dough hydration level on the cooking loss of spaghetti. Column bars followed by different letters are statistically different for $P \leq 0.05$.

The interaction between spaghetti formulation and cooking time on spaghetti cooked weight and cooking loss can be observed in Figure 25-a and -b, respectively. Both cooked weight and cooking losses increased with cooking time; however, the degree to which they increased depended upon the formulation of the spaghetti. Cooking losses were the greatest for samples with 100% whole wheat flour in the formulation, intermediate for samples with 51% of whole wheat flour, and the least for samples without whole wheat flour. Greater cooking losses in spaghetti containing whole wheat flour were attributed to the higher levels of starch damage in the flour in comparison to the semolina (Hatcher et al 2002). Having more starch damage increases the access of cooking water to the interior of the starch granule, which would increase the amount of amylose available to leach into the cooking water. Presence of flaxseed flour in the spaghetti formulation decreased the cooking losses of the spaghetti, effect that was expected since flaxseed lacks of starch in its composition as shown in Table 2 in Chapter 2.

Cooked weight was greatest for the formulation S and it was least when both whole wheat flour and flaxseed flour were in the same formulation (Fig. 25-b). The lower cooked weight showed by non-traditional pasta is thought to be related to the disruption of the protein matrix by the non-traditional ingredients which decreases the ability of the protein matrix to hold water.

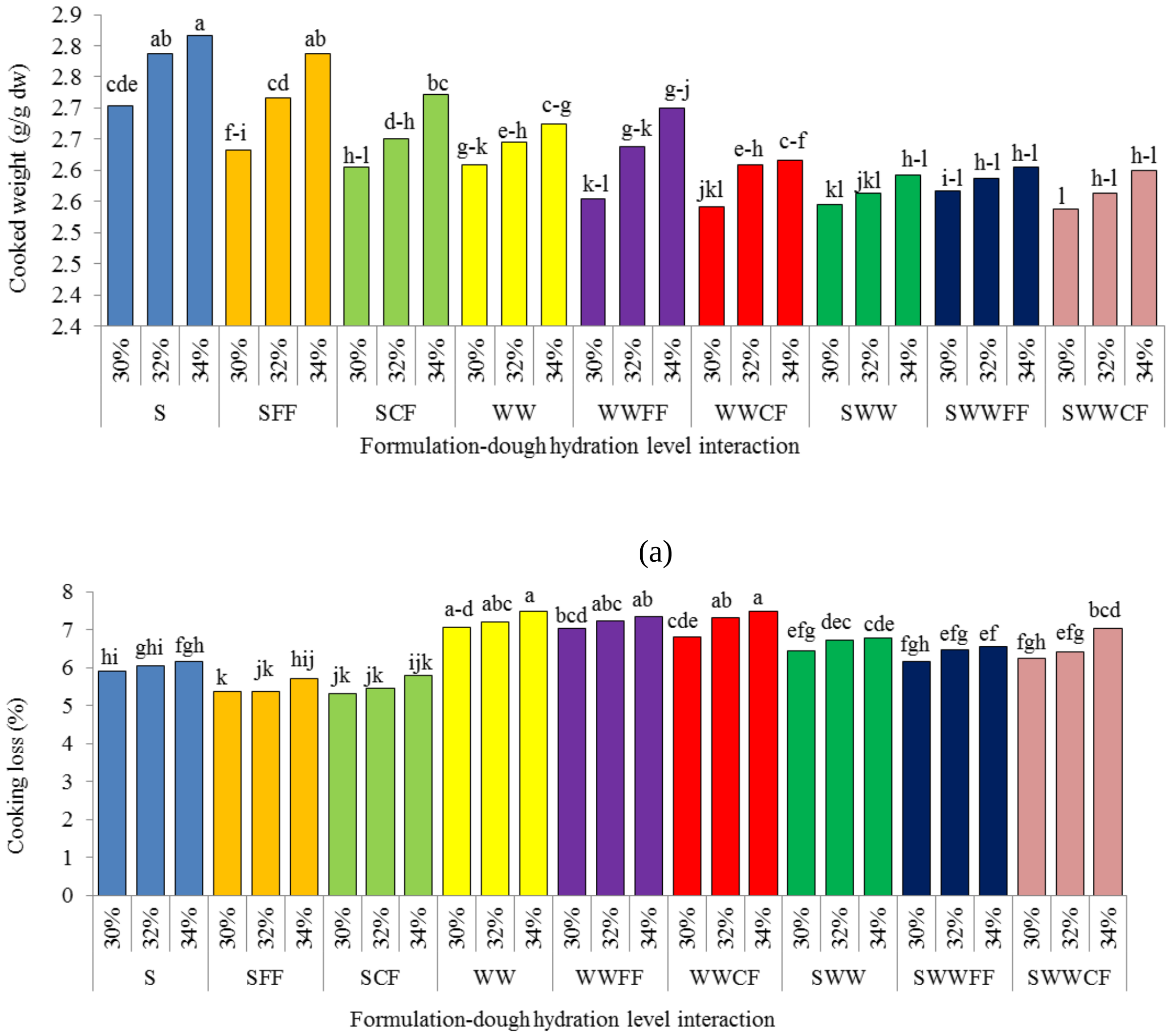


(a) (b)

Figure 25. Effect of cooking time and spaghetti formulation on the cooked weight (a) and cooking loss of spaghetti.

LSD (a) (*P*≤0.05)=0.058 Between two different formulation-cooking time combinations.
LSD (b) (*P*≤0.05)=0.510 Between two different formulation-cooking time combinations.
S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour-Fine Flaxseed (39:51:10), SWWCF: Semolina-Whole Wheat Flour-Coarse Flaxseed (39:51:10).

The interactions between formulation and dough hydration on cooking loss and cooked weight support the results discussed in Figure 25. The result of this interaction can be observed in Figure 26-a for cooked weight and in Figure 26-b for cooking loss. Cooked weight increased as dough hydration level was raised from 30 to 32 or 34%; however for cooking loss, this did not occur for all formulations. The increase of cooked weight as the hydration levels increased can be attributed to the development of the spaghetti dough. Increasing the hydration level from 30 to 34% increased the hydration of proteins, which in pasta dough are always under-hydrated (Matsuo et al 1978), helping to further develop the gluten matrix and increase water retention. The degree to which both parameters increased depended on the formulation of the spaghetti, as explained above.



(a)

(b)

Figure 26. Effect of formulation and dough hydration on the cooked weight (a) cooking loss (b) of spaghetti.

Column bars followed by different letters are statistically different for $P \leq 0.05$.
S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour-Fine Flaxseed (39:51:10), SWWCF: Semolina-Whole Wheat Flour-Coarse Flaxseed (39:51:10).

Conclusions

Spaghetti formulation and dough hydration affected the extrusion, physical properties, and change in cooked quality over cooking time of non-traditional spaghetti. The effect of the spaghetti formulation was attributed to the different physical and chemical properties of the ingredients present in the formulations. Semolina (100%) had the highest cooked quality in comparison to the rest of the formulations. Better cooked quality was obtained for SWW than for WW, indicating that it was better to have some semolina in the formulation. Inclusion of flaxseed flour had an overall detrimental effect on the cooked quality, but better results were obtained when adding the flaxseed flour to 90% semolina (SFF and SCF), than when adding it to WW or SWW. Only small differences in cooked quality were noted when using coarse or fine flaxseed flour.

The effect of the hydration level on the extrusion properties was attributed to the plasticizing effect it had on the dough. In contrast, the effect of dough hydration on the physical properties and cooked quality was more attributed to the effect it might have on the diameter and density of the spaghetti strand than to any chemical change it might have provoked on the spaghetti. Spaghetti extruded better, gave the brightest color, and cooked fastest at a hydration level of 34% than at 30%; however, an optimum hydration level should be determined to for each formulation to optimize extrusion properties and physical appearance of the strands.

Cooked firmness decreased, and cooking loss, and cooked weight increased as cooking time progressed. The biggest changes in the cooked quality of the spaghetti were obtained during the first 4 min of cooking. Little changes in cooked firmness were obtained after cooking the spaghetti 8 min beyond its optimum cooking time indicating the tolerance of the spaghetti to overcooking.

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PAPER 5. CHEMICAL CHANGES IN NON-TRADITIONAL PASTA DURING COOKING

Abstract

This study was conducted to determine the changes of biochemical components in non-traditional spaghetti that take place during cooking, and to determine how these changes were reflected in the quality of the cooked product. Semolina 100%, whole wheat flour 100%, and the following mixtures of semolina-whole wheat flour (49:51), semolina-flaxseed flour (90:10), whole wheat flour-flaxseed flour (90:10), and semolina-whole wheat flour-flaxseed flour (39:51:10) were the formulations used for the experiments. Chemical analysis included total starch, starch damage, pasting properties and protein quality and quantity of the flour mixes and spaghetti cooked for 0, 2, 4, 10, and 18 min. As cooking time progressed, total starch content decreased, starch damage increased, pasting parameters decreased, and protein solubility decreased in all six formulations. The differences observed in the magnitude of these chemical changes among the non-traditional formulations were attributed to a dilution of the starch and protein levels in comparison to the 100% semolina spaghetti. The changes in the starch damage level, total starch content, and pasting properties of spaghetti agreed with the cooking loss values recorded for the spaghetti. Low levels of albumin and globulins and high levels of the low molecular weight glutenin were related to high spaghetti firmness. High levels of the high molecular weight glutenin, high levels of the albumin and globulin fractions, and low levels of the low molecular weight glutenin were associated with high cooking losses and cooked weight, indicating the involvement of these proteins in the cooked quality of the spaghetti.

Introduction

Pasta fortification with non-traditional ingredients has been thoroughly studied over the last decades with the purpose of offering healthier alternatives to consumers. The variety of ingredients that can be utilized as fortification material is very broad; however, the trend is to use ingredients that provide additional nutritional benefit to its consumption (Marconi and Carcea 2001). Whole wheat flour and flaxseed flour are two excellent fortification ingredients because of their well-documented benefits to human health (Cardoso-Carraro et al 2012; Liu 2007). Both ingredients occur in multigrain pasta products sold in grocery stores.

Semolina contains up to 80% starch, 2-3% of non-starch polysaccharide (Lintas 1988), 1-2% of lipid (Youngs 1988) and 12-16% of protein (Feillet 1988). Semolina proteins are comprised of about 20% metabolic proteins (enzymes) and about 80% storage proteins. Storage proteins are composed of two classes of proteins, gliadins, a prolamin, and glutenin, a glutelin type of protein according to the Osborne classification (Manthey and Twombly 2006). Both wheat gliadins and glutenins interact with each other when hydrated and mixed to form gluten, the matrix provides the mechanical strength to pasta products.

Whole wheat flour and flaxseed flour have different chemical composition from semolina. Whole wheat flour and semolina differ in composition due to the presence of the bran and germ in the whole wheat flour. The proteins in germ and bran are mainly composed of albumins and globulins, which might result in dilution of gluten proteins and starch content of the flour and also adds new chemical components that are not present in the semolina such as non-starch polysaccharides, minerals, phenolic acid antioxidants, lipids and vitamins (Hirawan et al 2010). The protein composition of whole wheat flour is similar to that of semolina and therefore, the gluten forming capability does not change. Flaxseed on the other hand does not have gluten forming proteins, making it unable to form a gluten matrix. Flaxseed can contain from 10 to 37% protein, depending on the genetics and environmental conditions under which the seed grows (Oomah and Mazza 1993). The main protein fractions present in flaxseed are globulins and albumins, which can account for 56-73.4 and 20-42 % of the total flaxseed proteins, respectively (Udenigwe and Aluko 2011). Flaxseed contains a lipid content of about 40% of the total weight of the seed. It does not contain starch but it does contain 8% of mucilage gum located in the seed coat (Rubilar et al 2010). The water binding and hydro-colloidal properties associated with the flaxseed mucilage make it very susceptible to interacting with carbohydrates or protein molecules from other sources when in the presence of moisture (Chen et al 2006).

Because of the chemical differences among flaxseed, whole wheat flour and semolina, when combined in a pasta formulation they can alter the cooking properties of the fresh or dried pasta products (Manthey and Schorno 2002; Sinha and Manthey 2008). During cooking, boiling water penetrates progressively inside the pasta matrix causing the gelatinization of the starch and protein polymerization and denaturation. The extent to which starch gelatinization and protein denaturation happen are going to determine the cooked pasta quality and its acceptance by the consumer (Bruneel et al 2010). Since whole wheat flour and flaxseed flour differ in starch and protein content and composition from semolina, the protein polymerization process (Lamacchia et al 2011) and the gelatinization of starch (West et al 2013) could be altered. The evolution of starch gelatinization and protein changes as cooking progresses has been previously documented for traditional pasta products (Dexter and Matsuo 1979; Grzybowski and Donnelly 1979; Sözer and Kaya 2003); however, little or no research has been done in the observation of these changes on non-traditional pasta products.

The objective was to document the changes in the biochemical components of non-traditional spaghetti that take place during cooking, and to determine how these changes were reflected in the quality of the cooked product. The non-traditional ingredients used to fortify the spaghetti were different ratios of whole wheat flour and/or flaxseed flour.

Materials and Methods

Experiments were performed with commercial standard semolina (Durakota No.1) from North Dakota Mill (Grand Forks, ND) and whole hard white winter wheat flour (Ultragrain) from ConAgra Foods (Omaha, NE). Commercial golden flaxseed flour was obtained from Heartland flax (Valley City, ND) that was separated into fine and coarse particles by using a sieve with a 600µm 34XXG mesh screen. Fine particle fraction contained particles <600µm.

A total number of six different pasta formulations were used for this experiment: Semolina 100% (S), whole wheat flour 100% (WW), and a blend of semolina:whole wheat (SWW) 49:51, semolina:fine flaxseed 90:10 (SFF), whole wheat:fine flaxseed 90:10 (WWFF), and semolina:whole wheat:fine flaxseed 39:51:10 (SWWFF). Maintaining 51% of whole wheat flour in the formulations was necessary to be classified as a whole wheat product. Uniform blends were prepared by mixing ingredients for 5 min using a cross-flow blender (Patterson Kelly, East Stroudsburg, PA, USA). The 10% (w/w) level of fortification with flaxseed flour was selected because previous research indicated that at this level, the pasta dough had proper consistency for extrusion and the final product had acceptable quality (Sinha and Manthey 2008).

Proximate analysis

AACC International Approved Methods 08-12.01, 44-15.02, 76-13.01, 76-31.01 and 46-30.01 were used to determine ash content, moisture content, starch content, starch damage, and protein content, respectively, of the six ingredient formulations and on freeze-dried ground spaghetti samples cooked for 0, 2, 4, 10, and 18 min (AACC International 2013). The conversion factor used to determine protein content was %N x 5.7 for semolina and whole wheat flour and %N x 5.41 for flaxseed flour (Tkachuk 1969). Lipid content was determined using a 16 h Soxhlet extraction with hexane according to Method Ba 3-38 (AOCS 1998).

Pasta processing

Semolina and semolina-non-traditional ingredient blends were hydrated to 30, 32, and 34% absorption. The wetted ingredients were mixed at high speed in a Hobart mixer (Hobart Corp., Troy, OH, USA) for 4 min and placed in the mixing chamber of the pasta extruder. The mixtures were extruded under vacuum as spaghetti using a semi commercial laboratory extruder (DEMACO, Melbourne, FL). Extrusion occurred under the following conditions: extrusion temperature, 45°C; mixing chamber vacuum, 46cm of Hg; an auger extrusion speed, 25 rpm. The extrusion auger had a length to diameter ratio of 8.1:1, a constant root diameter and a uniform pitch the entire length of the auger. Pasta was dried in a laboratory dryer using a high temperature drying cycle (duration 10 h; peak temperature 73°C).

Pasta cooking

Pasta cooking quality was determined using the AACC International Approved Method 16-50.01 with minor modifications (AACC International 2013). Pasta was cooked in boiling distilled water for 2, 4, 6, 8, 10, 12, 14, 16, 18, and 20 min. Pasta:water ratio was 10 g:300 mL. Quality parameters of cooked pasta were monitored at each cooking time by determining the cooked weight, cooked firmness, and cooking loss. Cooking loss was measured by evaporating the cooking water to dryness overnight in a forced-air oven at 110°C. The dried weight of material leached from the spaghetti was divided by the dry weight of spaghetti before cooking.

Cooked weight was calculated as the increase in product weight and expressed as a percentage of sample weight before cooking. Cooked firmness was measured using the texture analyzer (TA-XT2, Texture Technologies Corp., Scarsdale, NY) by measuring the work (g·cm) required to shear five cooked strands of spaghetti. The experiments were performed with the following set-up: the pasta blade was a 1 mm x 5 cm flat blade, the contact force was 15 g, the return distance was 5 mm, and the return speed was 10 mm-sec. Five measurements were done per sample.

Immediately after the cooking tests, the samples were shaken gently to remove excess water and the cooked spaghetti strands were placed in a perforated plastic bag and stored in the freezer, until they were freeze-dried and ground into a fine powder with UDY cyclone mill (UDY Corporation, Fort Collins, CO).

Pasting properties

Pasting profiles of freeze-dried ground samples cooked at 0, 2, 4, 10, and 18 min were determined using a Rapid Visco Analyzer RVA (model 4SA; Newport Scientific 4, Jessup, MD), according to AACC International Approved Method 76-21.01 (AACC International 2013). The equipment was interfaced with a computer equipped with Thermocline and Thermoview software (Newport Scientific, Warriewood NSW, Australia). Ground sample (3 g, 14% mb) was added to pre-weighed deionized distilled water in an RVA canister. The rate of heating and cooling was 12°C/min, idle temperature was 95°C. Total run time for each sample was 13 min. Parameters recorded were peak viscosity (PV), hot paste viscosity (HPV), breakdown (BKD), cold paste (CPV) and setback (STB) viscosity. All measurements will be reported in Rapid Visco Units (RVU).

Protein extraction

Extraction of proteins from freeze-dried ground spaghetti samples cooked at 0, 2, 4, 10, and 18 min was done according to the method described by Gupta et al (1993) with minor modifications (Ohm et al 2009). Flour or freeze-dried ground spaghetti material (10 mg, db) was suspended in 1 mL of 0.5% SDS and 0.05 M sodium phosphate buffer (pH 6.9) and stirred for 5 min at 2,000 rpm using a pulsing vortex mixer (Fisher Scientific). No defatting was done for flaxseed flour or for samples containing flaxseed flour. The mixture was centrifuged for 15 min at 17,000 x *g* (Centrifuge 5424, Eppendorf). The soluble protein dissolved in supernatant was filtered through a membrane filter (0.45 µm PVDF Membrane, Sun Sri, Rockwood, TN). Immediately after filtering, the sample was heated for 2 min at 80 °C (Larroque et al 2000). The insoluble protein was obtained from the residue. The residues were sonicated for 30 sec at the power setting of 10 Watt output (Sonic Dismembranor 100, Fisher Scientific) with 1 mL of extraction buffer. Then, the mixture was filtered and heated before SE-HPLC analysis as described above for soluble proteins.

Size-exclusion high performance liquid chromatography (SE-HPLC)

Analysis of protein extract of was done by SE-HPLC (Batey et al 1991) using Aligent 1100 Series (Aligent Technologies, Waldbroann, Germany) HPLC fitted with a guard cartridge (Phenomenex BIOSEP SEC S4000, Phenomenex, Torrance, CA) and a narrow bore column (300 x 4.6 mm, Phenomenex, Torrance, CA) (Ohm et al 2009). Filtered supernatant (10 µL) was injected and eluted by 50% acetonitrile in water with 0.1% trifluoroacetic acid for 10 min with a flow rate of 0.5 mL/min. Solutes were detected at 214 nm using Agilent 1200 Photodiode Array Detector (Agilent Technologies, Waldbroann, Germany).

The absorbance area and area % values were calculated for 6 main fractions of SE-HPLC that included: F1 (3.5-4.5 min retention time), F2 (4.5-5.7 min), F3 (5.7-6.1 min), F4 (6.1-6.8 min), F5 (6.8-7.5 min), and F6 (7.5-8.1 min) (Sandhu et al 2011). Primary components constituting individual fractions were high molecular weight polymeric proteins for F1; low molecular weight polymeric proteins for F2; ω-gliadin for F3; γ-, β-, and α-gliadins for F4; and albumin and globulins for F5 and F6 (Larroque et al 1997).

Scanning electron microscopy

Scanning electron microscopy was conducted by the Electron Microscopy Center located at North Dakota State University, Fargo, ND. Air-dried spaghetti strands cooked at 2, 4, 10, and 18 min were selected from the set of samples hydrated to 32% and examined and photographed at 50, 500, and 1000 X magnifications using a JEOL JSM-6490LV scanning electron microscope (JEOL USA, Peabody, Massachusetts) at an accelerating voltage of 15 kV. Samples were mounted on aluminum mounts with silver paint and were then coated with gold-palladium using a Balzars SCD 030 sputter coater (Model SCD 030, Liechtenstein) to make them electrically conductive.

Statistical analysis

All formulations were analyzed for the total starch content, starch damage, and pasting properties. Analysis of variance for those variables was performed considering experimental layout as a split plot arrangement in which the whole plot factor was the spaghetti formulation (S, SFF, WW, WWFF, SWW, and SWWFF), and the subplot factor was five cooking times (0, 2, 4, 10, and 18 min). The experimental design for whole plot was a completely randomized design and all the experiments were performed in triplicate. Data were subjected to analysis of variance using the 'Mixed' procedure in SAS System for Windows (v 9.3, SAS Institute, Cary, NC).

Treatment means were separated by Fisher’s protected Least Significant Difference test calculated at $P=0.05$ using SAS 9.3 statistical package. Pearson correlation coefficient was also estimated between the pasta quality cooking parameters and the total starch content, starch damage, and pasting parameters ($P \leq 0.05$).

Absorbance data from SE-HPLC chromatograms of protein extracts were transformed and analyzed using an in-house program that was developed using MATLAB (version 6, The MathWorks, Natick, MA). Absorbance data were interpolated to 0.002 min intervals by spline methods and used to calculate absorbance area. The absorbance area was calculated by mean absorbance×time interval (0.002 min). The sum of absorbance area for each retention time interval of 0.01 min between 3.5 and 8.1 min of run time are used for data analysis. The percentage of absorbance area (A%) for each 0.01 min time interval was obtained by calculating the % of absorbance area of each time interval over sum of all the AAs between 3.5 and 8.1 min of run time. The A% could represent absorbance area adjusted to the same level of protein content. Simple linear correlation coefficients (*r*) were calculated between both absorbance area and A% values and quality parameters for each 0.01 min retention interval, and shown as a continuous spectrum over retention time (correllogram).

Results and Discussion

Proximate analysis

Proximate analysis of the raw ingredients and the different formulations studied in this experiment is presented in Table 13. The results were very similar to published values in the literature (Manthey and Twombly 2006; Rubilar et al 2010). Lipid, protein, and ash contents were higher in all formulations that contained flaxseed flour in comparison to the correspondent formulation with no flaxseed. This effect was expected since flaxseed flour contains more lipid, ash, and protein than does semolina or whole wheat flour as shown in Table 13. Starch damage of flour formulations was 3.8% for S and SFF, 5.6% for WW and WWFF, 4.4% for SWW, and 4.8% for SWWFF. The little or no effect of flaxseed flour on starch damage was expected since flaxseed lacks starch. Greater starch damage values in WW than in S were also anticipated since the WW flour is subjected to a greater particle reduction process than semolina.

Table 13. Proximate composition of ingredient formulations and flaxseed flour.

Formulations	Lipid	Protein	Ash	Starch
	% db			
FF	47.49 ± 0.22	22.13 ± 0.02	4.19 ± 0.02	0
S	5.27 ± 0.16	14.05 ± 0.06	0.82 ± 0.08	72.00 ± 0.86
WW	7.04 ± 0.33	13.76 ± 0.04	1.87 ± 0.02	64.46 ± 0.09
SWW	6.20 ± 0.18	13.83 ± 0.03	1.44 ± 0.03	69.26 ± 0.48
SFF	9.33 ± 0.10	15.00 ± 0.09	1.29 ± 0.05	67.51 ± 1.11
WWFF	11.55 ± 0.28	15.42 ± 0.17	2.20 ± 0.03	57.84 ± 0.96
SWWFF	9.18 ± 0.23	15.12 ± 0.13	1.84 ± 0.01	62.38 ± 0.07

S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour-Fine Flaxseed (39:51:10), FF: Flaxseed Flour 100%.

Starch damage and total starch

The cooking time main effect was significant for the starch damage of the non-traditional spaghetti ($P \leq 0.01$) (Table 14). The largest increase in starch damage occurred within the first 4 min of cooking in which an average of 8.1% of the starch damage level increased with respect to the uncooked spaghetti. An average additional increase of 2.9 and 0.7% was recorded when cooking for 10 and 18 min, respectively. Starch damage increased as cooking time increased, which reflects the progression of the starch gelatinization within the spaghetti strand. Starch damage continued to increase progressively throughout the entire period of time that measurements were taken. It was especially surprising to find that after 12 min of cooking time, which was 3-4 min past the optimum cooking time depending on the formulation (Table 11 Chapter 4), there was still some nongelatinized starch in the strands.

For a proper interpretation of the starch damage results showed in Table 14 it is important to understand the basis of the method utilized to determine the starch damage. The AACC International method 76-31.01 for the determination of starch damage is a fixed-time assay in which excess of the enzyme amyloglucosidase is allowed to degrade the starch-derived dextrins to glucose for 20 min. At 20 min the reaction is stopped, regardless of whether all dextrins have been degraded to glucose or not. This means that the results given by the test are relative to how much starch was degraded during the 20 min of reaction incubation, and not to the complete degradation of damaged starch present in the sample. For samples cooked for 18 min, the starch damage level indicated by the test was between 19-21% depending on the formulation of the spaghetti; however, the real absolute starch damage value was probably close to 100% because after cooking for so long it is very unlikely that there was any nongelatinized starch in the sample, as shown by Colonna et al (1990). Nevertheless, as long as this concept is understood, and all the samples are analyzed under the same conditions, this method provides reliable relative results of the levels of starch damage present in the samples.

Table 14. Change in the starch damage and total starch of non-traditional spaghetti as cooking time increased from 0 min to 18 min.

Formulation[a]	Total starch	Starch damage
	g/g d.w.	%
S	69.3 a	18.2 a
SFF	64.5 b	17.8 a
WW	60.6 d	16.6 a
WWFF	54.6 f	16.1 a
SWW	65.8 b	17.2 a
SWWFF	59.0 e	15.8 a
Cooking time (min)[b]		
0	65.5 a	9.6 d
2	63.1 b	15.4 c
4	62.2 c	17.7 b
10	61.0 d	20.6 a
18	59.8 e	21.3 a

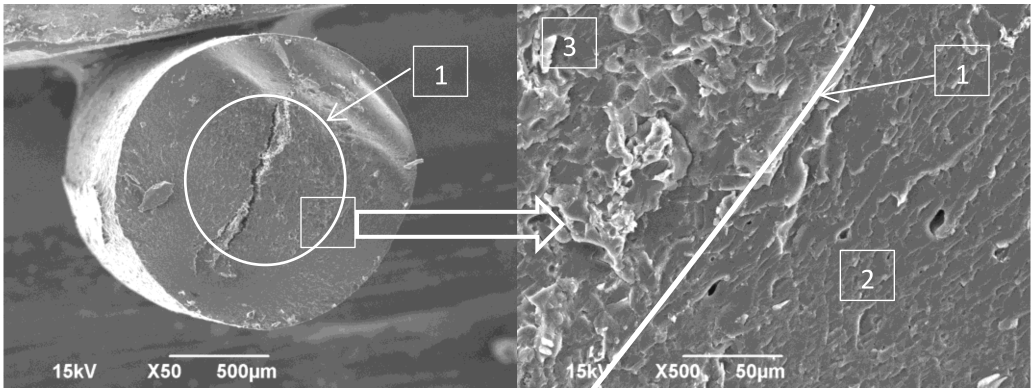
Values in the same column followed by different letters are statistically different for $P \leq 0.05$.

[a] Values averages cross cooking time.

[b] Values averaged across spaghetti formulation.

S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour-Fine Flaxseed (39:51:10).

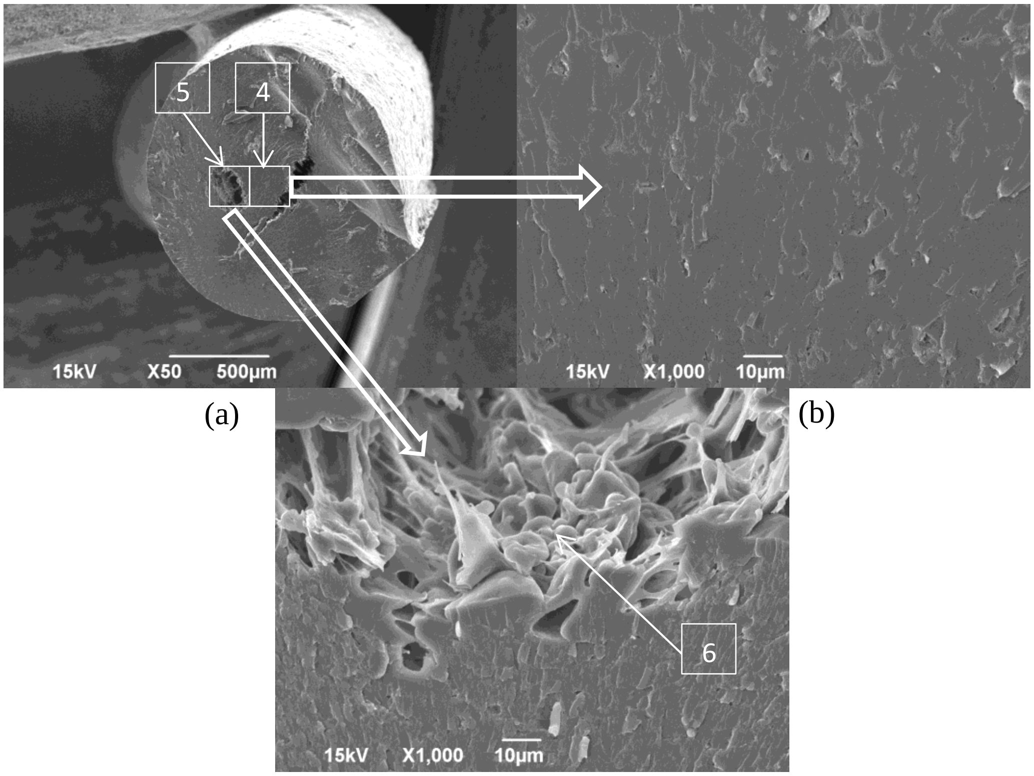
The change in starch damage or starch gelatinization at different cooking times can be noted in the SEM micrographs. Figure 27 corresponds to the cross section of a strand of 100% semolina spaghetti cooked for 4 min. In that micrograph it is possible to delineate the area of water penetration (cooked area), from the area in which water did not penetrate during the cooking period (uncooked area) (Fig. 27-a1 and -b1). The area corresponding to the cooked section displayed a smooth surface (Fig. 27-b2) due to the gelatinization of the starch granules and the denaturation of the protein matrix present in that section. Conversely, the area corresponding to the uncooked section (Fig. 27-b3) displayed a much rougher surface attributed to the presence native protein and intact starch granules (Dexter et al 1978) because the water had not penetrated that far inside the strand.



(a) (b)

Figure 27. Scanning electron microscope picture of an air-dried 100% semolina spaghetti strand cooked for 4 min. Cross section of the spaghetti strand (a), and amplification of fragment of the cross section (b). (1) Line that defines the extent of water penetration, (2) cooked section of the spaghetti strand, (3) uncooked section of the spaghetti strand.

Figure 28 represents the SEM micrograph of the cross section of a strand of 100% semolina spaghetti cooked for 12 min. The smooth surface displayed by amplified fragment of the center of the strand (Fig. 28-a4) reveals that the water had fully penetrated to the core of the strand (Fig. 28-b). This also agreed with the cooking time of 9.5 min determined for this particular sample in Chapter 4, which means that at 12 min of cooking, all the core of the strand should be fully cooked and the starch fully gelatinized. However, when magnifying the crack observed in the surface of the cross section (Fig. 28-a5), it was possible to observe that even though the strand seemed to be fully cooked, there was still small “pockets” in the strand in which there appeared to be nongelatinized starch granules (Fig. 28-c6). These “pockets” were observed in all six formulations studied in this experiment. The shape of those starch granules indicated that probably they underwent annealing or heat-moisture treatment due to the high temperature-low moisture combination generated inside the strand during cooking. The AACC International method 66-50.01 establishes that the optimum cooking time of pasta has been reached when the white central core of the spaghetti cannot be seen anymore after pressing the spaghetti strands between two plexiglass sheets (AACC International 2013). At that point it is assumed that all starch in the sample has been gelatinized. However, the presence of these “pockets” indicates that the assumption that all starch granules have been fully gelatinized when the white central core of the spaghetti strand disappears is wrong. The reason why those “pockets” did not fully cook is unknown. A theory that should be further investigated is the possibility that those “pockets” appeared due to the presence material that was not properly hydrated during the mixing stage. The presence of these starch granules would explain why there was an increase in the levels of starch damage when comparing the cooking times 10 min and 18 min. The fact that the strand cross section showed a crack in the very same place where the starch granules were located is not considered a coincidence. It is thought that during the air drying process of the cooked strand, the presence of those nongelatinized “pockets” created a tension in the generally cooked interior of the strand, which caused the strand to crack at that very same point.

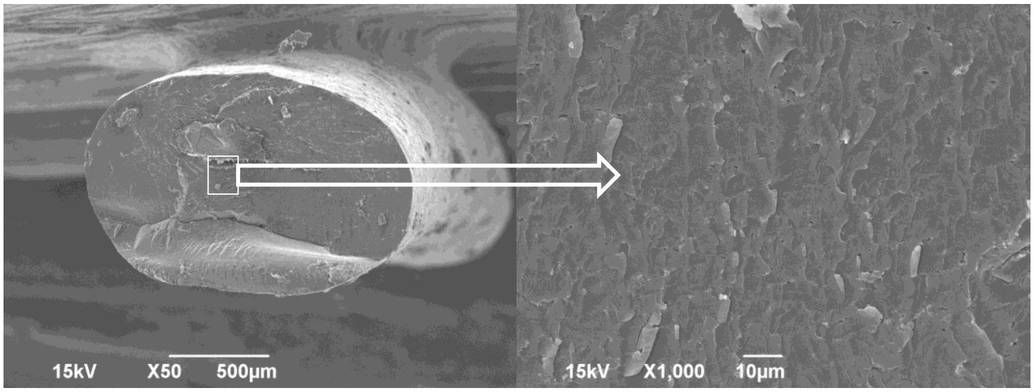


(a) (b)

(c)

Figure 28. Scanning electron micrograph of an air-dried 100% semolina spaghetti strand cooked for 12 min. Cross section of the spaghetti strand (a), amplification of fragment of the cross section (b), amplification of another fragment of the cross section. (4) Cooked core of the spaghetti strand, (5) "pockets" found in inside the spaghetti strand, (6) starch granules located inside the "pocket".

The appearance of the cross section of a strand of 100% semolina spaghetti cooked for 18 min can be observed in Figure 29. No "pocket" was identified at this point in the exposed cross sectional area (Fig. 29-a), and the core of the strand was fully gelatinized (Fig. 29-b). This observation was noted for all six formulations. Delcour et al (2000) reported that annealing increases the gelatinization temperature of the starch granules. This discovery could be applied to this situation, indicating that by prolonging the cooking time of the pasta, the temperature reached inside the spaghetti strand was high enough to fulfill the gelatinization process of the annealed starch granules. All the micrographs presented in Figures 27, 28, and 29 were representative of what was observed for the other spaghetti formulations studied in this experiment.



(a) (b)

Figure 29. Scanning electron microscope picture of an air-dried 100% semolina spaghetti strand cooked for 18 min. Cross section of the spaghetti strand (a), and amplification of fragment of the cross section (b).

The main effect of spaghetti formulation was not significant ($P \leq 0.05$) for the starch damage of the non-traditional spaghetti samples (Table 14), indicating that the progression of starch damage levels during cooking was independent of the initial starch damage of the uncooked non-traditional spaghetti. Starch damage was greater in uncooked dry spaghetti than in the corresponding blended flours (data not presented). This difference in that starch damage values between flour blends and dry uncooked spaghetti is attributed to the mechanical damage occurring during mixing and extrusion and to amylolitic enzyme activity during processing, especially during the drying cycle (Lintas and D’Appolonia 1979).

The spaghetti formulation by cooking time interaction was not significant ($P \leq 0.01$) but the main effects of spaghetti formulation and cooking time were significant ($P \leq 0.01$) for the total starch content of the non-traditional spaghetti samples cooked at different cooking times (Table 14). Total starch content of spaghetti was reduced as cooking time progressed due to its solubilization into the cooking water. When the starch granule loses its crystalline arrangement due to gelatinization, amylose leaches from the starch granule into the cooking water. In agreement with the starch damage test results, the largest decline in total starch level occurred within the first 4 min of cooking in which a 5.04% of the starch was lost indicating that this is the critical period of time when most of the starch loss takes place (Colonna et al 1990). Cooking for 6 more min, 10 min of total cooking time, and 14 more min, 18 min of total cooking time, only decreased the total starch content for an additional 1.83% and 3.66%, respectively. The constant loss of starch content throughout the 18 min cooking time agreed with the results observed for the starch damage and the SEM micrographs. The presence of nongelatinized starch granules after 10 min of cooking would explain the reason for a further decrease of starch content when comparing the starch levels at 10 and 18 min of cooking time.

Losses in total starch content were dependent on the initial starch content present in dry spaghetti. Figure 30 shows that all samples lost starch in a similar proportion for a given cooking time interval, indicating that the presence of non-traditional ingredients did not alter the rate at which the starch was lost during cooking.

The starch that leached from the spaghetti during cooking formed part of what is known as cooking losses, a parameter widely measure to determine the pasta cooking quality as described in Chapter 4. A significant correlation coefficient (r=-0.76, $P\leq0.01$, n=30) was found between the total starch content and the cooking loss values of the spaghetti samples cooked at different cooking times (Chapter 4) indicating a direct relationship between the decrease of the total starch content of the sample and the increase in the cooking loss values. Another significant correlation (r=0.88) between starch damage and cooking loss (Chapter 4) was also found ($P\leq0.01$) indicating that the greater the starch damage inside the spaghetti strand the greater the cooking losses recovered in the cooking water. These correlations reinforce the sequence of events ascribed to the modification of starch during cooking: (1) Starch damage levels increase due to the gelatinization of starch during cooking, (2) starch is lost to the cooking water when the starch granule loses its crystalline structure due to gelatinization, and (3) the starch that leached into the water is measured as cooking losses and used as a parameter that determines the quality of cooked pasta.

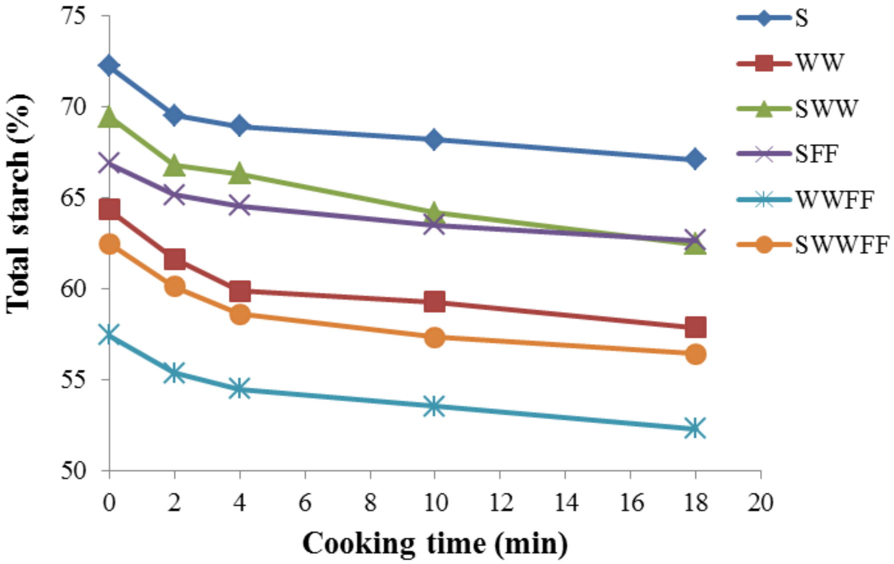


Figure 30. Change in the total starch content of non-traditional spaghetti as cooking time increased from 0 min to 18 min.

S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour-Fine Flaxseed (39:51:10).

Pasting properties

RVA determined the viscous/pasting properties of the non-traditional spaghetti samples cooked at 0, 2, 4, 10, and 18min. Spaghetti formulation by cooking time interaction was significant for each of the pasting parameters measured during the RVA test (*P*≤0.01). Table 15 presents the pasting properties of each of the studied formulations when averaged across the cooking time. Fortification of spaghetti with non-traditional ingredients resulted in a decrease in peak viscosity, breakdown, setback, holding strength, peak temperature, and final viscosity values in comparison with S, the traditional spaghetti formulation. The lower values in the pasting parameters found with spaghetti fortified with whole wheat flour and/or flaxseed flour was attributed to their lower level in starch content in comparison to semolina.

Table 15. RVA data for the different spaghetti formulations averaged across spaghetti cooking time.

Formulation	Peak viscosity	Trough	Breakdown	Setback	Final viscosity	Peak time
	RVU	RVU	RVU	RVU	RVU	min
S	78.9 a	63.3 a	15.6 a	62.5 a	125.8 a	6.01 a
SFF	61.8 b	48.8 b	13.0 b	35.9 d	84.7 d	5.47 c
WW	45.8 e	36.9 d	8.9 c	59.0 a	95.9 c	5.95 ab
WWFF	57.5 cd	42.1 c	15.4 ab	53.0 b	95.1 c	5.26 d
SWW	59.6 bc	49.2 b	10.4 c	62.2 a	111.4 b	5.79 b
SWWFF	55.5 d	41.3 c	14.2 ab	44.1 c	85.4 d	5.29 cd

Values in the same column followed by different letters are statistically different for $P \leq 0.05$.
S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour-Fine Flaxseed (39:51:10).

Decrease in the “Peak viscosity” parameter due to the presence of whole wheat flour and/or flaxseed is directly related to the total starch content of the samples. As reported in Table 13, the whole wheat flour used for this experiment had lower starch content than did the semolina, and flaxseed flour contained no starch. The RVA test was also run for a sample containing 100% flaxseed flour. Although it does not contain any starch it still formed a small viscosity peak (Fig. 31). This peak was attributed to the mucilage present in the flaxseed seed coat, which has the ability of forming a gel under the water-heating conditions of the RVA pasting test (Chen et al 2006). In the presence of starch from semolina or whole wheat flour, the release of both amylose and low molecular weight amylopectin during the gelatinization step promotes the formation of polymer complexes with hydrocolloids that adds viscosity to the system (Bahnassey et al 1994). Because of this gelling property of the flaxseed mucilage, samples containing flaxseed flour did not have a big decrease in the peak viscosity. Actually, in the case of the formulation WWFF, the flaxseed flour seemed to increase the viscosity significantly in comparison to the formulation WW.

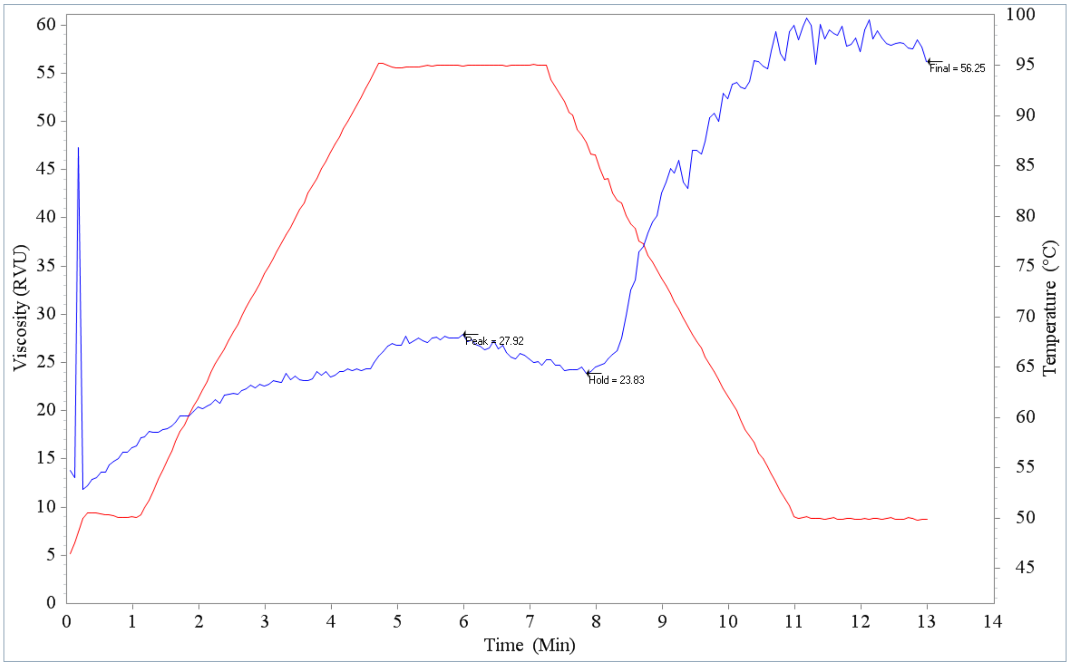


Figure 31. RVA pasting curve for flaxseed flour.

Presence of flaxseed decreased the "Peak time" or time required for the slurry to reach its peak viscosity in comparison to formulations with no flaxseed flour. Flaxseed contains around 40-50% of lipid, of which at least 44-52% is α-linolenic acid (Daun et al 2003). The effect of flaxseed on the "Peak time" parameter might be due to the formation of amylose-lipid complexes that increase the gelatinization temperature of the slurry. Tang and Copeland (2007) reported that the formation of amylose-lipid complexes, including α-linolenic acid, can alter the pasting properties of the starch.

The parameter "Breakdown" represents the difference between the parameters "Peak viscosity" and "Trough" or what is the same, the difference between the maximum and minimum point of samples viscosity recorded during the RVA test. Even though the formulation S was the one that underwent the greatest drop in viscosity, it still was the formulation that showed the highest holding strength or viscosity ("Trough" parameter) after starch granule disruption due to swelling and shearing forces. The overall trend detected was that the lower "Peak viscosity" of a given formulation, the lower was the holding strength of the gel.

The "Setback" parameter is the mathematical difference between the "Final viscosity" values and the "Trough" value for a given formulation. It represents the absolute value of increase in viscosity of a sample due to the retrogradation phenomenon that takes place during the cooling step of the RVA test. On the other hand, "Final viscosity" corresponded to the maximum viscosity value recorded after the drop of viscosity. Both "Setback" and "Final viscosity" are the reflection of the level of retrogradation of the starch; therefore, it makes sense that the parameters "Setback" and "Final viscosity" were highly correlated with each other (r=0.94 $P\leq$0.01). Samples that contained flaxseed had much lower "Setback" values than did samples without flaxseed. This effect was probably attributed to the difficulty of the amylose and amylopectin molecules to form crosslinks with each other due to the presence of flaxseed particles. Overall, samples with flaxseed also had lower "Final viscosity" values than the correspondent sample with flaxseed, with the exception of WW whose "Final viscosity" value was not significantly different from the one recorded for WWFF.

Table 16 contains the results for the pasting properties of samples at each cooking time when averaged across the spaghetti formulations. As expected, the longer the cooking time, the longer it took to reach the peak viscosity, the lower the peak viscosity, the smaller the breakdown and set back, and the lower the final viscosity of the ground spaghetti slurry (Fig. 32). This trend was true for all six spaghetti formulations (data not presented). No statistical differences were found for any RVA parameter when comparing spaghetti cooked for 10 or 18 min, which agrees with the starch damage results discussed above.

Table 16. RVA data for the different spaghetti cooking averaged across spaghetti formulations.

Cooking time	Peak viscosity	Trough	Breakdown	Set back	Final viscosity	Peak time
min	RVU	RVU	RVU	RVU	RVU	min
0	91.9 a	61.2 a	30.8 a	82.9 a	144.0 a	5.3 c
2	68.1 b	51.7 b	16.5 b	63.8b	115.5 b	5.4 c
4	56.2 c	48.0 c	8.2 c	51.7 c	99.6 c	5.5 b
10	40.9 d	36.7 d	4.1 d	34.1 d	70.9 d	6.1 a
18	42.2 d	37.2 d	5.0 d	31.4 d	68.5 d	5.9 a

Values in the same column followed by different letters are statistically different for *P*≤0.05.

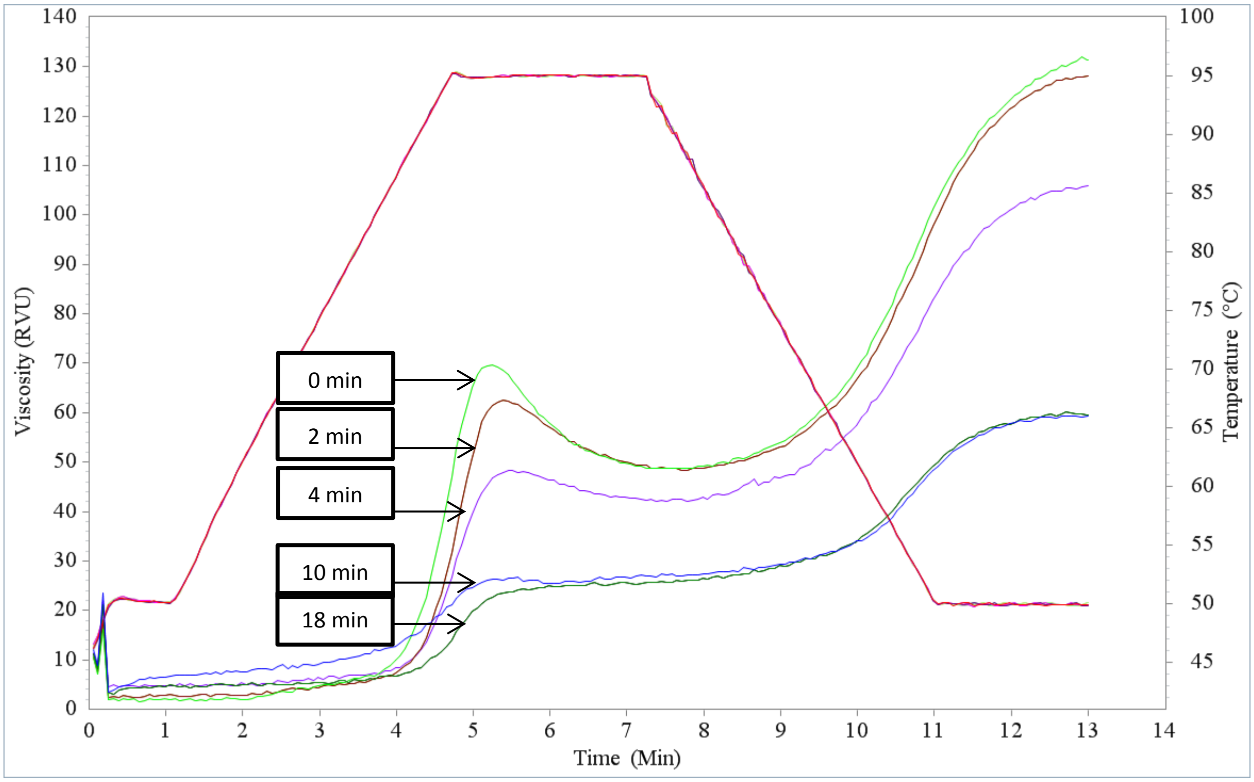


Figure 32. RVA curves for 100% whole wheat flour spaghetti samples cooked at different cooking times.

Multiple significant linear correlations were found between the RVA parameters and the pasta quality cooking parameters from Chapter 4, and total starch and starch damage values

(Table 17). These correlations were run considering the cooking parameter values and the RVA, total starch and starch damaged values of spaghetti cooked for 2, 4, 10 and 18 min. This means that most of the correlations are probably driven by the increasing cooking time since all the changes reported the spaghetti over cooking time were time dependent. When the correlations were observed for the individual cooking times, the number of significant correlations changed considerably (both increased and decreased) as cooking time progressed without following a particular trend and they never matched when compared among each other (data not shown). This response indicates that the pasting properties are a reflection of the overall changes that occurred to the spaghetti as cooking time progressed, but it does not reflect the quality of the spaghetti at a particular cooking time.

Table 17. Pearson correlation between RVA parameters, pasta quality cooking parameters, and total starch and starch damage (*n*=24).

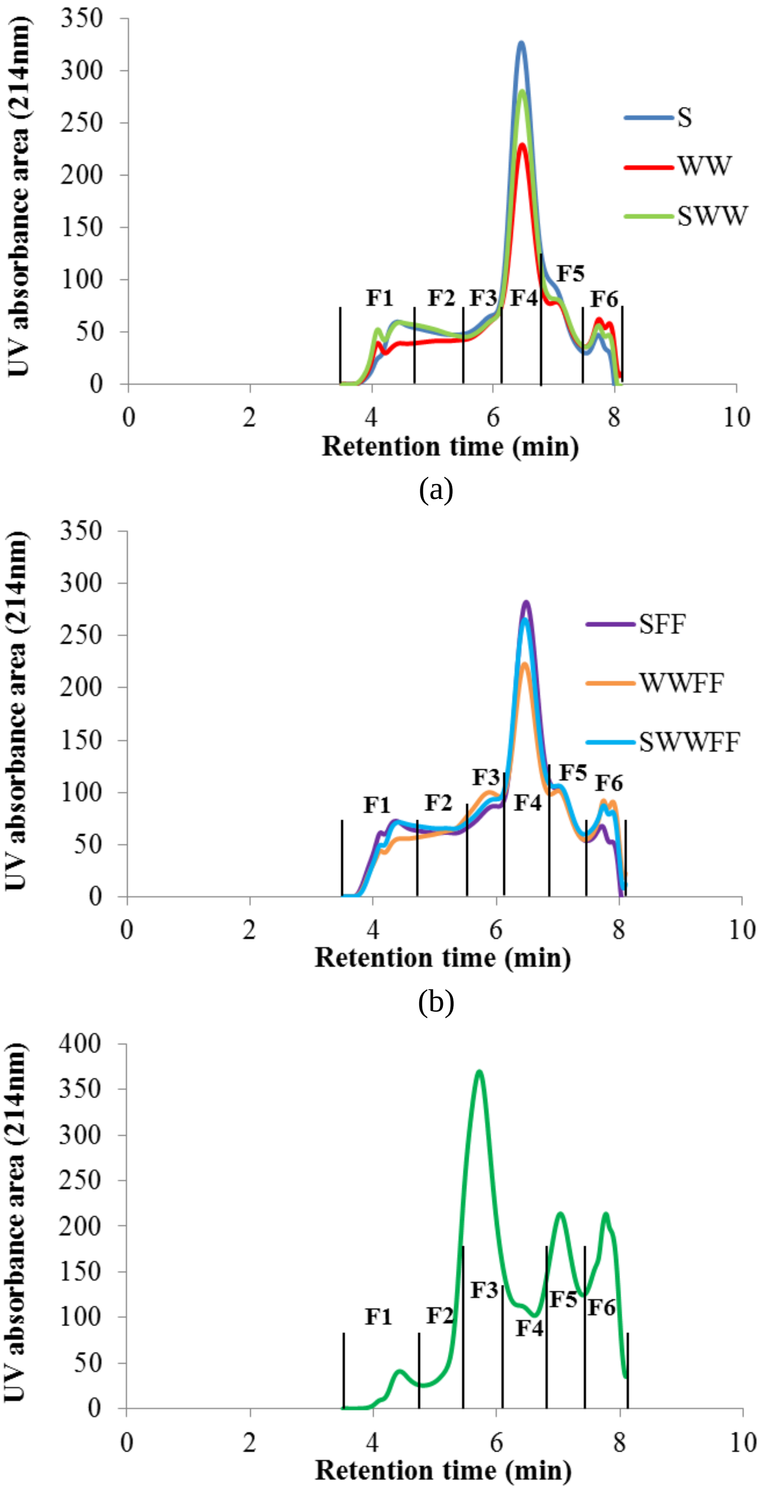
RVA parameters	Cooked spaghetti quality parameters			Starch parameters	
	Cooked firmness	Cooking loss	Cooked weight	Total starch	Starch damage
Peak time	-0.34 ns	0.34 ns	0.43 *	0.35 ns	0.41 *
Peak viscosity	0.80 **	-0.75 **	-0.64 **	0.52 **	-0.55 **
Trough	0.61 **	-0.64 **	-0.46 *	0.75 **	-0.33 ns
Breakdown	0.79 **	-0.61 **	-0.68 **	-0.15 ns	-0.71 **
Final viscosity	0.77 **	-0.70 **	-0.68 **	0.55 **	-0.63 **
Setback	0.76 **	-0.63 **	-0.72 **	0.32 ns	-0.73 **

* Significant at $P \leq 0.05$.
** Significant at $P \leq 0.01$.

Size-exclusion high performance liquid chromatography (SE-HPLC)

SE-HPLC showed the typical profile of protein extracted from traditional and non-traditional spaghetti (Fig. 33). Traditional pasta (S), displayed a higher F4 peak than WW spaghetti, which corresponds to the main gliadin fractions. The combination of semolina with whole wheat flour (SWW) gave an intermediate peak in between S and WW spaghetti (Fig. 33-

a). WW also showed lower F1 and F2 peaks than S and SWW spaghetti. Differences were attributed to the lower protein content present in whole wheat flour than in semolina. Presence of flaxseed flour in the spaghetti increased the heights of peaks F3, F5, and F6, and decreased peak F4 (Fig. 33-b) in comparison to samples with no flaxseed. The curve of protein extract from flaxseed flour is displayed in Figure 33-c. A similar fraction division displayed in Figure 33-a and -b was applied to the flaxseed flour chromatogram in Figure 33-c to compare how presence of flaxseed impacted the protein profile of spaghetti that contained this ingredient. The flaxseed chromatogram revealed high peaks in F3, F5, and F6, and the lack of peak in F4, in comparison to the spaghetti chromatogram. This explains why those samples in which flaxseed was incorporated showed higher peaks in F3, F5, and F6, and lower peak in F4 in comparison to those samples with no flaxseed. Flaxseed major storage proteins include a globulin-like salt soluble 11-12S with a molecular weight that ranges between 252-298 kDa and an albumin-type water soluble basic 1.6-2S protein with a molecular weight of 16-17 kDa (Madhusudhan and Singh 1985a,b). Flaxseed flour was not defatted or degummed prior the protein extraction so it reflects the state in which the flour is found in the spaghetti; however, the fat and gum not removed could interfere with the isolation of the protein making impossible the identification of the protein fractions obtained in Figure 33-c (Oomah and Mazza 1993).



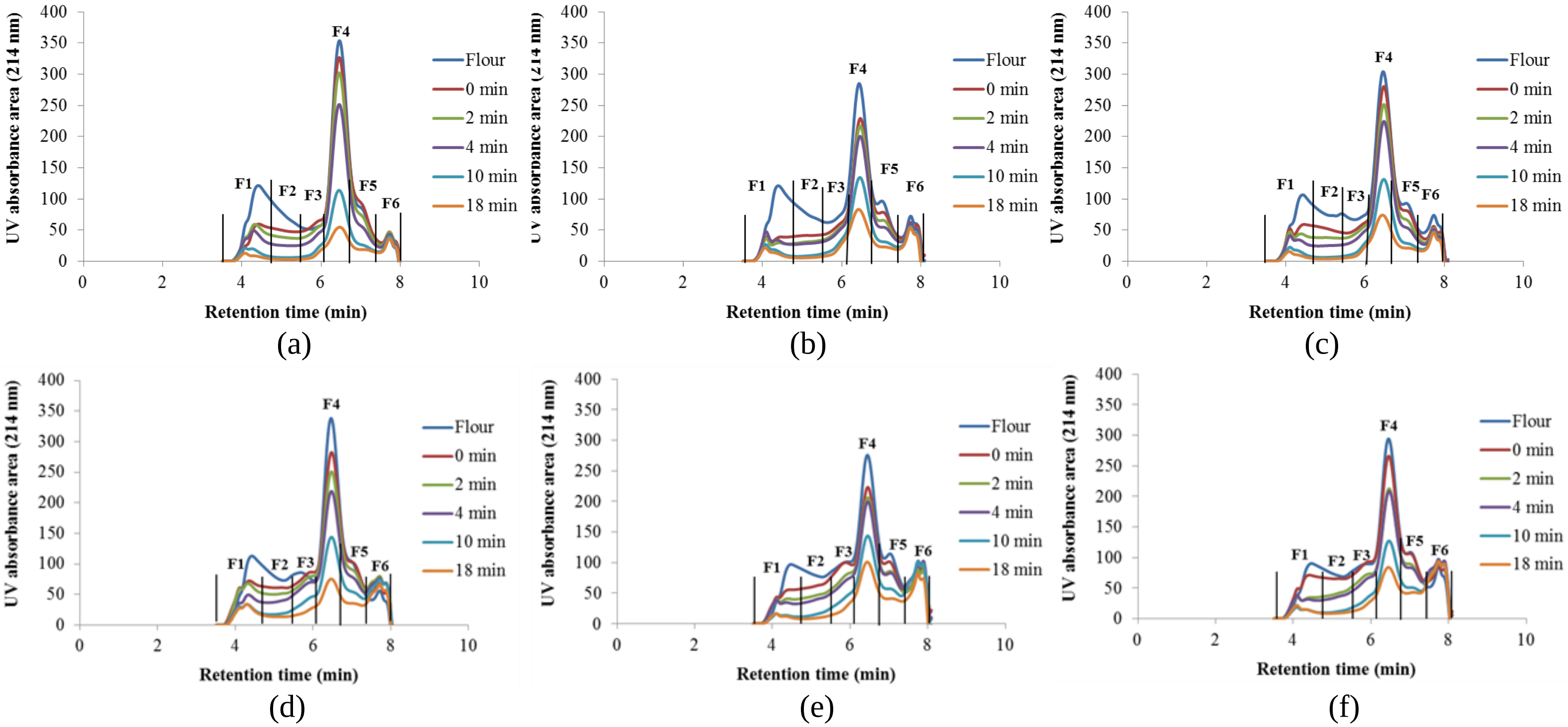
(a)

(b)

(c)

Figure 33. Size-exclusion HPLC profile of protein extract of 100% semolina spaghetti (S), 100% whole wheat flour (WW), and semolina:whole wheat flour (49:51) (SWW) (a) semolina:flaxseed flour (90:10) (SFF), whole wheat:flaxseed flour (90:10) (WWFF) and semolina:whole wheat flour:flaxseed flour (39:51:10) (SWWFF) spaghetti (b), and flaxseed flour (c).

Figure 34 displays the different chromatograms for the six flour formulations and for the correspondent spaghetti made from each formulation after the spaghetti had been cooked for 0, 2, 4, 10, and 18 min. It was possible to clearly identify the decrease in the peak heights of the curves correspondent to the uncooked spaghetti “0 min” when compared to the peaks of the curves “Flour”. The reduced curve area was attributed to the increase of the insoluble protein due to formation of polymeric aggregates that take place during pasta drying (Bruneel et al 2010) with increased molecular weight and insolubility even with sonication (Lamacchia et al 2011). This was verified with a small experiment in which the protein content of the residue left after protein extraction was measured for semolina and uncooked semolina (100%) spaghetti. Results showed that the protein amount left in the semolina after protein extraction with SDS and sonication was 1% (db). That percentage increased to 3% (db) after the semolina was transformed into spaghetti and dried. The heat treatment applied during the drying process has proven to induce changes in the protein molecular distribution of pasta products in comparison to the original flours by inducing polymerization processes via S-S bonding (Singh and MacRitchie 2004). Singh and MacRitchie (2004) and Schofield et al (1983) reported that when gluten protein were heated to 70°C changes in the size-exclusion HPLC profiles of protein extracts were noticed, especially in the glutenin fractions, which has proven to be more sensitive to high temperatures than the gliadin fraction. That might explain why the peaks F1 and F2 of the “Flour” curve, which corresponds to the glutenin fraction, decreased more dramatically than the gliadin fraction (F4) after being processed and dried into spaghetti (curve “0 min” in Fig. 34).



(a) (b) (c)

(d) (e) (f)

Figure 34. Size-exclusion HPLC profiles of protein extracts of a flour blend and spaghetti cooked for 0, 2, 4, 10 and 18 min. The composition of the spaghetti was: 100% semolina (a), 100% whole wheat flour (b), 49% semolina and 51% whole wheat flour (c), 90% semolina and 10% flaxseed flour (d), 90% whole wheat flour and 10% flaxseed flour (e), and 39% semolina and 51% whole wheat flour and 10% flaxseed flour (f).

The chromatograms in Figure 34 revealed that as cooking time progressed, the area under the curve of the SE-HPLC chromatograms decreased, indicating possible protein solubilization in the cooking water but also a decrease in protein extractability due to protein polymerization. Analysis of the residue left after protein extraction in the sample 100% semolina cooked for 18 min revealed that it contained a total protein content of 12.85% (db). The initial total protein content of this sample when it was uncooked was 14.4% (db), indicating that after 18 min of cooking only 1.55% of the spaghetti total protein content had been lost to the cooking water, and that the rest is has become unextractable even after sonication due to changes induced by the cooking process.

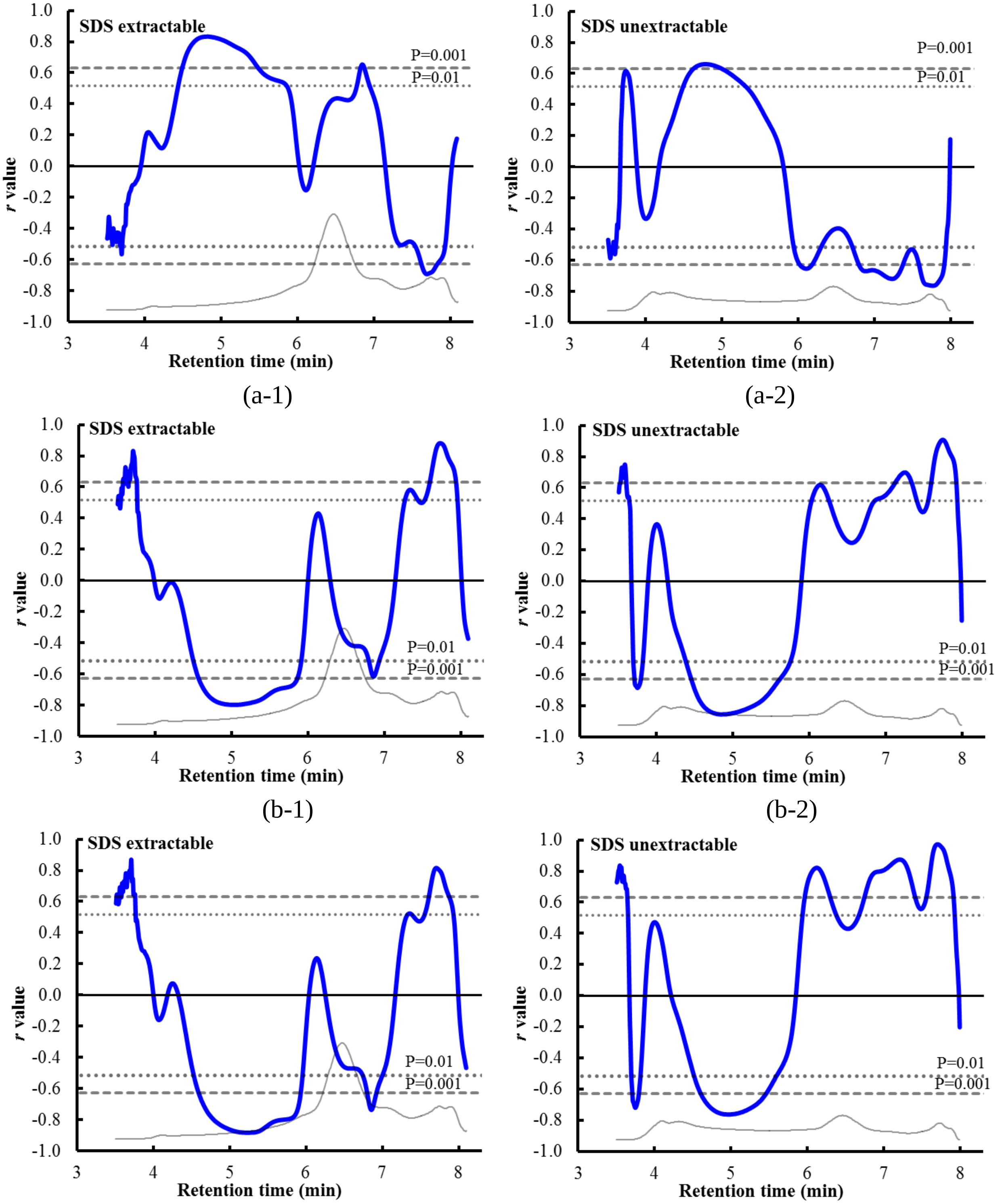
Cooking from 0 to 2 min resulted in a decrease in F2 and F4 peak area under the curve. From 2 to 10 min of cooking there was a decrease in the area under the curve for all fractions except F6. From 10 to 18 min of cooking, little to no changes were noted in the curves, except for the F4 peak, corresponding to the α-, β-, ɤ-gliadin fraction. These results indicate that the α-, β-, ɤ-gliadin fraction might contain the protein most susceptible to losing its extractability due to interaction with other proteins during the cooking process. The fraction that showed the highest resistance to the cooking process was F6, which corresponds to the albumins and globulins fractions. After 18 min of cooking, F6 remained almost the same for all six formulations indicating that the cooking process barely affected extractability of this particular protein fraction.

Two unexpected observations related to the F4 were noted: (1) all samples containing flaxseed flour displayed a higher F4 peak after being cooked for 10 and 18 min than the correspondent sample with no flaxseed; (2) even though S and SFF, with 100% or 90% semolina, respectively, displayed an initial higher F4 peak in their “Flour” curve and the “0 min” curve than the rest of the samples, after at 10 and 18 min of cooking, WW, WWFF, SWW, and SWWFF had a higher peak for the F4 fraction than did S or SFF. The first observation was particularly surprising since flaxseed flour did not show a peak for the fraction F4 as shown in Figure 33-c. This result might indicate some sort of interaction between the flaxseed and gliadin proteins when cooked for long periods of time or the possible polymerization of the flaxseed proteins that would increase their molecular weight causing them to elute along with the gliadin protein. The second observation indicates that semolina gliadins are more prone to become insoluble to SDS buffer extraction even after sonication during spaghetti cooking than the gliadin proteins found in the whole wheat flour used to make the spaghetti.

Correlations were shown specifically between the cooked firmness, cooking loss, and cooked weight from Chapter 4 and SE-HPLC A% (% of absorbance area) values as a spectrum over profiles of SDS soluble and insoluble protein from the spaghetti cooked for different cooking times (Fig. 32). At the retention time 4.5-5.7 min (F2), the cooked firmness of the spaghetti and the SE-HPLC A% had significant positive correlations for the SDS-soluble proteins ($r > 0.73$ $P \leq 0.001$) (Fig. 35-a1). Cooked firmness also showed significant positive correlations with the A% for the same retention time for the SDS-insoluble proteins, but it was lower ($r > 0.50$ $P \leq 0.01$) than for the SDS-soluble proteins (Fig. 35-a2). Cooked firmness and the A% of both SDS-soluble and -insoluble proteins also showed high negative correlations of up to r=0.65 and 0.75, respectively ($P \leq 0.001$) for the retention 7.5-8.1 min (F6). These results indicate that the firmness of spaghetti is mainly attributed to high levels of F2 or low molecular weight glutenin and low levels of albumins and globulins.

Very similar correlations were found between cooking loss and cooked weight and the A% for both SDS-soluble and -insoluble proteins (Fig. 35-b and -c). Very high positive and negative correlations (*r* >0.73, *P*≤0.001) were found between these two cooking parameters and the A% for the retention time 3.6-3.7, included within F1, and for the retention time of 4.5-5.7 min (F2), respectively, for both SDS-soluble and -insoluble proteins. For the retention time of 5.7-6.1 min (F3) both cooking loss and cooked weight correlated very well (*P*≤0.001) with the A% of only the SDS-insoluble protein, being much stronger the positive correlation for cooked weight (*r* > 0.80) than for cooking loss (*r* < 0.63) (Fig. 32-b2 and -c2). For the retention time 6.8-8.1 (F5 and F6) there were also very significant positive correlations (*r* > 0.80, *P*≤0.001) between cooking loss and cooked weight and the %A of both SDS soluble and insoluble fractions (Fig. 35-b1 and 2, -c1 and 2).

These results are evidence on the impact that the different protein fractions have on the different pasta cooking parameters. Based on these correlations, pasta firmness is associated with high levels of low molecular weight polymeric proteins (F2), which consist mainly of glutenins, and with low levels of the globulin and albumin protein fraction (F6). On the other hand, high levels of F1 and F3 and F6, and low levels of the low molecular polymeric protein fraction (F2) were associated with high cooking losses and cooked weight. Based on the information provided by Lamacchia et al (2007) and Zweifel et al (2003), protein polymerization during drying creates a protein network which entraps the starch granules. This network may restrict water absorption, thus preventing starch leaching (Bruneel et al 2010) which explains why the polymeric fraction of the protein showed such strong correlations with the pasta cooking parameters. It seems also relevant to highlight the lack of correlation of the main gliadin fractions (F4) with any of the three pasta cooking parameters. This is an indication that fraction F4 might not have a significant role in the final quality of cooked pasta and that, therefore, the quality and quantity of the fractions F1 and F2 might be more important than F4 in defining the quality of cooked pasta products as previously suggested by Feillet and Dexter (1996).



(a-1) (a-2)

(b-1) (b-2)

(c-1) (c-2)

Figure 35. Spectrum of simple linear correlation coefficients (*r*) between spaghetti cooked firmness (a), cooking loss (b), and cooked weight (c) and size-exclusion HPLC absorbance area percentage values (A%) of the SDS buffer soluble (1) and insoluble (2) proteins averaged for the six spaghetti formulations and the spaghetti cooked for 2, 4, 10, and 18 min.

Conclusions

The effect of having non-traditional ingredients in the formulation was attributed to a mere dilution effect of the starch and protein levels in comparison to the 100% semolina product. No chemical interaction was observed between the different ingredients. Conversely, cooking time had a major role in the modification of the functional properties of both protein and starch.

The starch and protein composition of non-traditional pasta changed throughout the entire 18 min that the pasta was subjected to cooking. Small but detectable differences were noted when comparing the level of protein extractability, total starch content and starch damage after cooking for 10 and 18 min. This indicated that cooking past the “optimum” cooking time does not have a big detrimental effect on the chemical composition of non-traditional spaghetti. SEM micrographs showed that complete disappearance of the white central core (assumed to be native starch) of the spaghetti strand does not imply 100% of starch gelatinization, and this was corroborated by RVA data.

Low levels of albumin and globulin and high levels of the low molecular weight polymeric proteins were related to high spaghetti firmness. High levels of the high molecular weight polymeric protein, high levels of the albumin and globulin fractions, and low levels of the low molecular polymeric fraction were associated with high cooking losses and cooked weight, indicating the involvement of these proteins in the cooked quality of the spaghetti.

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OVERALL CONCLUSIONS

Particle size distribution, level of starch damage, lipid content, and presence/absence of bran were the main parameters that influenced the behavior of the studied formulations during the pasta manufacturing process and the quality of the non-traditional pasta. These factors altered the flow properties of the ingredients in the processing line, changed the hydration requirements of the dough, affected the rheological behavior of the dough during extrusion, and influenced the quality of the dry and cooked product. However, with the proper modification of the pasta manufacturing line and adjustment of the dough hydration level, it was possible to optimize the manufacturing of non-traditional pasta products containing semolina and/or whole wheat flour with or without flaxseed flour.

Results of the flow study indicated that the minimum steepness that aluminum or stainless steel chutes and/or pipes should have for the free flow of non-traditional formulations was 46°, regardless of the hydration level of the formulations. The combination of the results of the rest of the studies allowed the identification of the formulation-hydration combinations that optimized the manufacturing of non-traditional pasta products from a processing and product quality point of view, which are: 30% for SFF, SCF, SWWFF, and SWWCF, 32% for S, WWFF, and WWCF, 33% for SWW, and 34% for WW. If a particular non-traditional formulation had to be chosen, decision should be made based on cooked quality. Better cooked quality was obtained for SWW than for WW, indicating that it is better to have some semolina in the formulation. Inclusion of flaxseed flour had an overall detrimental effect on the cooked quality, but better results were obtained when adding the flaxseed to 90% semolina (SFF and SCF), followed by SWW and then by WW. Almost no differences were detected when using CF or FF.

Consequently, if both whole wheat flour and flaxseed flour are desired in the formulation SWWFF/SWWCF were the best choices.

FUTURE RESEARCH AND APPLICATIONS

To complement the study of the flow properties of the ingredients it would be interesting to build pipes/chutes prototypes that would verify the design recommendations. There are also a large number of devices available such as Jenike's shear cell, annular shear cell, and direct shear cell tester and that allows the determination of other parameters that allow the measure of quantitative and qualitative flowing properties of food powders. Quantitative tests include angle of internal friction, the flow function, cohesion, and tensile strength. Qualitative tests include the measurement of powder compressibility and friction.

Future research needs to be done to better understand the hydration properties of the ingredients that influence the formation of wet agglomerates. Studies that involve the determination of the sorption/desorption kinetics of the formulations, would help to determine the capillary condensation, the plastic limit value of the different formulations.

The effect of adding flaxseed flour to the pasta had an overall negative effect on the quality of the product. In this line of study, it would be interesting to isolate the lipid and mucilage gum and, along with the residual flaxseed add them back individually and in combination to the pasta formulation in order to evaluate their individual and combinational effect on dough and pasta quality.

The information provided by this study is useful for pasta manufacturers that want to incorporate non-traditional ingredients in their pasta products. This study highlights the main problems derived from utilizing non-traditional ingredients in pasta making and provides possible solutions to the problems. Besides this study only deals with the incorporation of whole wheat flour and flaxseed flour, the information provided here can be applied to other non-traditional ingredients added to pasta; the magnitude of the problems will be different for each non-traditional ingredient, but the nature and origin of the problems will be the same.

APPENDIX

Table A1. Analysis of variance for angle of slide (º) and angle of repose (º) on an aluminum and stainless steel surface for different flour formulation-hydration levels combinations.

Dependent variable	Sources of variation	Df	MS	*F*-value
Angle of slide	Formulation (F)	8	170.48	51.84**
Aluminum	Rep (F)	18	3.29	1.53
	Hydration (H)	5	45.09	20.94**
	F*H	40	31.55	14.65**
	Error	90	2.15	
Angle of slide	Formulation (F)	8	169.62	21.61**
Stainless steel	Rep (F)	18	7.85	2.88**
	Hydration (H)	5	207.96	76.22**
	F*H	40	34.06	12.48**
	Error	90	2.73	
Angle of repose	Formulation (F)	8	260.76	49.18**
Aluminum	Rep (F)	18	5.30	1.11
	Hydration (H)	5	134.27	28.17**
	F*H	40	16.99	3.57**
	Error	90	4.77	
Angle of repose	Formulation (F)	8	337.60	92.23**
Stainless steel	Rep (F)	18	3.66	0.76
	Hydration (H)	5	102.09	21.26**
	F*H	40	20.00	4.17**
	Error	90	4.80	

* Significant at *P*=0.05; ** Significant at *P*=0.01; Df=degrees of freedom; MS=mean square.

Table A2. Analysis of variance for adhesion (g·sec), hardness (g), resilience (-), and springiness (mm) of dough wet agglomerates made out of different dough formulation-hydration levels combinations.

Dependent variable	Sources of variation	Df	MS	*F*-value
Adhesion	Formulation (F)	2	4992	1.92
	Rep (F)	6	2598	2.05
	Size of agglomerate (S)	3	14633	11.52**
	F*S	6	1114	0.88
	Error	18	1270	
Hardness	Formulation (F)	2	1553622	6.06*
	Rep (F)	6	256402	0.78
	Size of agglomerate (S)	3	18002788	54.53**
	F*S	6	278117	0.84
	Error	18	330154	
Resilience	Formulation (F)	2	0.0009	1.96
	Rep (F)	6	0.0005	2.80
	Size of agglomerate (S)	3	0.0145	83.21**
	F*S	6	0.0007	3.83
	Error	18	0.0002	
Springiness	Formulation (F)	2	0.005	0.18
	Rep (F)	6	0.027	6.14**
	Size of agglomerate (S)	3	0.069	15.98**
	F*S	6	0.002	0.44
	Error	18	0.004	

* Significant at *P*=0.05; ** Significant at *P*=0.01; Df=degrees of freedom; MS=mean square.

Table A3. Analysis of variance for bulk density (g/cm^3) of pasta dough made out of different dough formulation-hydration levels combinations.

Dependent variable	Sources of variation	Df	MS	*F*-value
Bulk density	Formulation (F)	8	5392.86	86.70**
	Rep (F)	18	62.20	1.79*
	Hydration (H)	4	673.35	19.39**
	F*H	32	154.65	4.45*
	Error	72	34.73	

* Significant at *P*=0.05; ** Significant at *P*=0.01; Df=degrees of freedom; MS=mean square.

Table A4. Analysis of variance for viscosity (kPa·s), consistency coefficient index (kPa·s^n), flow index (-) measured during dough extrusion in a capillary rheometer of different dough formulation-hydration levels combinations.

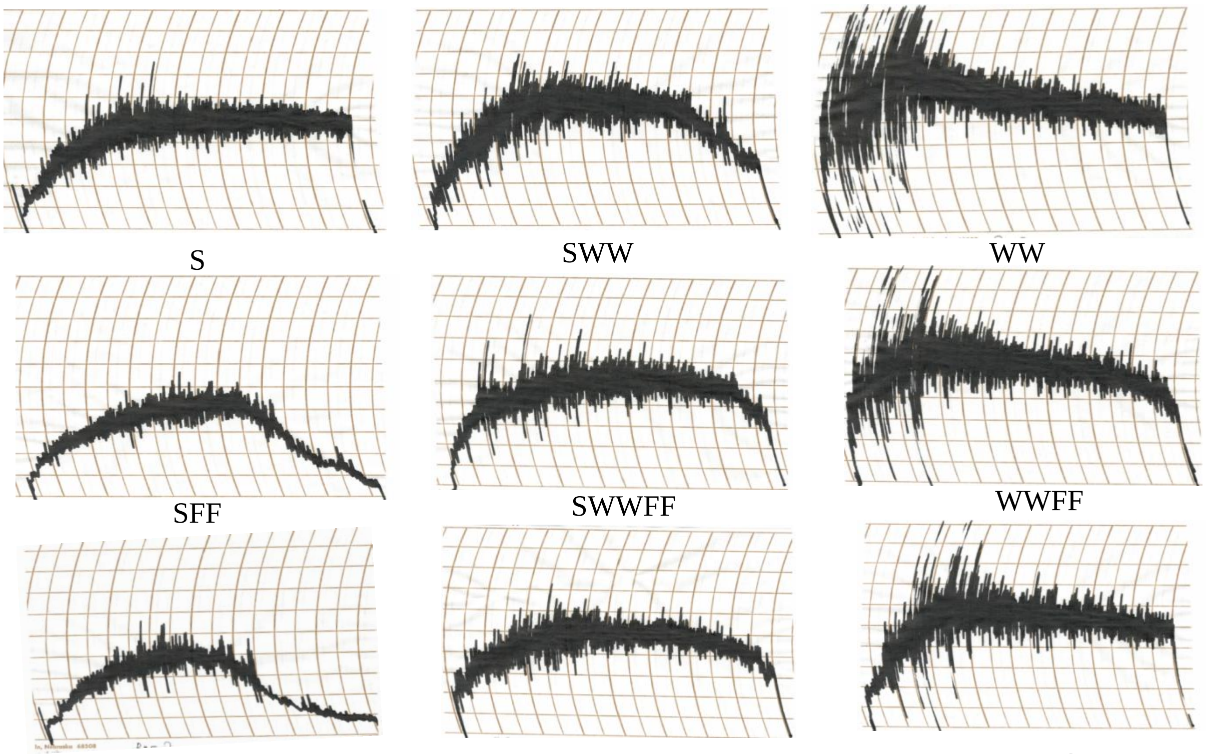
Dependent variable	Sources of variation	Df	MS	*F*-value
Viscosity	Formulation (F)	5	49.32	213.30**
	Rep (F)	6	0.23	1.49
	Hydration (H)	2	73.44	460.10**
	F*H	10	6.07	38.0**
	Error	12	0.16	
Consitency index, *k*	Formulation (F)	5	9683	12.89**
	Rep (F)	6	751	0.87
	Hydration (H)	2	43632	50.55**
	F*H	10	2390	2.77*
	Error	12	863	
Flow index, *n*	Formulation (F)	5	0.006	4.29
	Rep (F)	6	0.001	1.08
	Hydration (H)	2	0.042	32.11**
	F*H	10	0.005	3.82*
	Error	12	0.001	

* Significant at *P*=0.05; ** Significant at *P*=0.01; Df=degrees of freedom; MS=mean square.

Table A5. Analysis of variance for extrusion pressure (kPa), mechanical energy (J/sec), extrusion rate (g/sec), and specific mechanical energy (J/g), for the study of the effect of dough formulation-hydration levels combinations on the rheological behavior of pasta dough during extrusion.

Dependent variable	Sources of variation	Df	MS	*F*-value
Extrusion pressure	Formulation (F)	5	168204	45.60**
	Rep (F)	12	3689	1.26
	Hydration (H)	2	457911	156.12**
	F*H	10	8488	2.89*
	Error	24	2933	
Mechanical energy	Formulation (F)	5	25969	49.66**
	Rep (F)	12	523	0.85**
	Hydration (H)	2	103944	168.21**
	F*H	10	4920	7.96**
	Error	24	618	
Extrusion rate	Formulation (F)	5	0.897	7.80**
	Rep (F)	12	0.115	0.52
	Hydration (H)	2	2.159	9.78**
	F*H	10	0.226	1.02*
	Error	24	0.221	
Specific mechanical energy	Formulation (F)	5	1253	24.33**
	Rep (F)	12	51	0.93
	Hydration (H)	2	4933	88.67**
	F*H	10	384	6.89**
	Error	24	56	

* Significant at *P*=0.05; ** Significant at *P*=0.01; Df=degrees of freedom; MS=mean square.



S

SWW

WW

SFF

SWWFF

WWFF

SCF

SWWCF

WWCF

Figure A1. Mixograms of the nine formulations used in this study.

S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour- Fine Flaxseed (39:51:10), SWWFF: Semolina-Whole Wheat Flour- Coarse Flaxseed (39:51:10).

Table A6. Analysis of variance for *L*-value, *a*-value, and *b*-value of dry spaghetti made out of different formulation-hydration level combinations.

Dependent variable	Sources of variation	Df	MS	*F*-value
HL	Formulation (F)	8	585.38	27.24**
	Rep (F)	18	21.49	1.88
	Hydration (H)	4	77.47	6.77**
	F*H	32	11.25	0.98
	Error	72	11.45	
a	Formulation (F)	8	30.08	48.68**
	Rep (F)	18	0.62	25.34**
	Hydration (H)	4	2.43	99.55**
	F*H	32	0.07	2.96**
	Error	72	0.02	
b	Formulation (F)	8	250.72	433.29**
	Rep (F)	18	0.58	12.55**
	Hydration (H)	4	0.05	1.15
	F*H	32	0.16	3.4**
	Error	72	0.05	

* Significant at *P*=0.05; ** Significant at *P*=0.01; Df=degrees of freedom; MS=mean square.
Hunter *L*-value represents brightness; *a*-value represents redness when positive and greenness when negative; *b*-value represents yellowness when positive and blueness when negative.

Table A7. Effect of the spaghetti formulation hydration level interaction on the *L*-value color parameters of dry spaghetti.

Formulation[a]	HL
S	52.9 a
SFF	45.0 b
SCF	46.1 b
WW	34.6 d
WWFF	36.6 cd
WWCF	35.2 cd
SWW	38.4 c
SWWFF	38.3 c
SWWCF	36.2 cd
Hydration (%)[b]	
30	43.2 a
31	40.6 b
32	39.8 b
33	39.4 b
34	38.9 b

Values within same main effect in the same column followed by different letters are statistically different for $P \leq 0.05$.

[a] Values averaged across dough hydration levels.

[b] Values averaged across spaghetti formulation.

Hunter *L*-value represents brightness.

S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour- Fine Flaxseed (39:51:10), SWWFF: Semolina-Whole Wheat Flour- Coarse Flaxseed (39:51:10).

Table A8. Effect of the spaghetti formulation-hydration level interaction on the a- and b-value color parameters of dry spaghetti.

Formulation	Hydration	a	b
	%	-	-
S	30	3.75 u	23.1 a
	31	3.66 u	23.2 a
	32	3.79 u	23.1 a
	33	4.01 u	22.7 ab
	34	4.05 u	22.4 b
SFF	30	6.41 t	17.8 cd
	31	6.49 t	18.1 cd
	32	6.40 t	18.4 c
	33	6.53 t	18.2 c
	34	6.80 q-t	18.4 c
SCF	30	6.63 st	17.5 d
	31	6.72 rst	17.9 cd
	32	6.84 p-t	17.9 cd
	33	6.81 q-t	17.9 cd
	34	7.07 n-s	17.8 cd
WW	30	7.83 f-j	11.6 ij
	31	8.51 b-e	11.1 jk
	32	8.72 a-d	11.1 jk
	33	9.02 ab	11.4 jk
	34	9.08 a	11.3 jk
WWFF	30	6.75 rst	11.4 jk
	31	7.05 o-s	11.4 jk
	32	7.33 j-p	11.2 jk
	33	7.59 h-n	11.5 jk
	34	7.74 g-k	11.3 jk
WWCF	30	7.16 m-r	11.3 jk
	31	7.57 h-n	11.0 k
	32	7.77 g-k	10.9 k
	33	7.69 h-l	10.8 k
	34	8.08 e-h	10.9 k
SWW	30	7.90 f-i	13.9 f
	31	8.25 d-g	14.4 ef
	32	8.48 cde	14.4 ef
	33	8.58 a-e	14.6 e
	34	8.81 abc	14.5 ef

Table A8. Effect of spaghetti formulation-hydration level interaction on the *a*- and *b*-value color parameters of dry spaghetti (continued).

Formulation	Hydration %	a -	b -
SWWFF	30	7.19 l-r	12.8 gh
	31	7.34 j-p	12.8 g
	32	7.45 i-o	12.8 gh
	33	7.70 h-l	12.8 g
	34	8.06 e-h	13.1 g
SWWCF	30	7.30 k-q	12.2 hi
	31	7.67 h-m	12.6 gh
	32	7.83 f-j	12.7 gh
	33	8.07 e-h	12.8 gh
	34	8.34 c-f	12.8 g

Values in the same column followed by different letters are statistically different for $P \leq 0.05$.
Hunter *a*-value represents redness when positive and greenness when negative; *b*-value represents yellowness when positive and blueness when negative.
S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour- Fine Flaxseed (39:51:10), SWWFF: Semolina-Whole Wheat Flour- Coarse Flaxseed (39:51:10).

Table A9. Analysis of variance for density (g/cm^3), diameter (cm), mechanical strength (g), and flexibility (mm) of different non-traditional spaghetti formulation-hydration level combinations.

Dependent variable	Sources of variation	Df	MS	*F*-value
Density	Rep (F)	18	0.0021	7.04**
	Formulation (F)	8	0.0016	0.77
	Hydration (H)	2	0.0019	6.54**
	F*H	16	0.0006	1.93
	Error	36	0.0003	
Diameter	Rep (F)	18	0.00005	6.06**
	Formulation (F)	8	0.00001	0.27
	Hydration (H)	2	0.00011	12.29 **
	F*H	16	0.00002	2.28
	Error	36		
Mechanical strength	Rep (F)	18	7.96	0.98
	Formulation (F)	8	145.89	18.34**
	Hydration (H)	4	22.48	2.78*
	F*H	32	3.46	0.43
	Error	72	8.09	
Flexibility	Rep (F)	18	3.37	1.61
	Formulation (F)	8	709.09	210.12**
	Hydration (H)	4	48.76	23.32**
	F*H	32	4.75	2.27**
	Error	72	2.09	

* Significant at *P*=0.05; ** Significant at *P*=0.01; Df=degrees of freedom; MS=mean square.

Table A10. Analysis of variance for cooking loss (%), cooked weight (g/g db), cooked firmness (g·cm), and optimum cooking time (sec) of different non-traditional spaghetti formulation-hydration level combinations.

Dependent variable	Sources of variation	Df	MS	*F*-value
Cooking loss	Rep (F)	18	0.38	2.97**
	Formulation (F)	8	5.2	13.61**
	Hydration (H)	4	0.08	0.65
	F*H	32	0.15	1.15
	Error	72	0.13	
Cooked weight	Rep (F)	18	4.15	14.30**
	Formulation (F)	8	11.91	2.87*
	Hydration (H)	4	0.29	1.01
	F*H	32	0.52	1.80*
	Error	72	0.29	
Cooked firmness	Rep (F)	18	10.34	3.15**
	Formulation (F)	8	56.19	5.44**
	Hydration (H)	4	26.51	8.09**
	F*H	32	3.68	1.12
	Error	72	3.28	
Cooking time	Rep (F)	18	0.21	5.12**
	Formulation (F)	8	2.28	19.79**
	Hydration (H)	4	0.04	56.02**
	F*H	32	3.68	1.00
	Error	72	0.04	

* Significant at P=0.05; ** Significant at *P*=0.01; Df=degrees of freedom; MS=mean square.

Table A11. Effect of spaghetti formulation and dough hydration level on the cooking loss (%) and firmness (g·cm) of non-traditional spaghetti.

Formulation[a]	Cooking loss	Firmness
	%	g·cm
S	5.19 c	5.22 a
SFF	4.87 c	4.79 ab
SCF	4.81 c	4.85 ab
WW	6.54 a	4.65 bc
WWFF	6.26 a	4.29 cd
WWCF	6.34 a	4.11 d
SWW	6.13 a	5.04 ab
SWWFF	5.62 bc	4.29 cd
SWWCF	5.72 b	4.22 cd
Hydration (%)		
30	5.74 a	4.94 a
31	5.76 a	4.76 a
32	5.71 a	4.71 ab
33	5.87 a	4.47 b
34	5.75 a	4.49 b

Values in the same column within the same main effect followed by different letters are statistically different for $P \leq 0.05$.

[a] Values averaged across dough hydration levels.

[b] Values averaged across spaghetti formulation.

S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour- Fine Flaxseed (39:51:10), SWWFF: Semolina-Whole Wheat Flour- Coarse Flaxseed (39:51:10).

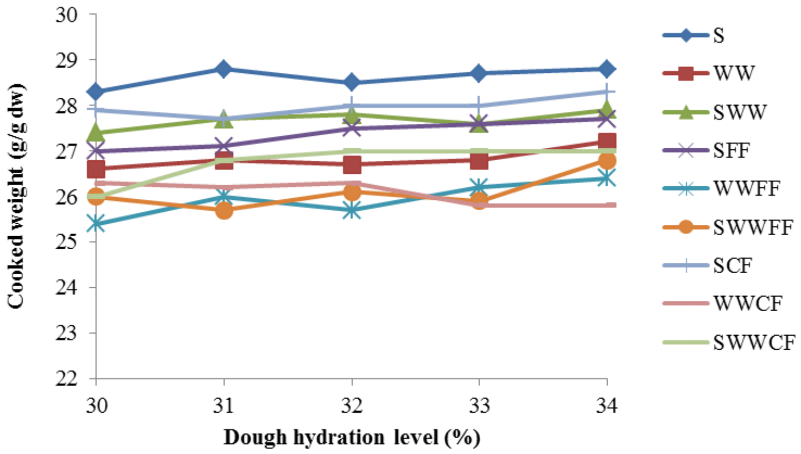


Figure A2. Effect of the spaghetti formulation and dough hydration level interaction on the cooked weight (g/g db) of non-traditional spaghetti.

LSD=1.724.
S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), SCF: Semolina-Coarse Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), WWCF: Whole Wheat Flour-Coarse Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour- Fine Flaxseed (39:51:10), SWWFF: Semolina-Whole Wheat Flour- Coarse Flaxseed (39:51:10).

Table A12. Analysis of variance for the cooking loss (%), cooked weight (g/g db), and cooked firmness (g·cm) of different non-traditional spaghetti formulation-hydration level combinations when spaghetti was cooked at 2, 4, 6, 8, 10, 12, 14, and 18 min.

Dependent variable	Sources of variation	Df	MS	*F*-value
Cooking loss	Formulation (F)	8	33.967	24.99**
	Rep (F)	18	1.359	7.96**
	Hydration (H)	2	11.519	67.43**
	F*H	16	0.412	2.42*
	Rep(F*H)	36	0.171	2.01**
	Cooking Time (CT)	7	272.398	3200.34**
	F*CT	56	0.476	5.59**
	Hy*CT	14	0.142	1.67
	F*Hy*CT	112	0.077	0.95
	Error	378	0.085	
Cooked weight	Formulation (F)	8	0.347	11.54**
	Rep (F)	18	0.031	4.88**
	Hydration (H)	2	0.447	72.54**
	F*H	16	0.013	2.06*
	Cooking time (CT)	7	21.586	5870.38**
	Rep(F*H)	36	0.006	1.68*
	F*CT	56	0.016	4.41**
	H*CT	14	0.001	2.61**
	F*H*CT	112	0.004	0.95
	Error	378	0.004	
Cooked firmness	Formulation (F)	8	1211.158	12.45**
	Rep (F)	18	97.273	1.66
	Hydration (H)	2	2053.838	35.03**
	F*H	16	154.331	2.63**
	Rep(F*H)	36	58.635	0.86
	Cooking Time (CT)	7	56515	824.72**
	F*CT	56	984.091	14.36**
	Hy*CT	14	367.788	5.37**
	F*Hy*CT	112	103.158	1.51**
	Error	378	68.526	

* Significant at P=0.05; ** Significant at *P*=0.01; Df=degrees of freedom; MS=mean square.

Table A13. Analysis of variance for the total starch content (%) and starch damage (%) of non-traditional spaghetti hydrated to 32% moisture cooked for 2, 4, 10, and 18 min.

Dependent variable	Sources of variation	Df	MS	*F*-value
Total starch	Formulation (F)	5	417.451	311.30**
	Rep (F)	12	1.341	3.31**
	Cooking time (CT)	4	83.746	206.70**
	F*CT	20	0.714	1.76
	Error	48	0.405	
Starch damage	Formulation (F)	5	13.539	0.96
	Rep (F)	12	14.043	2.38*
	Cooking time (CT)	4	399.649	67.65**
	F*CT	20	5.325	0.90
	Error	48	5.908	

* Significant at P=0.05; ** Significant at *P*=0.01; Df=degrees of freedom; MS=mean square.

Table A14. Analysis of variance for peak time (min), peak viscosity (RVU), breakdown (RVU), trough (RVU), setback (RVU), and final viscosity (RVU) of non-traditional spaghetti hydrated to 32% moisture cooked for 2, 4, 10, and 18 min.

Dependent variable	Sources of variation	Df	MS	*F*-value
Peak time	Formulation (F)	5	1.667	24.56**
	Rep (F)	12	0.068	1.98*
	Cooking time (CT)	4	2.025	59.20**
	F*CT	20	0.519	15.20**
	Error	48	0.034	
Peak viscosity	Formulation (F)	5	1764.285	97.39**
	Rep (F)	12	18.116	1.34
	Cooking time (CT)	4	8016.946	591.56**
	F*CT	20	106.107	7.83**
	Error	48	13.552	
Breakdown	Formulation (F)	5	112.195	10.77**
	Rep (F)	12	10.421	2.34*
	Cooking time (CT)	4	2214.844	496.79**
	F*CT	20	83.069	18.63**
	Error	48	4.458	
Trough	Formulation (F)	5	1294.244	156.23**
	Rep (F)	12	8.284	0.98
	Cooking time (CT)	4	1913.801	226.53**
	F*CT	20	31.433	3.72**
	Error	48	8.448	
Setback	Formulation (F)	5	1746.633	55.09**
	Rep (F)	12	31.702	1.56
	Cooking time (CT)	4	8252.615	407.03**
	F*CT	20	62.621	3.09**
	Error	48	20.275	
Final viscosity	Formulation (F)	5	3839.867	72.02**
	Rep (F)	12	53.314	1.23
	Cooking time (CT)	4	18070	416.95**
	F*CT	20	119.137	2.75**
	Error	48	43.338	

* Significant at P=0.05; ** Significant at *P*=0.01; Df=degrees of freedom; MS=mean square.

Table A15. Total protein content of formulation blends and non-traditional spaghetti hydrated to 32% moisture cooked for 2, 4, 10, and 18 min.

Formulation	Cooking time	Protein content (db)
	min	%
S	Flour	14.0
	0	13.9
	2	14.0
	4	14.0
	10	14.1
	18	14.4
WW	Flour	13.8
	0	13.5
	2	13.5
	4	13.7
	10	14.2
	18	14.0
SWW	Flour	13.8
	0	13.7
	2	13.8
	4	13.9
	10	14.0
	18	14.2
SFF	Flour	15.0
	0	14.7
	2	14.9
	4	14.8
	10	14.9
	18	15.1
WWFF	Flour	15.4
	0	14.6
	2	14.6
	4	14.8
	10	15.1
	18	15.0

Table A15. Total protein content of formulation blends and non-traditional spaghetti hydrated to 32% moisture cooked for 2, 4, 10, and 18 min (continued).

Formulation	Cooking time	Protein content (db)
	min	%
SWWFF	Flour	14.7
	0	14.6
	2	14.7
	4	14.8
	10	14.9
	18	15.1

S: Semolina 100%, SFF: Semolina-Fine Flaxseed (90:10), WW: Whole Wheat Flour 100%, WWFF: Whole Wheat Flour-Fine Flaxseed (90:10), SWW: Semolina-Whole Wheat Flour (49:51), SWWFF: Semolina-Whole Wheat Flour- Fine Flaxseed (39:51:10).